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Machining-induced white layers: Quantitative characterisation and non-destructive detection

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Summary

Machining-induced white layer is a critical surface integrity feature that is not tolerable at any thickness on a machined gas turbine engine component, due to the detrimental effect it can have on the fatigue performance. The assessment of white layers in the aircraft gas turbine industry is currently a destructive evaluation process but a non-destructive white layer assessment method would allow for faster inspection without the need to sacrifice components. A review of the current state-of-the-art in the characterisation of machining-induced white layers identified gaps in the knowledge concerning their properties and formation mechanisms in titanium alloys. Although a small number of studies have investigated the non-destructive detection of white, however, no technique exists that can detect this anomalous feature in all aeroengine alloys. This thesis considers the state-of-the-art and improves the fundamental understanding of white layer characterisation and detection.

Quantitative characterisation of the mechanical and metallurgical properties of white layers generated in three aircraft gas turbine engine alloys was undertaken. White layer formation was attributed to severe plastic deformation and subsequent dynamic recrystallisation in the microstructure of the near-surface region. It was found that white layers in all materials were composed of nanoscale grains having a higher hardness relative to the bulk and a large compressive surface residual stress state. White layers in titanium were measured to possess a basal crystallographic texture and an increased concentration of alpha-stabilizing elements.

Methods for non-destructive detection of white layers were proposed based on the knowledge obtained from the characterisation work. A grain size-sensitive x-ray diffraction peak breadth comparison method was shown to facilitate white layer detection in all three alloys, to a resolution comparable with the existing destructive method. Texture-based x-ray diffraction and surface acoustic wave methods and a residual stress-based Barkhausen noise method were also shown to facilitate the detection of a white layer, however, they had a lower resolution than the peak breadth method. The newly developed peak breadth comparison method for white layer detection could be utilised in industry for the non-destructive detection of white layers, thereby adding value to the manufacturing process by improving confidence in manufacturing quality, reducing the inspection time and offering a potential reduction in quality control costs.

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Nomenclature

Symbol	Name
Machining features	
SI	Surface integrity
WL	White layer
SG	Swept grain
SPD	Severe plastic deformation
Machining parameters	
a_e, a_p	Axial and radial depth of cut (milling)
w	Depth of cut (turning)
α	Tool rake angle
t_1, t_2, t_i	Uncut, cut and instantaneous chip thickness
r	Cutting edge radius
ϕ	Shear plane angle
R', R	Force of the tool on the chip and the force of the workpiece on the underside of the chip
F_μ, F_R, F_S, F_N	Machining forces parallel to the rake face, normal to the rake face, parallel to the shear plane and normal to the shear plane.
F_c, F_r, F_t	Machining forces in the cutting, radial and thrust directions
f_t, f_r	Feed per tooth and per revolution
F_x, F_y, F_z	Machining forces in Cartesian coordinates
h	Instantaneous chip thickness
K_{tc}, K_{rc}	Specific machining forces in the cutting and radial directions
K_{te}, K_{re}	Specific machining forces due to the edge effect in the cutting and radial directions
VB	Flank wear land
V_c	Cutting Speed
φ	Instantaneous immersion angle

Workpiece and sample designations	
CD	Cutting direction
CR	Cross-rolled
CRD	Cross rolled direction
FAST	Field assisted sintering technology
FD	Feed direction
FRD	Final rolling direction
ND	Normal direction
OD	Outside diameter
R1	Arbitrary axis in the plane perpendicular to the billet axis
R2	Axis perpendicular to R1 in the plane perpendicular to the billet axis
RD	Rolling direction
SCMV	Super Chrome-Molybdenum-Vanadium
TD	Transverse direction
U/D	Uni-directional
Characterization	
AFM	Atomic force microscope
C	DSIMS intensities
D	Diffusion coefficient
DSIMS	Dynamic secondary ion mass spectrometry
E	Elastic modulus
EBSD	Electron back-scatter diffraction
EDXS	Energy-dispersive x-ray spectroscopy
FEG	Field emission gun
H	Hardness
SEM	Scanning electron microscope
t	Diffusion time
TEM	Transmission electron microscope
ν	Poisson's ratio
x	Diffusion distance
XRD	X-ray diffraction
σ_y	Yield strength
σ_{UTS}	Ultimate tensile strength

Crystallography and recrystallization	
BCC	Body centred cubic
BCT	Body centred tetragonal
CDR	Continuous dynamic recrystallization
d	Lattice spacing
DR	Dynamic recrystallization
DDR	Discontinuous dynamic recrystallization
FCC	Face centred cubic
HCP	Hexagonal close packed
HPT	High pressure torsion
MRD	Multiples of random density
ODF	Orientation distribution function
TI	Texture index
α, β	Pole figure angles
ψ, ϕ, ω	Goniometer angles (tilt axis, normal axis, rotation axis)
Non-destructive testing	
AE	Acoustic emission
AET	Acoustic emission testing
BN	Barkhausen noise
DPI	Dye penetrant inspection
E	Energy
EDXRD	Energy-dispersive x-ray diffraction
ECT	Eddy current testing
f	Frequency
MPI	Magnetic particle inspection
NDT	Non-destructive testing
NDE	Non-destructive evaluation
PAS	Positron annihilation spectroscopy
R	Electrical resistance
RMS	Root mean square
SAW	Surface acoustic wave
SRAS	Spatially resolved acoustic spectroscopy
ρ	Electrical resistivity
v	Propagation velocity
δ	Skin depth of eddy currents
μ_r	Magnetic permeability relative to a vacuum
λ	Wavelength

Micro-grid strain measurement	
DIC	Digital image correlation
EBL	Electron beam lithography
F	Deformation gradient
v, V	Deformed and undeformed vectors
OM	Orthogonal milling
R	Rotation tensor
U	Stretch tensor
θ_p	Principal strain angle
Q	Transformation matrix
ϵ_T	True strain
Barkhausen noise	
B	Magnetization
B_R	Magnetic Remanence
BN_{max}	Maximum Barkhausen noise for a magnetizing frequency sweep
f_0	Characteristic frequency
f_{max}	Frequency at which maximum Barkhausen noise is observed
H	Magnetic field strength
H_R	Coercive field
k_B	Boltzmann constant
MAE	Magneto-acoustic emission
MBE	Magnetic Barkhausen emission
MCS	Magnetizing current sweep
MFS	Magnetizing frequency sweep
mp	Magnetoelastic parameter
MVS	Magnetizing voltage sweep
NC_1	Normalized current at which maximum slope is observed
NC_2	Normalized current at which maximum second derivative is observed
S_1	Maximum slope of the magnetizing current sweep
S_2	Maximum second derivative of the magnetizing current sweep
V_{pp}	Voltage peak-to-peak
τ	Mean waiting time

X-ray diffraction non-destructive testing

C	Lattice strain peak breath constant
D	Diffraction domain size
EP	Electropolished
FWHM	Full-width at half maximum
HT	Heat treatment
I	Intensity of x-rays
I_0	Intensity of x-rays at the surface
IR	Intensity ratio
k	Scherrer equation constant
L	X-ray path length
n	Number of samples in set
PB	Peak broadening
POD	Probability of detection
POFP	Probability of false positive
s	Standard deviation of peak breadths
s^2	Variance of peak breadths
t	White layer thickness
$t_{90/95}$	Minimum detectable feature size
$tdist$	Value from t-distribution
w	Weighting
W-H	Williamson-Hall
x	Depth beneath the surface
β	Threshold value
ε	Lattice strain
σ	Lattice stress
μ	Linear attenuation coefficient
θ	Diffraction angle

1 Introduction

1.1 Motivation

Machining is used in the manufacturing of almost all aircraft gas turbine engine (aeroengine) components and is critical to finalizing the shape and surface quality of a part before entering service. The quality of a machined surface, its so-called surface integrity, has a direct effect on the functional capabilities of a component. The safety-critical nature of the aerospace industry, which arises due to the immediate catastrophic consequences of failure relative to other industries such as land-based power generation, necessitates a thorough inspection of machined components to assess their surface integrity.

The surface integrity inspection process in industry invariably requires a form of cross-sectional optical microscopy as some aspects of the surface integrity, such as white layers and swept grain regions, cannot be identified from visual inspection of the machined surface. This microscopy inspection inherently requires parts to be destructively sectioned into small (sub 30 mm) samples to permit access to the cross-section before being mounted in resin, ground, polished and chemically etched. The chemical etching is necessary to reveal the microstructure of the sample and hydrofluoric acid, a highly toxic chemical, is required for the case of titanium components. The quality and accuracy of the microscopy assessment are dependent on the level of grinding and polishing pad wear, the chemical etchant concentration and the etching time and this, therefore, gives rise to inherent variability in the inspection process. Due to the number of steps involved, this surface integrity assessment process is time-consuming and by necessity should take place in a laboratory away from the machine shop floor. However, despite the limitations of this method, destructive cross-sectional microscopy is critical to surface integrity assessment as it is currently the only way of inspecting for certain anomalous features that can arise during machining operations, particularly in aeroengine materials.

Of all the anomalous surface features that are assessed during surface integrity inspection, machining-induced white layers are perhaps the least understood. Whilst previous studies investigating the formation mechanisms and machining conditions that encourage white layer formation have been undertaken for specific alloys, there is a lack of a coherent general review that establishes the state-of-the-art.

White layers are known to cause a reduction in fatigue life [1] and as such, if any white layer were identified on a surface, it would cause the component to fail surface integrity assessment. The drive for

increased production rates in industry, to reduce overall costs, has necessitated the adoption of higher metal removal rates, which can include the use of higher cutting speeds. White layer formation has been shown to increase with cutting speeds as well as tool wear. Therefore, as cutting speeds are pushed higher and higher to enhance production rates, it is logical to predict that white layer formation and the ability to detect this anomalous feature will become increasingly relevant.

A non-destructive alternative to the current destructive inspection process would offer several improvements. These include the ability to inspect all components before they enter service, rather than sacrificing one component from a batch and assuming all other components produced in the same way are identical. Additionally, a non-destructive method could eventually offer on-machine surface integrity inspection as part of the production line, significantly reducing lead-time for inspection results. This allows for advancements in the machining process, such as the development of optimal cutting conditions or more effective tool geometry, to be researched and validated at a faster rate. By providing a quantitative measure from inspection that is related to an underlying key property of a white layer, a non-destructive method could have the additional benefit of removing the variability introduced during sample preparation and the subjectivity in the interpretation of an optical micrograph by an inspector. These benefits would improve the reliability and reproducibility of the white layer inspection method and offer value to the partners supporting this research and the wider industry.

The manufacturing industry is moving towards automation of operations as part of smart factories through the 4th industrial revolution (industry 4.0). One of the key concepts of industry 4.0 is the digital twin, whereby every part on the production line will have a digital representation. This digital twin is compared to the desired component using data captured from sensors and, from this comparison, decisions can be made on actions required to change the real-world component. Cross-sectional microscopy is an analogue method for surface inspection that is not compatible with industry 4.0. By contrast, a non-destructive inspection method could facilitate the assessment of a surface on-machine, from which decisions could be made on the actions required for that component.

Due to the shortcomings of the current inspection methods, the benefits of a non-destructive alternative and the requirement to align surface integrity assessment with the future manufacturing operational concepts, the development of a non-destructive method for assessment of machining-induced

white layers is highly relevant. To develop such a method, a coherent review of the literature in relation to the formation and properties of white layers in a range of different aeroengine materials is required. Therefore, this study includes a consolidation and a critical review of the current literature, in addition to experimental work filling in the identified gaps, specifically the determination of the key properties of white layers in a titanium alloy. This knowledge is used to develop a novel non-destructive testing method that can be used to detect machining-induced white layers with improved resolution compared to the current optical microscopy process.

1.2 Aim

The aim of this study is to develop a technique to detect machining-induced white layers non-destructively by initially characterizing machining induced white layers in aeroengine alloys and then using the acquired knowledge of the measured key properties, to identify the most suitable non-destructive testing methods.

1.3 Objectives

1. To review previous work and the current state-of-the-art in destructive and non-destructive evaluation of machining induced white layers
2. To characterise the key physical properties of white layers and use this knowledge to propose suitable non-destructive testing methods based on the physical phenomena on which these methods are based.
3. To enhance the understanding of the formation of machining induced white layers.
4. To develop a non-destructive testing method for detecting machining-induced white layers and then perform proof-of-concept measurements on machined samples.
5. To identify the limitations of any successful technique and outline a strategy for implementation in industry.

1.4 Thesis structure

The thesis has been structured in the following format to introduce the current published knowledge in characterisation and non-destructive detection of machining-induced white layers before outlining the experimental methodologies employed for generating and characterising surfaces containing a white layer. Following this, the results from characterisation work are presented and suitable non-destructive testing methods are proposed. Finally, the results from non-destructive measurements on machined surfaces containing white layers are presented. The detailed structure of the thesis is as follows:

Chapter 1 outlines the motivation for the investigation into the non-destructive detection of machining-induced white layers. The scope, aims and objectives are stated.

Chapter 2 introduces the three material groups widely used in aeroengines, titanium, nickel and iron-based alloys. Their key properties and areas of application are discussed. An overview of turning and milling, the machining processes used in this study, is provided. The chapter concludes with a discussion on the difficulties faced when machining components made from aeroengine materials and how this can affect their resultant surface integrity. An overview of the types of anomalous features incorporated under the term surface integrity is provided and selection of the feature of interest in this study is made.

Chapter 3 introduces the key feature, white layers, in detail before reviewing the state-of-the-art in white layer formation mechanisms in machining and identification of the machining conditions that encourage white layer formation. The chapter also includes a discussion of the key physical properties of white layers identified in the literature.

Chapter 4 outlines the available non-destructive surface testing methods and reviews the state-of-the-art in detecting white layers with these techniques. Due to the limited prior research in this specific application, comparisons are drawn between the underlying phenomena of each technique and the key physical properties of white layers reported in chapter 3. The chapter concludes with a preliminary assessment of the feasibility of using these methods to detect white layers. Each technique is assigned a usefulness score and a practicality score, facilitating a ranking of the potential techniques to guide the work in later chapters.

Chapter 5 outlines the experimental methods employed throughout this study to generate and characterise white layers. The materials and machining methods are described and then the destructive methods used for the quantitative characterisation of white layers are explained.

Chapter 6 presents the results of the quantitative characterisation of machined surfaces in three aeroengine alloys, in particular, the results from nanohardness, microstructure, phase and chemical composition, crystallographic texture, residual stress profile and micro-grid strain measurements are reported. Following this, the chapter includes a discussion of the key physical properties of white layers in relation to the bulk material properties and those reported in the literature. The results, as well as previously described mechanisms from chapter 3, are used to infer a mechanism for white layer formation

in machined titanium. The key physical properties are used to re-evaluate the proposed non-destructive testing methods from chapter 4 and propose methods for detecting white layers in each aeroengine material group.

Chapter 7 defines the methodology of the non-destructive tests used to detect white layers in the machined surfaces characterised in the previous chapter. The methods discussed include a grain-size-sensitive x-ray diffraction (XRD) method, texture-sensitive XRD and surface acoustic wave (SAW) methods and a residual stress-sensitive Barkhausen noise (BN) method.

Chapter 8 presents results from XRD, SAW and BN non-destructive evaluation of machined surfaces. The results are discussed in the context of the underpinning physical phenomena of each technique. An assessment of each method in relation to their effectiveness for the detection of white layers is provided, based on the detection limits and inspection time among other performance indicators. The chapter concludes with a comparison between the proposed non-destructive methods and the existing destructive cross-sectional microscopy inspection method.

Chapter 9 summarises the key findings and conclusions from the study.

Chapter 10 outlines areas for further research based on the findings of this body of work.

Chapter 11 makes recommendations for the adoption of non-destructive methods for white layer detection in industry and discusses the potential to detect other surface integrity features.

2 Materials and machining methods in aeroengine manufacturing

This chapter describes the materials and machining methods used in aeroengine manufacturing. The three common material groups are introduced with a focus on a specific alloy of each material. Following this, the concept of chip formation, the fundamental theory underpinning conventional machining operations, is described before specific operations, turning and milling, are explained. The chapter finishes with a discussion around the challenges faced when machining the three common aeroengine alloys and how this affects the quality of a component, post-machining.

2.1 Materials for aeroengines

Modern aeroengine are composed of three primary alloy groups, titanium-based, nickel-based and iron-based (steel) alloys. The need for a range of materials is due to the vastly different thermomechanical loads experienced across an engine. For example, the fan blades at the front of the engine, which operate at ambient air temperature, require high strength, low weight, high fracture toughness and high fatigue strength [2]. By contrast, turbine blades, which are exposed to temperatures above 1000 °C, primarily require strength at high temperature and creep resistance [3]. Consequently, a different material is selected for the fan blades (titanium alloy) and the turbine blades (nickel-based superalloy) because titanium alloys do not maintain their strength at the high temperatures reached in the turbine and conversely, the density of nickel superalloys is too high to use as the fan blade material.

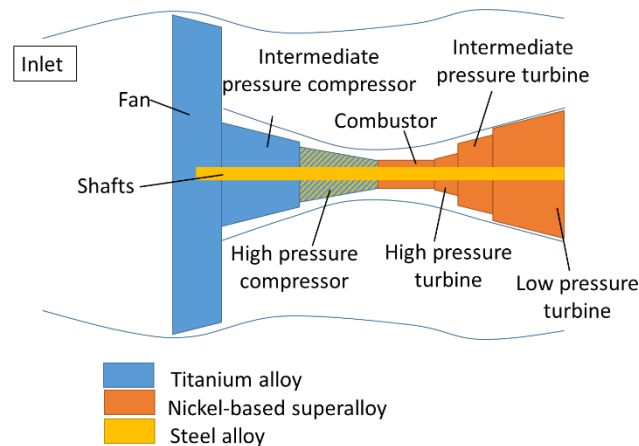


Figure 2.1- Schematic highlighting the material usage in a high-bypass ratio aeroengine, after [3].

The distribution of materials within an aeroengine is highlighted in Figure 2.1 and can be loosely summarised as follows. Titanium-alloys are used in the compression (cold) side of the engine, nickel

superalloys are used in the turbine (hot) side, and steels are used as the shaft material as well as for the static parts of the combustion end of the compressor.

2.1.1 Titanium alloys

Titanium is an allotropic light metal, widely used in aerospace applications [4] due to the high specific strength that is achievable when alloyed with elements such as vanadium and aluminium [5]. Alloying can, for example, increase the ultimate tensile strength from around 300 MPa in commercially pure titanium, up to 1000 MPa in the heat treatable aeroengine titanium-alloys [4]. Titanium exists as one of two phases, the low-temperature α -phase, which has a hexagonal close packed (HCP) crystal structure and the high-temperature body centred cubic (BCC) β -phase [6], as shown in Figure 2.2.

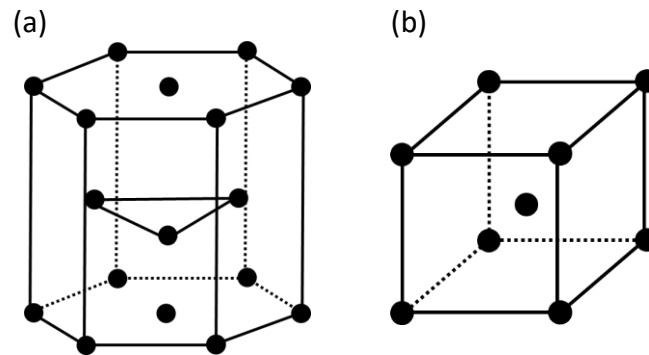


Figure 2.2 – Schematic outlining the (a) HCP crystal structure and (b) the BCC crystal structure found in α and β -titanium respectively.

Alloying additions are used to stabilise one or both of the phases with the classification of the addition derived from its effect on the β -transus temperature. Additions such as Al, O, N and C, increase the β -transus temperature and so are classified as α -stabilizing, by contrast, β -stabilizing additions such as V, W, Mo and Nb suppress the transus [7]. Titanium alloys are grouped into five categories based on their microstructural composition, α , near- α , α/β , near- β or β alloys. Alloys composed primarily of the α phase (α and near- α alloys) retain up to approximately 5% of the β -phase at room temperature, due to the low (1-2%) β -stabilizing alloy addition [8]. Near- α alloys such as Ti-6Al-2Sn-4Zr-2Mo are chosen for their high-temperature strength, which allows them to be used in applications such as in the high pressure compressor at temperatures up to 540°C, too hot for alloys containing a higher concentration of β -stabilizing elements [6]. Increasing β -stabilizing alloying addition yields an α/β alloy. These alloys primarily consist of the α -phase at room temperature however up to 40% of the β -phase can be retained [8]. The increase in retained β gives improved strength due to the heat treatment options and desirable processing characteristics.

β -titanium alloys retain 100% of the β phase when quenched from above the β transus temperature. These alloys can have tensile strengths as high as 1380 MPa, and therefore offer the highest mechanical strength among the titanium-based alloys. The good response to heat treatment, due to increased β -stabiliser addition, facilitates this high strength [4]. β -alloys were used in the Lockheed SR-71 blackbird airframe where a high strength to weight ratio and thermal stability were needed due to the high skin temperatures at supersonic speeds. Today, β -alloys are used as a spring material and as a material for aircraft landing gear [9], however, their poor strength at high temperature compared to near- α and α/β alloys means they are less suitable for use in aeroengines.

In aeroengine applications, Ti-6Al-4V, an α/β alloy, is the most widely used, it accounts for 60% of the total alloy production [4] and constitutes approximately 1/3 of the weight of a typical aeroengine [9]. The chemical composition limits for this alloy are outlined in Table 2.1, with Al (α -stabilizing) and V (β -stabilizing) the largest additions. These alloying additions raise the β -transus temperature to approximately 996 °C [9]. The microstructure consists of primary α -phase grains in addition to residual grain boundary β , as shown in Figure 2.3. Ti-6Al-4V is used for static and rotating components in the colder front section of the aeroengine with typical applications being fan blades as well as disks and blades in the compressor [3]. In addition to Ti-6Al-4V, other α/β alloys such as Ti-6Al-2Sn-4Zr-6Mo are used in aerospace applications [10], however, they possess a lower thermal capability, despite improved mechanical properties, so have not replaced Ti-6Al-4V.

Table 2.1 - Minimum and maximum alloy additions in Ti-6Al-4V [11].

	Al	V	Fe	O	C	H	N	Y
Min	5.5	3.5	0.3	0.14	0.08	0.01	0.03	50ppm
Max	6.75	4.5	-	0.23	-	-	-	-

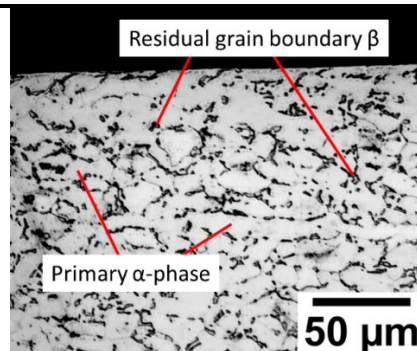


Figure 2.3 – The microstructure of Ti-6Al-4V.

2.1.2 Nickel-based superalloys

Nickel-based superalloys (herein termed superalloys) are alloys in which nickel is the most significant element by weight percentage. In comparison to titanium alloys, superalloys contain a significant proportion of alloying addition, typically 20–50% [12]. Alloying elements can provide solid-solution strengthening, precipitation strengthening or corrosion resistance. Multiple alloy additions are used to balance these three beneficial effects to tailor the alloy to the desired application. Although pure nickel is ferromagnetic, alloying additions result in superalloys being paramagnetic, similar to titanium-alloys. Paramagnetic materials are only weakly attracted to magnets, this is due to unpaired electrons in the outer shell of atoms [5], which means, in the absence of a magnetic field, paramagnetic materials lose their magnetisation, unlike ferromagnetic materials.

The aerospace industry accounts for around 80% of superalloy usage, largely due to its adoption in the hot end of aero-engines [13]. Up to 50% of the weight of an aero-engine is superalloy, this is due to its greater density of 8.2 g/cm³ compared to that of titanium, 4.5 g/cm³ [14]. The use of superalloys in aeroengines is primarily due to their high-temperature strength, in particular, their resistance to creep but also their corrosion resistance and toughness. Superalloys can operate at temperatures within 10% of their melting points [14] and are therefore used for turbine blades and disks, as well as parts of the combustor and exhaust system where temperatures as high as 1500 K can be experienced [3, 15]. Operating temperatures for uncoated and uncooled superalloy components are only as high as 1150°C [16], therefore additional measures are required to facilitate their use at the highest operating temperatures. Through intelligent use of ceramic coatings and complex cooling of parts, both internally and externally, the operating ceiling can be extended beyond the melting point of the superalloy [17].

The microstructure of superalloys consists of two primary phases, γ and γ' . The face-centred cubic (FCC) γ -phase typically contains many of the alloy additions used in superalloys, such as Co, Cr, Mo and Rh [18] and acts as the matrix in which the other phases reside. The γ' , is a FCC Ni₃(Al,Ti) precipitate phase which is coherent with the γ matrix. Precipitation of γ' is used to improve the strength of the superalloys through precipitation hardening [7]. The precipitate phase can also exist as the body-centred tetragonal (BCT) γ'' -phase in nickel-iron superalloys and superalloys with high Nb alloy addition. Here, the unit cell of the FCC crystal structure is stretched along one axis, to form a square-based rectangular prism. The γ'' -phase is also coherent with the FCC γ matrix and, when present, is uniformly distributed within matrix. Metal

carbides, a secondary phase, form at the grain boundaries and are used to pin grain boundaries to reduce creep by restricting grain boundary sliding [5]. Solid solution strengthening, in which atoms of elements such as Mo replace other atoms in the γ phase, is also used to improve the mechanical performance of superalloys. The resultant change in the size of the particles in the lattice introduces internal stresses which make dislocation movement more difficult [19].

IN-718 is one of the most used and studied superalloys [19-22], its approximate chemical composition by wt% is 52.50 Ni–18.50 Fe–19.00 Cr–5.10 Nb–3.00 Mo–0.50 Al–1.01 Ti–0.08 C [20]. IN-718 is a nickel-iron superalloy and its strength is achieved through precipitation of the γ'' (Ni_3Nb) phase rather than the γ' $\text{Ni}_3(\text{Al,Ti})$ phase. A secondary, orthorhombic, δ -phase, which is not coherent in the matrix, precipitates at the grain boundaries in this alloy, as shown in Figure 2.4. The relative amount of γ'' and γ' is adjusted to tailor the microstructure to provide properties that meet the thermomechanical requirements for a particular application within the aeroengine. In turbine disks, for example, the ideal microstructure of 20% γ'' and 5% γ' can be achieved through careful heat treatment [19, 23].

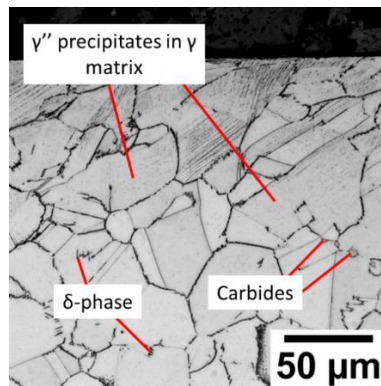


Figure 2.4 – The microstructure of IN-718.

2.1.3 Steels

Steels are ferrous alloys, i.e. iron is the base element, with the distinction for steels being that the alloying system is primarily iron-carbon [5]. Steels are the most widely produced alloys for engineering applications due to their low production and processing costs compared to materials such as titanium-alloys [4]. In aerospace applications, stainless steels and ultra-high-strength steels are the most extensively used; these are low and high-alloyed materials respectively.

There are two primary phases present in steels, ferrite (α -Fe), the low-temperature BCC iron phase, is typically the most machinable [5]. Austenite (γ -Fe) is a high-temperature FCC phase formed when ferrite

is heated above the austenitisation temperature. Through alloying and heat treatments, the austenite phase can be made stable at room temperature. Austenitic steels are paramagnetic, unlike ferritic steels, and are typically more ductile creep resistant [24]. Similar to titanium, alloy additions to steel can be classified based on their stabilizing influence on the two primary phases. Austenite formers such as Mn and C stabilise austenite whereas ferrite formers e.g. Cr, Mo and Si have the opposite effect [25].

Heat-treatments are used to modify the microstructure of steels to meet the needs of the application, with the cooling rate having a significant impact on the resultant microstructure [5]. A slow cooling rate from above the austenitisation temperature allows a proportion of the carbon atoms in the austenitic phase to diffuse out and form cementite (Fe_3C), with a concurrent transformation of the austenite to the low-temperature ferrite phase [26]. This results in an alloy that is more readily formable. In contrast, rapid cooling, faster than the critical cooling rate, results in BCT martensite, as shown in the time-temperature-transformation graph in Figure 2.5.

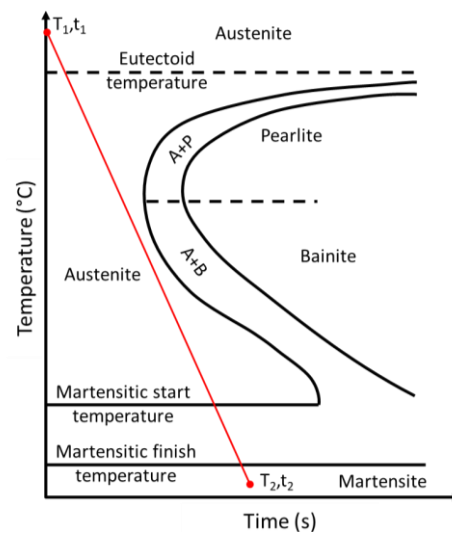


Figure 2.5 – A typical time-temperature-transformation graph showing a cooling rate (red line) which results in a fully martensitic microstructure. After [5].

Martensite is a high strength but brittle phase found in many aerospace steels [6]. Alloying additions to the steel can reduce the critical cooling rate, thus facilitating a wider processing window for forming martensite without the presence of other phases such as ferrite, pearlite or cementite. Martensite can be annealed through heat treatment, to improve ductility and fracture toughness, by allowing carbon atoms to diffuse out to form cementite and ferrite [26]. In a typical aerospace steel heat treatment, the alloy is solution treated above the austenitisation temperature so that the ferrite phase is taken into solution [27].

The alloy is then rapidly quenched to give a supersaturated solid solution of carbon in martensite. To improve the ductility the steel is then tempered below the austenitisation temperature to encourage precipitation of cementite, which provides precipitation strengthening.

In aeroengine applications, steels are used as a shaft material due to the very high strengths achievable in these alloys through thermomechanical processing, as well as the high stiffness and good fatigue properties [3]. Super chrome-molybdenum-vanadium (SCMV) steel is a tempered, martensitic steel used as a low-pressure shaft material in gas turbine engines due to its high strength to volume ratio and its strength at high temperatures. SCMV is an evolution of BS 3S 132, created as a result of the desire to increase the efficiency of aeroengines [28], the chemical composition is given in Table 2.2. To improve efficiency, the weight of the aeroengine must be reduced or the engine bypass ratio, the ratio of air passing around the core to the air passing through the core, increased. To achieve a high bypass ratio without increasing the size of the outer casing of the engine, which will result in increased momentum drag, the size of the engine core must be reduced and this requires more compact shaft design. SCMV was therefore designed as a material that facilitates this change by improving the yield and ultimate tensile strength as well as fatigue resistance, without significant increases in density over older alloys thus allowing smaller shafts of the same strength to be made [29].

Table 2.2 – Composition of SCMV, after [28].

Element	C	Cr	Mo	Va	Si	Mg	Fe
wt%	0.35-0.43	3.0-3.35	0.80-1.10	0.15-0.25	0.10-0.35	0.40-0.70	bal

Table 2.3 – Mechanical properties of SCMV, from MSRR6119 [30].

UTS (MPa)	$\sigma_{0.2\%}$ (MPa)	Hardness (HV)	Density (g/cm ³)
1470-1670	1220-1370	450-520	7.8

It should be noted that martensite is not just a term used for a particular steel microstructure, it is also a general term used for the microstructure that results from a diffusionless transformation, e.g. the martensitic microstructure that can form during quenching of titanium.

Due to the low carbon content of SCMV, the martensite has a lath structure, which means the martensite forms in long thin plates that are grouped into blocks within the prior austenite grains, as shown in Figure 2.6. Tempering of a SCMV pre-form results in the precipitation of carbides. It is the further

growth of these carbides at temperatures above 560°C that results in the deterioration of the mechanical properties of the SCMVs and sets the limit on its high-temperature capability as a shaft material.

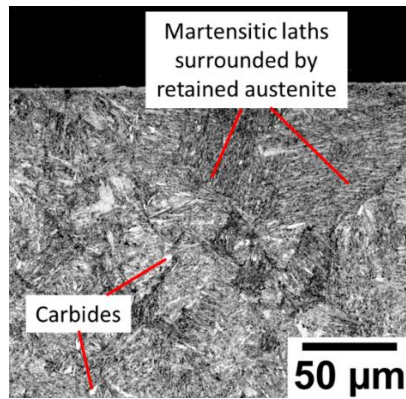


Figure 2.6 – The microstructure of SCMVs.

2.2 Deformation of materials

For materials to be processed into useful geometries for aeroengine applications, they must typically be deformed, whether this is through forging, forming, machining or other processing methods. Materials can deform elastically or plastically. During elastic deformation the shape of the material is recovered when the stress is removed, conversely, plastic deformation is irreversible and occurs when the elastic limit of the material is exceeded. Plastic deformation can be accommodated by deformation slip or twinning. In deformation slip, regions of material move relative to one another along slip planes, as shown in Figure 2.7 (a), when the shear stress exceeds a critical value, termed the critically resolved shear stress [5]. Slip occurs on close-packed planes (i.e. planes with a greater density of atoms) in close-packed directions (i.e. directions with a large number of atoms per unit length) which together comprise a slip system. The von Mises criterion states that five independent slip systems are required for deformation to occur [31]. This is satisfied in a FCC and BCC crystal, however, in a HCP crystal there are only 4 independent slip systems so activation of a twinning mode is required to accommodate deformation [32]. Twinning is the movement of atoms within a crystal such that the orientation of atoms in the twinned state is the mirror image of the prior orientation, as shown schematically in Figure 2.7 (b). Higher strain rates and lower temperatures result in increased frequency of deformation through one or more of the twinning modes [33, 34].

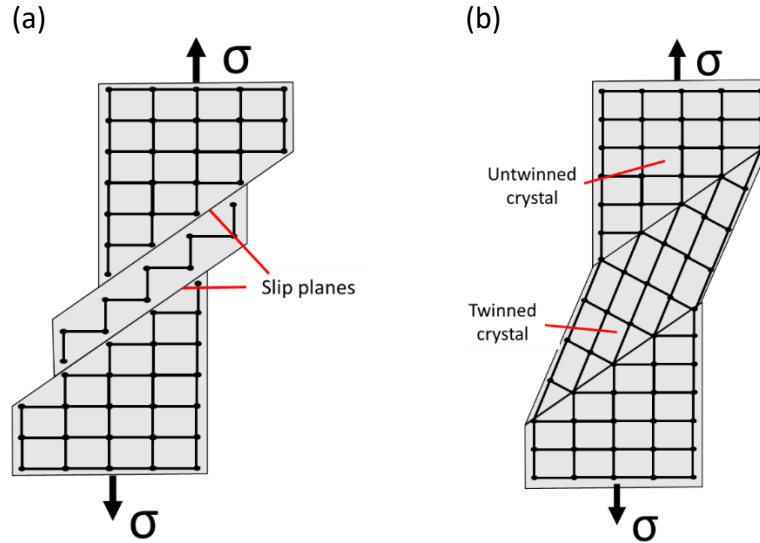


Figure 2.7 – Schematics outlining the (a) slip and (b) twinning deformation mechanisms. After [5].

The resultant crystallographic texture of a material following plastic deformation depends on the slip or twinning modes that facilitated the deformation. It has been shown [35, 36], for example, that the HCP crystal typically re-orientates under compression to produce a basal $\{0002\}$ texture, due to activation of the $\{10\bar{1}2\}$ tensile twinning mode and this involves an 85° rotation of the crystal structure. The prior crystallographic orientation of the material dictates which specific systems drive the re-orientation of a particular grain, as highlighted by Bridier et al. [37] and the resulting texture is a consequence of slip or twinning modes that accommodate the deformation.

In this study, the slip and twinning systems in titanium are referenced when discussing deformation during machining. As such, the HCP crystal structure, slip modes and twinning modes are outlined in Figure 2.8 and Table 2.4. Twinning systems are defined by a plane, which is shared by both the un-twinned and twinned region of the crystal lattice, as well as the shear direction. However, to identify a twin, it is also necessary to know its relationship to the parent orientation and this is defined by the axis and angle of rotation, as outlined in Figure 2.9 for the $\{10\bar{1}2\}$ twin. As can be seen from Table 2.4, a number of twins can be defined as either compressive or tensile twins. This description refers to the extension or compression of the c-axis that is accommodated by the twin, rather than the applied stress state.

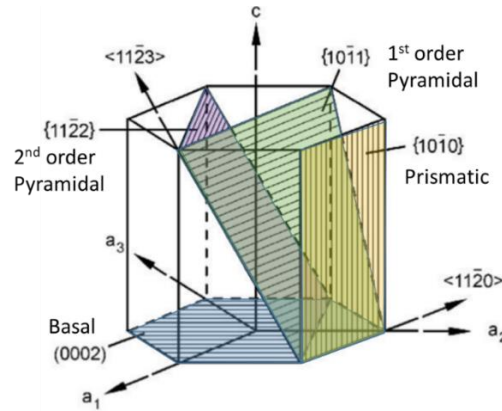


Figure 2.8 – HCP crystallographic structure with available slip systems (combinations of slip planes and slip directions) highlighted. After [32].

Table 2.4 – Twinning systems in HCP titanium. After [38].

Twinning system	Rotation axis	Rotation angle	Twin type
$\{10\bar{1}2\}\{\bar{1}011\}$	$\langle 2\bar{1}\bar{1}0 \rangle$	84.78°	Tension
$\{10\bar{1}1\}\{\bar{1}012\}$	$\langle 2\bar{1}\bar{1}0 \rangle$	57.42°	Compression
$\{11\bar{2}2\}\{\bar{1}\bar{1}23\}$	$\langle 10\bar{1}0 \rangle$	64.62°	Compression
$\{11\bar{2}1\}\{\bar{1}\bar{1}26\}$	$\langle 10\bar{1}0 \rangle$	35.1°	Tension
$\{11\bar{2}4\}\{\bar{2}\bar{2}43\}$	$\langle 10\bar{1}0 \rangle$	76.66°	-
$\{11\bar{2}3\}\{\bar{1}\bar{1}22\}$	$\langle 10\bar{1}0 \rangle$	86.98°	-

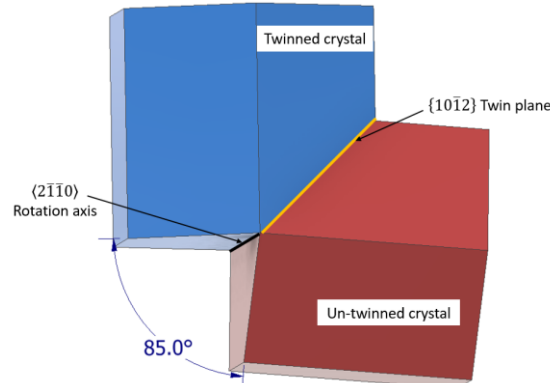


Figure 2.9 – Schematic showing the $\{10\bar{1}2\}$ twinning system in α -titanium.

The crystallographic texture of a material can significantly influence its mechanical properties. This is readily observable for titanium due to the mechanical anisotropy of the HCP α -phase. It has been shown [39] that the elastic modulus and strength are greatest when loading parallel to the HCP c-axis in textured titanium sheet. This dependence of elastic modulus on crystal orientation is highlighted in Figure 2.10. It can be seen Elastic modulus is nearly 50% greater when loading parallel to the c-axis direction compared to perpendicular loading. This highlights that the deformation mechanism and resultant texture are important for understanding the mechanical properties of a material, particularly in titanium.

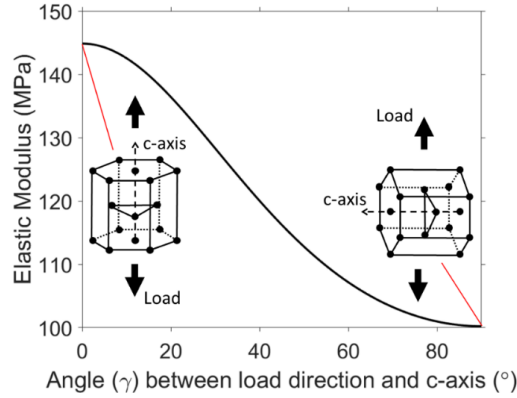


Figure 2.10 – A plot outlining the dependence of elastic modulus on the angle between the loading direction and the c-axis in HCP α -titanium. After [40].

2.3 Machining of aeroengine materials

Machining is a subtractive manufacturing process wherein a component is produced by the removal of material through the interaction of a cutting tool with a workpiece. Mechanical machining uses the principles of metal cutting in which work materials are purposefully fractured to produce desired parts [41], therefore, plastic deformation is fundamental to machining. The manufacturing of almost all components in an aeroengine, from turbine blades to compressor discs, requires a machining step in order to achieve the required shape and surface finish. The principle mechanical machining operations include turning (used to shape cylindrical billets), milling (used to create surfaces of varying complexity), grinding (machining with abrasive grits) and drilling (the primary hole generation operation) [42]. This study focuses on turning and milling operations due to their widespread use in aeroengine manufacturing and the convenience of the cylindrical and flat surfaces that can be produced via turning and milling, respectively, which lend themselves more easily to non-destructive testing compared to holes, due to their easier surface access.

2.3.1 Mechanics of chip formation

To discuss the mechanics of turning and milling operations, the mechanics of chip formation must first be introduced. The basic theory of chip formation states a chip is formed through mechanical shearing of the workpiece in a plane that extends from the cutting edge to the surface of the workpiece in front of the tool [43]. The angle of this shear plane to the cutting direction is called the shear angle, ϕ . Merchant [43] showed that the shear plane angle could be related to the tool rake angle (α) and the ratio of the uncut chip thickness (t_1) to the cut chip thickness (t_2) through Eq. 2.1;

$$\tan(\phi) = \frac{(t_1/t_2)\cos(\alpha)}{1 - (t_1/t_2)\sin(\alpha)} \quad \text{Eq. 2.1}$$

The formation of a continuous chip during orthogonal cutting, the simplest form of cutting, in which the cutting edge is perpendicular to the direction of cutting, is shown schematically in Figure 2.11. The relief angle is that between the flank face of the tool and the machined surface. When this angle is 0°, the flank face rubs on the workpiece surface. Merchant's model assumes orthogonal cutting [43], allowing 2-dimensional analysis [42], which significantly reduces complexity. In practice, however, orthogonal cutting is not encountered during typical aeroengine machining operations.

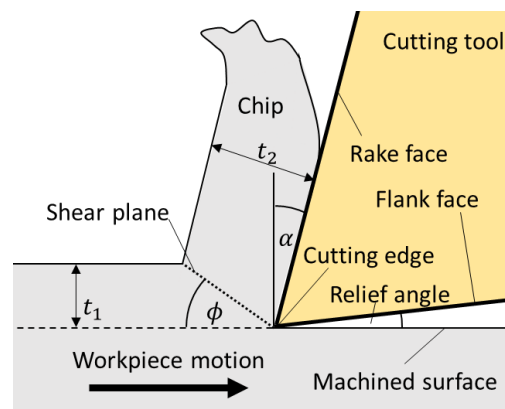


Figure 2.11 - Schematic of chip formation in orthogonal cutting showing the shear plane angle ϕ , the rake angle α and the ratio of chip thicknesses r (t_1/t_2) after [43].

There are 4 main types of chips that can arise from plastic deformation in the shear plane [42]. These are continuous chips, continuous chips with a built-up edge, discontinuous chips and segmented (shear-localised) chips. Aeroengine materials typically form segmented chips due to their low thermal conductivity and high strength [44]. A segmented chip from an aeroengine alloy is shown in Figure 2.12. Due to the low workpiece thermal conductivity, the heat energy which arises from plastic deformation in the primary shear zone does not dissipate quickly into the surrounding material and so is confined to a narrow region termed the shear band [45], this leads to thermal softening. As the shear band is softer, a larger amount of strain occurs there, leading to further thermomechanical deformation and consequently further thermal softening. This cyclic nature of this process as the tool advances across the workpiece, leads to the saw-tooth profile of a segmented chip.

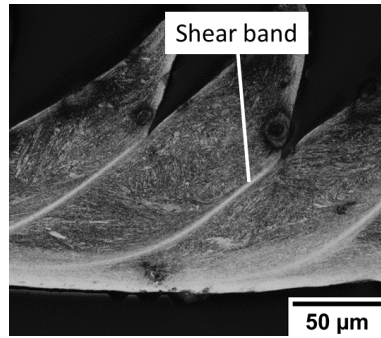


Figure 2.12 - A SCMT segmented chip with a shear band highlighted.

2.3.2 Turning

In turning, a stationary tool is used to remove material from a rotating workpiece [46] as shown in Figure 2.13. Material is removed perpendicular to the axis of workpiece rotation so the machined surface is concentric with the axis of rotation [47]. Turning is carried on a lathe using a single-point cutting tool, with the workpiece held in the chuck at one end and a tailstock at the other, if the part has a large length to diameter ratio. In aeroengine manufacturing, turning is used in the production of many parts, including turbine disks [13], due to the rotationally symmetric nature of these parts.

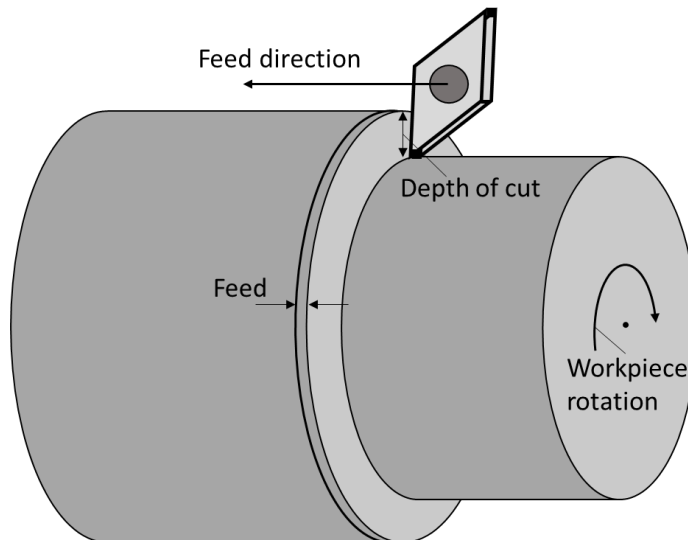


Figure 2.13 – A schematic of a typical turning operation.

The controllable parameters in turning are, cutting speed (V_c), depth of cut (w) and feed (f_r) [42]. The cutting speed is the product of the rotational speed of the lathe spindle and the radius of the workpiece. Feed is the distance moved by the cutting tool along the axis of the workpiece during one rotation. The depth of cut is the thickness of material removed from the workpiece. These parameters are indicated schematically in Figure 2.13.

To analyse the mechanics of orthogonal cutting, Merchant [43] considered the chip as a separate rigid body. The chip is supported under the balance of two primary equal and opposite resultant forces as shown in Figure 2.14. These are

1. The force of the tool on the tool/chip interface side (R')
2. The force of the workpiece on the underside of the chip (R)

R' can be considered a frictional force along the tool rake face (F_μ) and a normal force (F_R) perpendicular to this. Similarly, R can be decomposed into a shear plane component (F_s) and a force normal to the shear plane (F_N). When recording forces using a dynamometer during orthogonal cutting, it is not possible to measure these forces, instead, the cutting (F_c) and thrust (F_t) forces are recorded. The geometrical relationship between these forces is visible in Figure 2.14. The fundamental chip formation forces can be determined from F_c and F_t if the shear plane angle and tool rake angle are known.

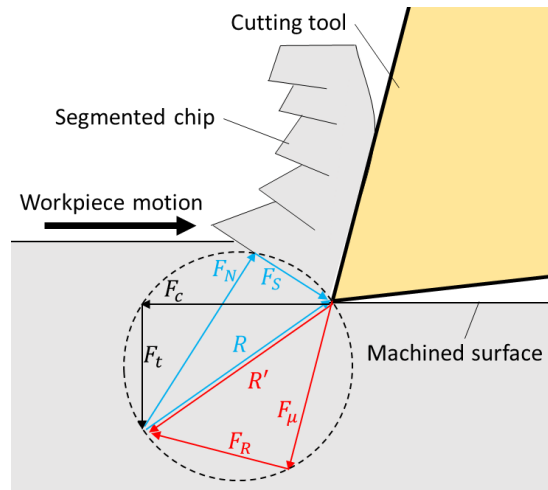


Figure 2.14 – Schematic of the forces involved in chip formation, after [43].

Since the early pioneers developed the basic theory for the mechanics of metal cutting, many authors have covered the topic in their books [41, 42]. However, an in-depth review of more advanced cutting models is beyond the scope of this literature review.

2.3.3 Milling

Milling is a machining operation that uses single and multi-point tools [47], the main difference to turning is that, in milling, it is the tool which rotates. There are two main types of milling, defined by the orientation of the tool rotation axis relative to the surface being machined. In horizontal milling, the tool rotation axis lies parallel to the work surface, whereas in vertical milling the tool rotation axis is

perpendicular [42]. In aerospace applications, milling can be used to form a variety of surfaces, ranging from flat shoulders to varying geometry aerofoils [13]. The complex shapes seen in blades and blisks (bladed disks) can only be generated through multi-axis milling where, as well as the conventional x, y and z movement of the workpiece or tool, the machining centre allows for workpiece rotation in one or more axes.

The controllable parameters during a milling operation are the feed f_t , the axial engagement a_e and the radial engagement a_p [42], as shown schematically in Figure 2.15. The feed in milling is usually given in feed per tooth for multi-point cutting or feed per tool revolution for single-point milling.

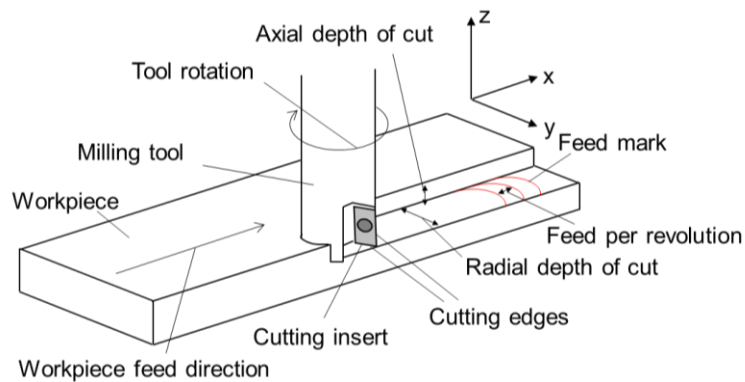


Figure 2.15 – A schematic of a vertical milling operation.

Two tool rotation conventions are used in milling. In climb-milling (also called down-milling), the tool rotates so that the cutting edge moves in the same direction to the workpiece feed as the chip is formed, as shown in Figure 2.16 (a); conversely, in conventional-milling (also called up-milling), cutting edge motion is reversed, as shown in Figure 2.16 (b). Conventional-milling was adopted in the early days of machining, to reduce the backlash of the workpiece when climb-milling [47]. Backlash occurs due to play between the screw thread and the nut that moves the machine bed or tool, this leads to a delay between turning the screw thread and movement of the corresponding axis. Climb-milling is preferred when backlash is not an issue as the cutting forces hold the workpiece on the machine bed and thus reduce vibrations resulting in an improved surface finish. Additionally, a thick-to-thin chip is formed in climb-milling and this means higher feeds can be tolerated. [48].

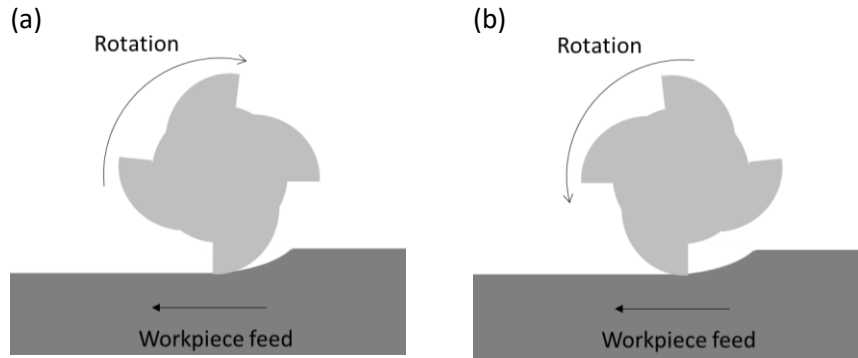


Figure 2.16 - Schematic of the two milling rotation conventions (a) climb milling and (b) conventional milling. The shaded regions show the material that is removed with each pass of a cutting edge and it shows the thick-to-thin chip seen in down milling, after [47].

Unlike turning, milling typically cannot be represented by an orthogonal cutting model. This is because the curved path of the cutting edge over the workpiece means that the chip thickness varies throughout tool rotation and the cutting edge is not normal to the chip flow, this is termed oblique cutting. The simplest form of milling is orthogonal milling, which utilises a cutting edge that is perpendicular to the chip flow and larger than the workpiece thickness. It should be noted that the chip thickness still varies over the period of tool engagement during orthogonal milling. As with turning, a review of cutting models for milling is beyond the scope of this study however further information can be found from models for oblique cutting [49].

Similar to turning, it is not possible to measure the forces in the shear zone during chip formation. Instead, x, y and z forces can be measured using a machine-bed force dynamometer and these can be converted from a workpiece frame-of-reference to a cutting tool frame-of reference using geometrical relationships. This conversion allows for a simpler understanding of the cutting forces as they are broken down into components parallel to the cutting edge and perpendicular to the cutting edge. These forces are dependent only on the instantaneous tool rotation angle and, for small uncut chip thicknesses, the ploughing effect [50]. The ploughing effect arises when machining with a tool that has a cutting edge radius which is of similar size or larger than the uncut chip thickness, this results in the deformation of the workpiece without chip formation.

2.3.4 Machinability of aeroengine materials

Machinability is a measure of how easy a metal is to cut; this encompasses the resultant cutting forces and tool wear as well as the cutting speeds needed to carry out useful machining [51]. The three common aero-engine materials are all considered difficult-to-machine, i.e. they result in high cutting forces when

machined, generate high levels of tool wear and necessitate the use of lower cutting speeds compared to a more machinable metal such as aluminium [52]. This difficulty, encountered when machining titanium, nickel and some steel alloys, is due to their high strength, particularly strength at high temperature, as well as low thermal conductivity and a propensity for workpiece-tool adhesion, which leads to built-up edge formation. High strength alloys give rise to larger forces when they are machined at the same cutting speeds as a more machinable alloy. This is because an increased resistance to deformation dictates that more energy is required to form a chip. The low thermal conductivity of difficult-to-machine alloys causes localisation of heat in the near-surface of the workpiece, similar to the localisation within the shear band of a segmented chip. The high temperatures that result from this heat accumulation at the workpiece surface, in addition to the mechanical deformation caused by the tool, can result in widespread surface damage [53]. To mitigate this damage, the primary solution is to reduce the cutting speeds, which decreases the cutting forces and the rate of heat generation. As a consequence, difficult-to-machine alloys are machined at cutting speeds as much as 10 times lower than more machinable alloys [54], as highlighted in Figure 2.17.

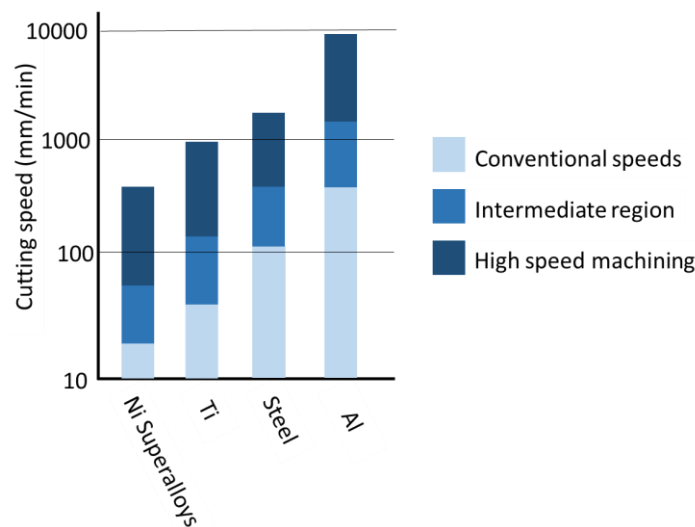


Figure 2.17 – A plot of the conventional, intermediate and high-speed machining cutting speeds for difficult-to-machine aeroengine alloys compared to aluminium. After [54].

In addition to the properties shared by all difficult to machine materials, titanium alloys and nickel superalloys suffer from additional complexities. Titanium alloys have a propensity for built-up edge formation and are highly reactive with all tool materials [55], which leads to accelerated wear rates particularly when oxide ceramics are used [52]. Nickel superalloys suffer from significant work hardening of the γ -matrix during machining [56]. Work hardening during the first pass of the cutting tool results in a

harder material, which is then faced by the cutting tool during successive passes leading to increased notch wear rates. Additionally, metal carbides, which form at the grain boundaries in polycrystalline nickel superalloys, can result in increased abrasive wear rates [56].

2.4 Surface integrity

The difficult-to-machine nature of aeroengine alloys increases their susceptibility to surface and near-surface damage under the thermomechanical loads imparted on the workpiece by machining. The altered condition of a surface post-machining is defined as its surface integrity (SI) [57] and it is widely acknowledged that SI of a machined component has a significant effect on its performance, reliability and fatigue life [1, 58]. In this context, the surface inspection of parts used in critical applications is a vital step in the validation of the manufacturing process, particularly for aeroengine components, which are more susceptible to machining damage due to their material properties.

A large number of anomalous surface and near-surface features can arise due to machining, these all fall within the remit of SI assessment. The scope of anomalous SI features is highlighted in Table 2.5, it can be seen that all of the features can be detrimental to component performance in-service. For this reason, it is crucial to assess the SI of machined aeroengine components to prevent parts with anomalous features above critical size thresholds entering service.

Table 2.5 – Surface integrity features and their characteristics.

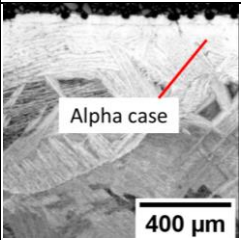
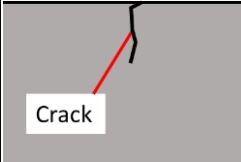
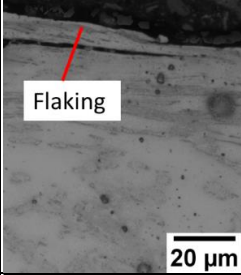
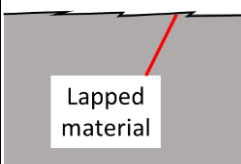
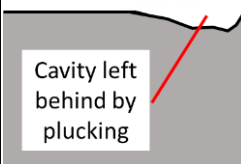
Anomalous feature	Characteristics	Example image(s)
Alpha-case	Alpha-case is a temperature-dependent oxidation of the near-surface seen only in titanium alloys. This feature can lead to an embrittled surface that is prone to crack initiation and propagation [59].	 Alpha case 400 μm After [60].
Cracks	Cracks are fissures in a continuous material that significantly reduce the fatigue life [61]. Most features under the remit of surface integrity are detrimental to a component as they result in, or enhance, crack formation.	 Crack
Flaking	The local separation of a layer of material during machining. The surface that is left behind can act as a site for crack initiation due to enhanced roughness [62].	 Flaking 20 μm
Foreign material	The deposition of non-parent material into the machined surface, this can lead to stress-raising and a reduction in fatigue life through enhanced crack initiation [61].	 Embedded tool tip
Laps	Laps are workpiece material that has been folded back on itself during machining without complete separation. A lap can act as a site for crack initiation if it enhances the surface roughness [62].	 Lapped material
Plucking	Plucking arises when a section of material is removed from the machined surface during cutting. This can occur through adhesion of the material to the tool flank face or built-up edge. The crater left behind can act as a site for crack initiation due to enhanced roughness [62].	 Cavity left behind by plucking
Residual stresses	Residual stresses arise from the thermomechanical loads which are imparted on to a surface and not recovered post-machining. Tensile residual stresses can reduce the fatigue life of a component [63].	N/A

Table 2.5 continued

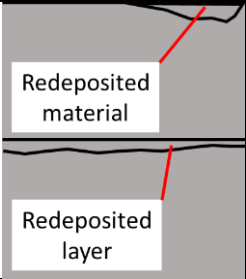
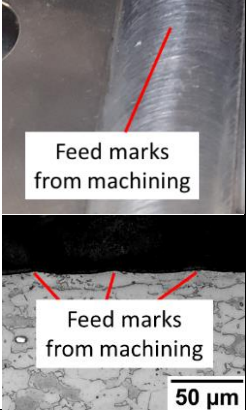
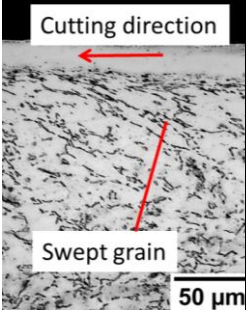
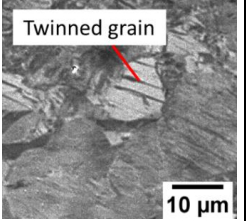
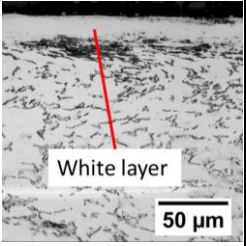
Redeposited material / Redeposited layer	Redeposited material is that which has been machined from the workpiece but has then been reintroduced to the surface by, for example, passing under the tool. The redeposited material is liable to separate from the machined surface [11].	 Redeposited material Redeposited layer
Strain/work hardening, Re-hardening, Softening	A change in workpiece near-surface hardness. Typically associated with the presence of other SI features e.g. a white layer [64].	N/A
Surface roughness	The deviation of a surface from flatness including feed marks, scores and scratches. High surface roughness can decrease the fatigue life of a component [62].	 Feed marks from machining Feed marks from machining 50 μm
Swept grain	A layer beneath the machined surface in which the grain structure has been distorted in the direction of machining by the action of the cutting tool. Swept grain can lead to a reduction in fatigue life [1], large residual stresses [65] and work hardening [66].	 Cutting direction Swept grain 50 μm
Twinning	Re-orientation of the crystal lattice to facilitate deformation via slip during machining. Twins have been shown to act as sites for fatigue damage [67].	 Twinned grain 10 μm
White layer	A layer immediately beneath the machined surface in which the grain structure cannot be resolved at a magnification which facilitates observation of the bulk microstructure. White layer formation can reduce fatigue life [58], lead to an embrittled surface [68], cause large residual stresses [69] and result in surface hardening [70].	 White layer 50 μm

Table 2.6 outlines the ANSI [71] recommended SI assessment methods alongside the SI features that are identifiable from each method. It can be seen that features such as white layers and swept grain are only identifiable when viewing the machined surface in the cross-section. This is why SI assessment is largely a destructive inspection process.

Table 2.6 – The recommended surface integrity data set for assessing surface integrity [71] and the anomalous feature or information that can be identified or obtained from the data.

Data acquired	Information obtained
Surface topography – roughness measurement, topographical imaging.	Surface contacting methods can be used to measure roughness (Ra, Rz) but non-contacting optical profilometers can also identify surface imperfections such as laps, flaking and plucking.
Macrostructure images on the surface (low magnification <10x) – chemical etching, dye-penetrant tests.	Laps, flaking, plucking, redeposited material, foreign material, cracks.
Cross-sectional microstructure images (high magnification up to 1000x) – polished surfaces, chemically etched.	White layer, swept grain, alpha-case, laps, flaking, plucking, redeposited material, cracks, foreign material.
Cross-sectional microhardness or nanohardness measurements.	Work-hardening or softening of the near-surface region.
Profile of residual stress with depth.	Residual stress in the machined surface compared to the bulk.
Fatigue tests of samples.	Low and high cycle fatigue life.

Of all the SI features outlined in Table 2.5, it is perhaps the metallurgical anomaly, white layer, which is the most feared by materials engineers. This is primarily due to the negative effect its presence can have on fatigue life [1, 58, 72]. In addition, there is greater uncertainty, compared to other SI features, around the formation mechanisms and imposed machining conditions that generate this anomalous feature in different materials. White layers are also one of the most difficult features to inspect, as detection requires cut-ups of the machined surface, which is both costly and time-consuming. For these reasons and through

communication with the industrial sponsors of this study, white layers were identified as the anomalous feature of interest.

2.5 Summary

In this chapter, the three key aeroengine material groups have been introduced and the physical properties and metallurgy of these alloys have been highlighted. Specific alloy examples were presented and the experimental work in this study will focus on these alloys. A portion of this study will concentrate on understanding the formation mechanism of a SI feature in titanium, so the deformation mechanics of titanium alloys were discussed.

Also within this section, the mechanics of chip formation were outlined, to aid understanding of machining processes. Following this, two of the primary machining methods employed in aeroengine manufacturing, turning and milling, were introduced, as these are the operations employed in this study. The difficulties faced when machining aeroengine alloys were then highlighted and the concept of surface integrity was introduced. The key surface integrity features and the typical methods for their assessment were outlined and the importance of machining-induced white layers was highlighted. The next chapter will review the current literature in the field of white layer characterisation.

3 The key properties and formation mechanisms of machining-induced white layers

A thorough understanding of the key mechanical and metallurgical properties of machining-induced white layers is necessary to be able to identify suitable non-destructive methods for detecting this feature. Therefore, this chapter reviews the current state-of-the-art and published literature in the field of white layer assessment using conventional destructive assessment methods to enable a subsequent assessment of viable non-destructive testing methods. The formation mechanisms of white layers are also discussed, due to their effect on the resultant properties.

3.1 The fundamentals of white layers

White layers in aeroengine materials have typically been characterised as a region of embrittled material [68] which possesses an ultra-fine grain size [73], high hardness [64] and extremes of residual stress [69]. In industry, the white layer is often defined as a region at the machined surface in which the microstructure cannot be distinguished, in a cross-sectional micrograph, at a magnification which allows the bulk microstructure to be easily resolved, as demonstrated in Figure 3.1. The white appearance of this region has traditionally been attributed to a decreased etching rate, relative to the bulk material [74, 75], which means the grain structure is not revealed in the white layer. More recently, it has been proposed that the scattering of the incident light used to illuminate the sample during subsequent optical microscopy inspection in the cross-section, may result in the appearance of the white layer [70]. Due to its comparatively large wavelength, white light is thought to be scattered from the small-diameter grains in the white layer, resulting in a featureless layer. White layers are typically 1-10 μm in thickness, although layers over 20 μm thick have been observed in extreme cases [64].

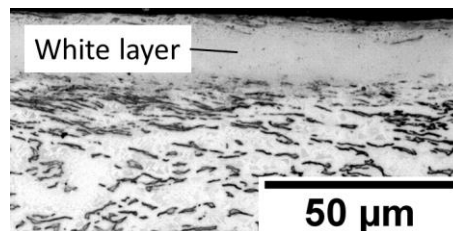


Figure 3.1 – A cross-sectional micrograph of a white layer at 500x magnification. The bulk microstructure can be resolved whereas the white layer appears amorphous.

It has been reported [1] that the presence of a 10 μm white layer can reduce the fatigue life of a drilled part by as much as 30 times, compared to a surface with no white layer. Even after shot-peening a drilled

surface with a white layer, in the same study, the fatigue life was still 10 times lower than the benchmark life. Additionally, it has been shown [58, 72] that the rolling contact fatigue life can be reduced by 80% or more when white layers between 7 μm and 14 μm in thickness are present in steels. This reduction is predicted to be due to reductions in both crack initiation life (75%) and crack propagation life (89 %) [72]. Herbert et al. [1] reported that tensile residual stress in the white layer can facilitate the initiation of fatigue cracks at the surface of the part during cyclic loading, and crucially the propagation rate is accelerated by the nanoscale grains in the white layer. This high propagation rate is a result of fatigue cracks at the surface merging with micro-cracks within the white layer.

White layers have been identified in turned [76], milled [64], drilled [77], broached [78] and wire electrical-discharge machined surfaces [79]. Additionally, this SI feature has also been reported outside of the field of machining, in worn rail tracks [80, 81], ballistic impacts [82] and high-velocity shaping and forming [83].

3.2 White layer formation mechanisms

Three primary mechanisms have been proposed for the formation of white layers, (a) phase transformation, (b) grain refinement through plastic deformation and (c) surface reaction [74]. Mechanisms (a) and (b) have been associated with machining, whilst mechanism (c) is believed to be connected with worn surfaces in oxygen or nitrogen-rich environments.

High temperatures and rapid cooling rates during machining can result in a phase transformation of the near-surface region of metallics [76]. Heat is generated during mechanical cutting operations due to severe plastic deformation (SPD) of the workpiece during chip formation and frictional contact conditions underneath the tool due to cutting tool-workpiece interaction. SPD occurs in the primary, secondary and tertiary deformation zones, as outlined in Figure 3.2, with the direct effect of the tertiary deformation zone on the machined surface immediately apparent. Guo et al. [84] showed, using particle image velocimetry, that the deformation effect of the primary shear zone extends into the workpiece such that material that does not form part of the chip is still deformed in a fan-shaped deformation zone in front of the tool. This has also been observed, using micro-grid methods whereby a proportion of the material that passed through the deformation zone ahead of the chip was still compressed underneath the cutting edge

[85]. It is, therefore, appropriate to consider that the primary shear zone, in addition to the tertiary shear zone situated at the tool and workpiece surface interface, may affect the workpiece surface integrity.

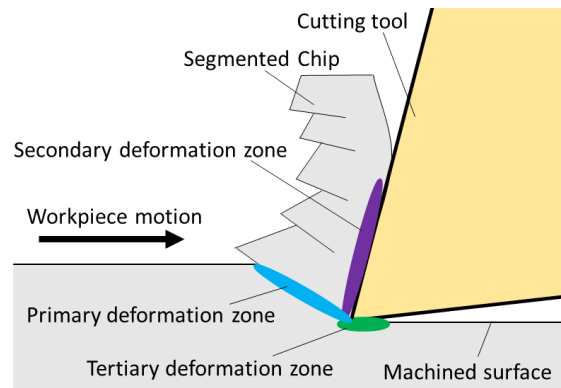


Figure 3.2. Schematic highlighting the primary, secondary and tertiary shear zones during cutting, after [86].

Heat is removed from the shear deformation zones via evacuation of the chip, through the application of coolant, via conduction into the tool and by conduction into the workpiece bulk [87]. The low thermal conductivity of aeroengine alloys such as titanium alloys, nickel superalloys and high strength steels, results in localisation of the heat in the near-surface of the workpiece. By extension, this leads to high temperatures and steep temperature gradients when machining these materials [88].

It has been reported [76, 89] that white layers can form as the result of a phase transformation mechanism in steel components. This indicates that the temperature at the machined surface can rise beyond the austenitic transformation point and a fully martensitic microstructure can be created by the imposed rapid cooling [89]. By contrast, when temperatures do not reach the austenitic transformation temperature, a white layer is formed via the SPD mechanism, as shown by Hosseini et al. [90]. In steels, researchers [70, 91] have identified a thermally softened layer, often termed a dark layer, beneath the hardened white layer. Dark layers have only been found beneath white layers formed from the phase transformation mechanism [92].

White layers formed via phase transformation possess a fine-grained microstructure that arises due to the rapid heating and quenching of material in the near-surface region [74]. Phase transformation occurs in two steps, initially, nucleation of the new phase occurs in the region of material undergoing transformation [93]. The nuclei of the new phases then grow, at the expense of the old phase until the microstructure is completely transformed. Following this, grains will continue to grow if held at sufficient temperatures. Due to the rapid heating and cooling under the high strain rates, short contact time and

coolant application experienced in machining, the new grains have limited time to grow after the phase transformation. This is in contrast to austenitisation heat treatment in steels, for example, where the material can be held for several hours at high temperatures [94], resulting in a large-grained microstructure. The reduced time for grain growth in the white layer results in a large number of small grains.

In the SPD mechanism, Griffiths [74] suggests that a shear deformation within the material adjacent to the machined surface occurs due to the action of the cutting tool. This action leads to fragmentation of the grains in the near-surface, which gives rise to the refined grain structure present in the white layer. Further to Griffiths' hypothesis, Poulachon et al. [89] proposed that white layer formation through grain refinement is a two-step process. Initially, dislocations pile-up within grains due to SPD, resulting in regions of high dislocation density. In turn, this facilitates the formation of new grain boundaries within the original grains. Several studies [64, 73] refer to this white layer formation mechanism as SPD with dynamic recovery and it has been observed in the machining of steels [76], nickel superalloys [77] and titanium alloys [95].

Recent research from Xu et al. [96] reports that the formation of a nano-grained structure in the surface of high-speed machined titanium occurs through both SPD and phase transformation. This suggests that the two mechanisms are not mutually exclusive and are simply two ends of a spectrum of white layer formation. At high temperatures phase transformation appears to dominate whilst at low temperatures SPD appears dominant. Under thermomechanical conditions in between, both mechanisms can contribute. Xu et al. [96] proposed that the grain-refined surface region formed due to continuous dynamic recrystallisation under high strain rate. Dislocation slip alongside $\{10\bar{1}1\}$ and $\{10\bar{1}2\}$ twinning were thought to facilitate the deformation in the surface layer. Xu et al. [96] also identified β precipitation under the strain and high temperatures experienced during high speed milling. It was proposed that these precipitates prevented grain growth in the recrystallised white layer grains by pinning them. The formation mechanism proposed by Xu et al. [96] is, therefore, a mixture of SPD and phase transformation, with the appearance β precipitates indicating that one mechanism is not always solely responsible for white layer formation. In the adiabatic shear band of a titanium chip, which can be considered to be a form of a white layer due to the physical similarities, Wan et al. [97] measured through x-ray diffraction that the α'' -phase

was present in the white layer, in contrast to the as-received material. This indicates that the temperatures during cutting were sufficient for an $\alpha \rightarrow \beta$ transformation followed by a $\beta \rightarrow \alpha''$ transformation under cooling. Thus if a white layer formed via phase transformation in titanium there should be evidence of α'' formation or an increased concentration of retained β -phase.

3.2.1 Grain recrystallisation

It is apparent from the reduced grain size of white layers relative to the bulk that recrystallisation is a key mechanism in their formation when phase transformations are not involved. Recrystallisation is the formation of new dislocation-free grains within a deformed grain [98]. If recrystallisation occurs during deformation then it is termed dynamic recrystallisation (DR), if it occurs after deformation, it is defined as static recrystallisation. As white layers are formed during machining rather than subsequent heat treatment, any grain refinement must be a consequence of DR.

Studies have shown there are two forms of DR, continuous (CDR) sometimes called in-situ or rotational DR and discontinuous (DDR) which is also called migrational DR. In DDR, the recrystallisation process occurs in distinct stages of nucleation and growth at prior grain boundaries [98] whereas, in CDR, the recrystallisation is thought to occur in a single step over the entire volume [99]. Derby [100] reported that both DDR and CDR can occur under conditions of large strain and high temperature, which are the conditions faced by the workpiece during machining. Meyers et al. [101] additionally showed that, at high strain rates, which are common in machining operations at high cutting speeds [102], CDR prevails over DDR due to the short time at high temperature.

The resulting texture from CDR is typically representative of the deformation i.e. the recrystallisation itself has little effect on the texture, whereas in DDR the deformation texture is weakened and so it could be said the texture is more representative of the recrystallisation [99]. Thus, the resultant crystallographic texture following SPD could be used to infer whether CDR or DDR took place. A deformation texture following grain refinement is evidence of CDR, whereas a recrystallisation texture is evidence of DDR.

3.3 The influence of machining parameters on white layer formation in aeroengine alloys

White layer formation is fundamentally dependent on the thermal and mechanical effects of machining on the workpiece material. These effects are governed by the interaction of the cutting tool and the workpiece material, which, in turn, is strongly influenced by the imposed cutting parameters. Early surface

integrity research [103] found that abusive cutting conditions, particularly the use of tools with high wear levels, results in the formation of a white layer during milling of AISI 4340 steel and Ti-6Al-6V-2Sn. It has since been shown that the tendency for the formation of a white layer when machining is also increased significantly by raising the cutting speed, using improper cooling strategies and through under-lubrication of the cutting operation [104, 105].

The friction between the cutting tool and the workpiece increases when machining with improper lubrication and a worn cutting edge, thereby increasing heat generation and shear deformation of the surface [106]. Increased cutting forces, in particular the thrust (compressive) force, have been associated with increasing white layer thickness when high levels of tool wear are present [75, 107]. Improper cooling can result in higher cutting temperatures as the rate of heat removal is lower than can be achieved with effective cooling [108]. High cutting speeds typically result in a higher rate of deformation in the machining shear zones [109] and hence a higher rate of mechanical work input to the workpiece. Consequently, there is a greater overall heat input to the machined surface [110]. This is more pronounced for materials with higher strain hardening exponents such as nickel superalloys, which have to be machined using low cutting speeds to prevent tool failure and workpiece damage [56]. Thus, during machining, there is a balance between thermal softening and strain hardening, which is material dependent [111].

In Ti-6Al-4V, it has been shown [112] that white layers are formed at the end of tool life, during turning, when the average flank wear is 0.2 mm or greater, due to enhanced plastic deformation of the workpiece. Puerta Velásquez et al. [65] reported the presence of a layer in turned Ti-6Al-4V, which was likened to white layers observed in other studies. However, from the evidence provided it appears that the layer, which formed at a low cutting speed (20 m/min), was simply a region of high grain distortion. This is because the microstructure is resolvable at all locations in the images provided, by contrast, the layer which formed at 260 m/min could be a white layer as the structure is more difficult to resolve, as shown in Figure 3.3. This uncertainty in the identification of white layers from microscopy is common, possibly due to a lack of a unified definition as well as the inherent variability in the chemical etch response between different materials. Another factor that indicates that this layer formed at 20 m/min was not a white layer, is that it was generated at low cutting speeds. This is a trend contrary to the majority of research carried out on white layers, but one that is often indicative of swept grains [113].

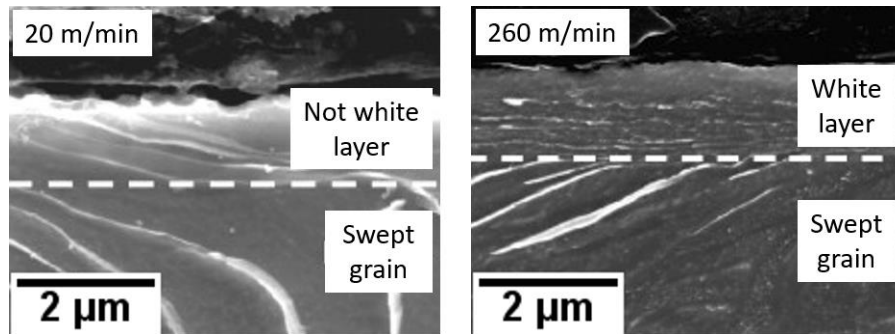


Figure 3.3 – SEM micrographs of the machined surface cross-section at 20 m/min and 260 m/min. After [65].

For nickel superalloys, specifically RR1000, it has been reported [77] that white layers can be generated using abusive drilling conditions i.e. a high metal removal rate and large average flank wear. A similar observation has been made when turning Inconel-718 [105] where it was reported that a white layer was only formed once tool wear reached levels greater than 0.15 mm and cutting speeds were high (300-400 m/min).

In addition to cutting speed and tool wear, it has been shown, through finite element simulations [114] and experiments [115], that cutting edge radius affects the SI, due to the increased level of plastic strain imparted on the machined surface when forming a chip. A larger ratio of the uncut chip thickness (t_1), with respect to the cutting edge radius (r), can result in ploughing. This is because, for small t_1/r , the workpiece material experiences a negative tool rake angle and the resultant force vector deforms the material underneath the cutting edge rather than forming a chip [116]. Crawford et al. [117] reported that machining with inserts that have a larger cutting edge radius resulted in a surface with a greater depth of deformation in titanium alloys. This was attributed to a larger contact area for the inserts, which resulted in a deeper distribution of stress in the workpiece. It has been predicted [118] that as the t_1/r ratio decreases, white layer formation will be enhanced. Herbert et al. [115] observed 15 μm of swept material due to SPD when plunge milling with a 25 μm depth of cut using a tool with a 30 μm cutting edge (t_1/r ratio of 0.833). By contrast, when machining with a 50 μm depth of cut (t_1/r ratio of 1.667), there was only 3 μm of swept material. SPD is one of the two mechanisms for white layer formation in machining so it is conceivable that the combination of low t_1/r ratio in addition to high tool wear and cutting speeds could exacerbate the problem of white layer generation.

White layers can form during machining of steels and it has been reported [118, 119] that, once again, white layer thickness increases with cutting speed and tool wear. However, Hosseini et al. [92] measured

that the effect of flank wear, on white layer formation, was greater than cutting speed over the full range of speeds investigated (30-260 m/min). The temperatures reached during the machining of steels are critical to the surface integrity, considering the temperature-dependent phase transformation mechanism of white layer formation. This is evidenced by the tendency to produce white layers through phase transformation when high-speed machining is used [119]. Cryogenic cooling during machining has been shown to reduce the thickness of any resultant white layer [108] and it has been proposed that this is due to a significant reduction in the microstructural transformation as a consequence of the lower temperatures experienced during cutting [120]. Despite the reduction in white layers formed by phase transformation, the formation due to SPD was still observed at different cutting parameters. When machining at high speeds, the temperatures reached in the workpiece near-surface region can be sufficient for austenitisation [76, 92] with the martensitic transformation under subsequent cooling causing white layer formation. By contrast, during cutting at lower speeds the temperatures are not high enough for austenitisation, so SPD is the dominant white layer formation mechanism. In both SPD and phase transformation layers in steels, it has been suggested [121] that carbide dissolution does not occur. This is due to the small amount of time that the workpiece material is at high temperatures.

The effect of cryogenic cooling on white layer formation in titanium alloys and nickel superalloys is less well documented but it is predicted that the impact may be less significant than in steels. This is due to the lack of evidence of white layers formed via phase transformation in these alloys, even when cutting at high speeds [65, 77], except in the instance reported in titanium where both formation mechanisms contributed [96]. However, cryogenic cooling may still reduce white layer formation due to the SPD mechanism, because of reduced thermal softening. It has been shown [122], for example, that in Ti-6Al-4V the work hardening coefficient is larger at a lower temperature. Thus, when machining with cryogenic cooling, the lower local temperature in the deformation zones compared to conventional cooling means that the workpiece material can be expected to undergo reduced thermal softening and, consequently, provide greater resistance to plastic deformation. This may reduce white layer formation through the SPD mechanism, compared to machining at the same speeds using conventional cooling.

It should be noted that with the move to high metal removal rate strategies within industry [123], the occurrence of white layers can be expected to increase due to the higher cutting speeds employed. The

correct identification of this anomalous surface feature during an inspection is therefore critical for optimizing and validating modern machining methods.

3.4 Mechanical and microstructural characterisation of the white layer

3.4.1 Microscopy observations

Optical microscopy studies have often incorrectly characterised the white layer as being amorphous, as outlined earlier. However, higher-resolution analysis using transmission electron microscopy (TEM) [77] and electron back-scatter diffraction (EBSD) [124] has shown that white layers are not amorphous, they are composed of nanoscale grains. In TEM, very thin samples are examined by firing electrons through the sample to a detector, the interaction of the transmitted electrons with the sample yields information on the microstructure. TEM can be used to resolve grain structure down to 10's of nm [73], which is 100-1000 times greater resolution than can be achieved with conventional optical microscopy. EBSD is a grain-orientation measurement technique used in a scanning electron microscope (SEM) or TEM. EBSD analysis of a white layer is more difficult than as-received material, as high levels of lattice strain can lead to issues indexing the grains [125].

Ultrafine grains (< 200 nm) have been measured within the white layer in Nickel superalloys using TEM [77], an observation mirrored in hard turned steel [73]. EBSD analysis of the white layer in several nickel superalloys also revealed a fine equiaxed grain structure, as shown in Figure 3.4, that is indistinguishable under optical light microscopy [124]. At the point of writing, there has been no detailed TEM or EBSD investigation into white layers in titanium alloys.

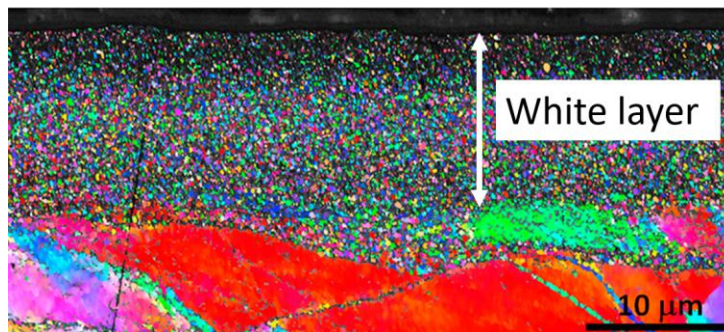


Figure 3.4 – A high-resolution cross-sectional EBSD image showing the ultra-fine grain structure (approximately 100 nm) in the white layer of drilled RR1000 nickel superalloy, after [124].

In one study, the automated measurement of the white layer thickness from cross-sectional micrographs was developed to reduce the inspection time [126]. The technique, whilst effective for the

cases presented in the study, relies on the assumption that all white layers have a dark layer beneath, which is not always the case. Consequently, the technique would fail to characterise a white layer formed from the SPD mechanism with no dark layer.

3.4.2 Nanoindentation measurements

Nanoindentation is the only hardness testing technique that facilitates indentation within the white layer, in the cross-section, whilst still adhering to widely adopted standards for hardness testing such as ASTM E384 [127]. Typical indent sizes range from several micrometres down to 100's of nanometres and these are obtained through loads that are of the order of 10 mN or smaller [91, 128].

One aspect of nanoindentation that must be considered during measurements is the small size of the indent compared to the size of the grains in aeroengine alloys [129]. Thus for an array of indents (typically 10's of μm in size [130]), individual hardness measurements could be occurring within different grains. It has been identified [131] that hardness variation between α and β grains in titanium can cause up to a 30% difference in measured values. Hardness can also vary within different crystallographic orientations of the same phase, for example, a 30-40% variation in hardness between two differently orientated HCP α -phase grains has been reported [132].

Nanoindentation within the white layer of steel alloys [70] and nickel superalloys [64] has been used to show that the feature is typically harder than the bulk material, as shown in Figure 3.5. It has, however, been recorded [133] that for IN-718, hardness can be lower in the white layer than the swept grain region beneath. This is due to the dissolution of the γ'' that is precipitated, which offsets the hardness increase that encompasses a reduced grain size. Nanoindentation, in combination with TEM [134], has been used to show that the Hall-Petch grain size relationship, which can be used to describe the hardness increase in the white layer, is opposite for high and low cutting speeds, due to the difference in the formation mechanisms. At low cutting speeds, dislocation hardening was found to be dominant, whereas for higher cutting speeds in steels, phase transformation influenced the hardness and grain diameter relationship.

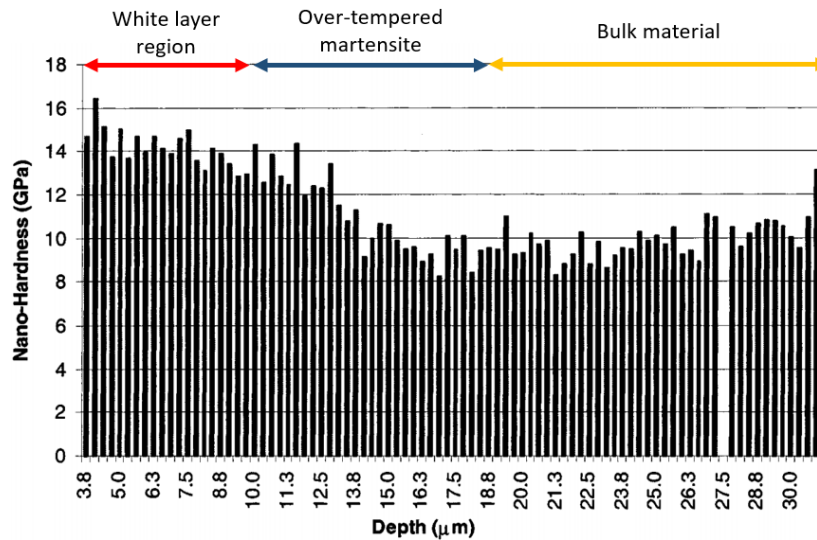


Figure 3.5 – Nanoindentation measurements with increasing depth in a steel sample. The white layer and bulk regions are highlighted. After [70].

Wusatowska-Sarnek et al. [64] observed that the depth of the hardened region corresponded to the width of the white layer measured from optical microscopy in IN-100 nickel superalloy. In titanium, a similar hardened surface region has been recorded [128, 130] and although there is no specific mention of a white layer, the optical micrographs presented suggest a white layer may have formed. Li et al. [128] measured a hardened region, despite indenting too close to the surface compared to the recommended procedure outlined in ASTM E384 [127]. This would typically result in lower hardness measurements. Further evidence is required to establish whether white layer formation leads to a hardened surface in titanium, due to the limited number of studies.

White layers can be confused with the chemical SI feature, alpha-case which forms in titanium and also results in increased hardness and has a white appearance [59]. Alpha-case formation is typically considered a time and temperature-dependent anomalous feature with prolonged heating necessary for formation [135]. During machining, where parts are heated for a matter of seconds rather than hours, the time at temperature would appear insufficient for alpha-case formation. However, diffusion rates in nanocrystalline material are typically greater than in the same material with a large-grained microstructure [136], due to enhanced grain boundary diffusion, particularly when the nanostructure is formed through plastic deformation [137]. Therefore, the presence of a nano-grained white layer may increase the diffusion of elements into the workpiece.

3.4.3 Energy-dispersive X-ray spectroscopy measurements

In energy-dispersive X-ray spectroscopy (EDXS), samples are illuminated by an electron beam and the emitted characteristic x-rays are used to determine the chemical composition of the material. The technique is typically destructive due to the analysis of specimens in the cross-section within a vacuum chamber. EDXS has been used to show that there had been a change in the composition of the α and β phases in milled Ti-6Al-4V at the machined surface compared to the bulk [131]. In the near-surface material (up to 7-8 μm in depth), after machining, the proportion of Vanadium in the β phase was reduced to 3.7 % from a value of 11.6 % in the bulk. Although not explicitly identified by Moussaoui et al. [131], this β -depletion and the lack of a visible white layer in the optical micrographs presented is indicative of alpha-case formation rather than a white layer formed through phase transformation. However, due to the lack of prolonged heating of the material during drilling, further investigation would be required to determine which SI feature is present. In steels, no change in chemical composition, even for thermally driven white layer formation, was reported after EDXS analysis of surfaces with and without white layers [138]. By contrast, in the white layer of a nickel superalloy, an increase in the concentration of Al and Ti was recorded alongside a decrease in Cr, relative to the bulk material [64]. This change was attributed to the dissolution of the primary precipitate phase into the matrix under the temperatures experienced during cutting. One key limitation of EDXS for white layer characterisation is that the technique is not suited to the detection of elements with low atomic numbers, e.g. C, N and O, all of which are α -Ti stabilisers.

3.4.4 X-ray diffraction phase analysis

In x-ray diffraction (XRD) phase analysis, x-rays are guided onto the surface of a sample and are reflected into a detector by the crystal lattice due to Bragg diffraction [139]. The angles at which peaks in reflected intensity are observed are used to determine the phases present. In electro-discharge machined Ti-6Al-4V [140], white layers have been found, via XRD phase analysis, to be formed due to a microstructural transformation during cooling from a molten state. By contrast, In chip forming machining operations in titanium alloys, apart from a recent study that identified β precipitation in the nano-grained surface layer formed during high-speed machining [96], there have been no observations of phase change in the white layer [65]. There have, however, been contrasting reports of phase transformations in the adiabatic shear band of titanium chips, one study [97] identified phase transformation through the appearance of the α''

phase, whereas another study [141] reported that only bulk phase composition was observed in the chips. Re-orientation of the grain structure in the machined surface of Ti-6Al-4V, evidenced by a change in XRD peak height, has been reported [142] however this phenomenon was not explored further aside from attributing it to machining damage.

In the machining of steel alloys, the temperature of the workpiece at the tool contact point can raise as high as the austenitic phase transformation limit [73]. XRD studies [92, 143] have shown that austenitic transformation of the machined surface at high temperature followed by rapid cooling results in a higher retained austenite concentration, as shown in Figure 3.6. The resulting microstructure with ultra-fine grains, in this instance, is a white layer. In nickel superalloys, a method of melting followed by rapid cooling has been proposed for white layer formation in ground surfaces [144]. This mechanism should be identifiable by the formation of metastable phases, such as the HCP ζ Ni₃Nb phase in superalloys with high Nb content [145], however, these phases have not been recorded in white layers at the time of writing [105].

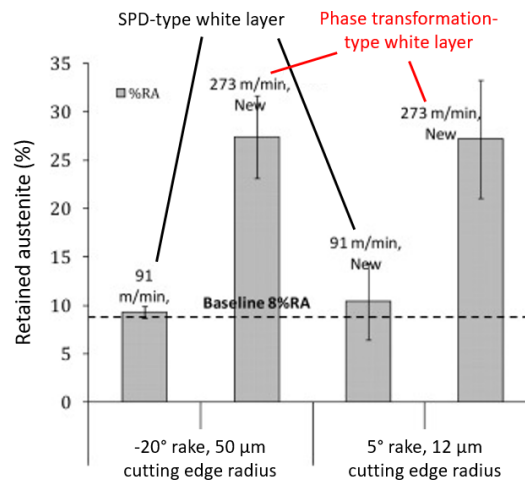


Figure 3.6 – Retained austenite content measured through XRD for hard-turned steel. Refined subsurface microstructures were present in the first 500 nm of each sample. After [143].

In situations where the white layer is formed by the SPD mechanism, it has been demonstrated that there is no appearance of different phases in an XRD pattern for nickel superalloys [77] or high strength steel alloys [92]. This same result would be expected for a white layer in titanium formed via the SPD mechanism.

3.4.5 Residual stress measurements

Residual stress measurements can be obtained with XRD using the $\sin^2(\psi)$ method [146], in which the changes in the angular position of peaks in intensity from high-angle diffractions, due to lattice strains [147], are measured. In an alternative technique, blind-hole drilling, a hole is drilled in the sample and strain gauges or optical techniques are used to measure the resultant strain of the material after the drill is removed [148]. In both techniques, the residual stress is calculated from the measured strains.

Tensile residual stresses as large as 2000 MPa have been measured in the white layer after drilling of RR1000 superalloy [69], as shown in Figure 3.7. Whilst these residual stresses exceed the yield strength of the bulk material, the white layer is hardened [69] and so the tensile strength of this region is high enough to accommodate such extreme residual stresses. In titanium alloys, it has also been reported that tensile residual stresses can be present in the white layer of a machined surface [65]. In the same study [65], it was reported that when using a lower cutting speed, a severely deformed surface region is formed, with compressive near-surface residual stresses. This agrees with the theory that tensile residual stresses are typically produced when temperature effects during cutting have been dominant [149], i.e. the phase transformation at high cutting speeds. Compressive residual stresses, by contrast, can be used to imply that plastic deformation was overriding [14], indicating the occurrence of the SPD mechanism [150].

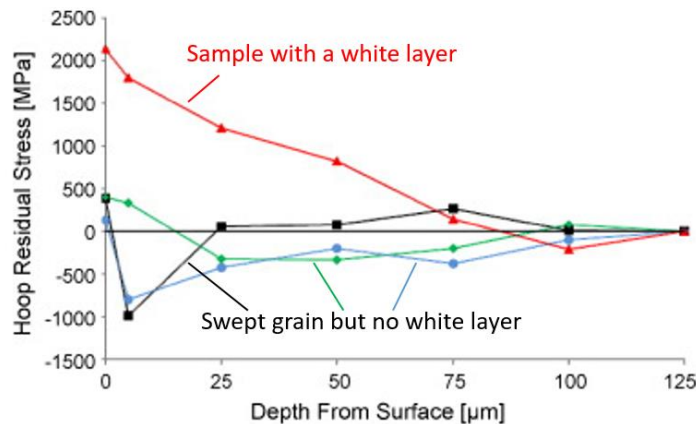


Figure 3.7 – Residual stresses profiles with increasing depth for the hoop direction in drilled RR1000 nickel superalloy, after [69].

Compressive residual stresses have typically been measured in the white layer in hardened steel [108, 151], with the maximum compressive stress located beneath the machined surface rather than adjacent to it due to the greater thermal effects at the surface, which tend to encourage a more tensile residual stress. This observation was also recorded in another study into white layers in steel [104] where it was

shown that the compressive residual stress was greatest at the interface between the white layer and the dark layer when it is present.

3.4.6 Crystallographic texture measurements

Crystallographic texture can be measured through XRD or EBSD using the principles of x-ray and electron diffraction respectively [139]. Transmission EBSD has been used to show that the white layer formed when turning steels at high cutting speeds [143] has a strong recrystallised texture indicative of DDR. This contrasts with the shear texture seen for white layer formation at lower cutting speeds in steels [143] and when turning [124] and broaching [78] nickel superalloys, which is indicative of CDR, as outlined previously. No study investigating the crystallographic texture of white layers formed in titanium exists at the point of writing.

3.4.7 Strain measurement

It is not possible to measure the plastic strain in the white layer from optical micrographs of the etched surface due to the ultra-fine grain size, which prevents grain distortion from being visualised. By contrast, the strain in the swept grain is more easily identified by measuring the angle of grain distortion in the cross-section [152]. To improve the understanding of the mechanisms for white layer formation, knowledge of the strain imparted during machining would be beneficial.

In addition to microstructural observations of machined surfaces, non-contact full-field and local deformation characterisation techniques such as Digital Image Correlation (DIC) and micro-grid techniques, respectively, have been used to determine localised strain in deformed materials [153, 154]. Finite strain plasticity principles are implemented in both techniques to determine local strain distributions from local displacement fields. In the DIC technique, the plastic deformation is measured by monitoring the evolution of the displacement field which is made visible by tracking a speckle pattern on the surface [155]. The incremental displacement field from one image frame to the next is typically used to build up a picture of the overall deformation as tracking the speckle pattern increases in difficulty as the particles move further from their previous position.

In the micro-grid method, a fine grid, such as that presented in Figure 3.8, is printed or carved onto the polished surface of the workpiece using a technique such as electron beam lithography [154], electro-polishing [156], or focused-ion beam etching [153] and then under loading, this grid deforms with the

material. To calculate the strain, an image of the grid structure after deformation is compared to the undeformed grid structure and the evolution grid intersection displacement is used to calculate the level of imparted strain, using deformation mechanics [157]. The DIC method can only be used when machining dry, due to the need to be able to image the workpiece at all times and additionally the method is less suited to strain measurement in the workpiece region immediately adjacent to the tool, i.e. at the machined surface [155] due to fuzzy delimitation of the cutting tool in the image. Micro-grids require only an image pre and post deformation and therefore avoid the need for constant imaging the part, meaning the grids can be sandwiched between workpieces, thereby restricting out-of-plane deformation. However, the grid patterns are complex and time-consuming to produce, particularly when aiming for sub-micron sized grids [154]. Ghadbeigi et al [154] directly compared both DIC and micro-grid methods for strain measurement in an in-situ tensile test. Reasonable agreement (16% error) was found between both methods, with the micro-grid method being judged more appropriate for the quantitative measurement of plastic strain.

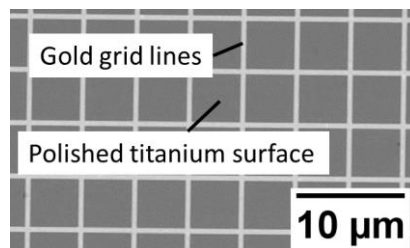


Figure 3.8 – A 5 μm pitch, gold grid printed onto a polished titanium surface using electron beam lithography.

Attempts have already been made to measure the plastic deformation during machining when a white layer is formed [153] or not formed [85, 158, 159]. Early work by Childs [159] measured flow patterns during orthogonal cutting using grids with a 25 μm pitch and a 5 μm line width, however, these grids were inscribed into the surface and therefore may have influenced subsequent deformation due to changes in local stress concentration near grids. The strain imparted in the machined surface during orthogonal cutting of aluminium alloy AA 5182 using 10 μm pitch micro-grids with a 5 μm line width has also been measured [85]. These grids were produced by EBL and were capable of measuring the strain even in regions of large strain gradient in the grids adjacent to the machined surface. In a recent study [158], the surface drag in machined Ti-6Al-4V was quantified using a grid produced by micro-milling, with a pitch of 40 μm . It was measured that the depth of deformation observable with the micro-grid method exceeds that indicated during conventional optical microscopy surface integrity inspection.

Only one study has investigated the experimental measurement of strain within the white layer [153], the micro-grid was produced through FIB etching and had a 10 μm pitch and 3 μm line width and depth. It was measured that a plastic strain threshold of 0.65 was required for white layer formation in the AISI52100 material under investigation. Considering the 10 μm pitch of the grids, a white layer thicker than the 1.82 μm layer presented would be required for higher resolution strain measurement from such comparatively large grids, in addition to the effects of stress concentrations introduced by machining the grids into the workpiece. Strain prediction in the white layers of other materials that are prone to white layer formation has only been undertaken using modelling. One study [114], investigating white layer formation in nickel superalloys predicted a strain of up to 1.2 in the swept grain material, with greater strain at lower speeds and larger cutting edge radii. There has been no quantitative experimental or theoretical investigation into the plastic strains associated with white layer formation in a titanium alloys. As such, measurement of strain in the machined surface of a titanium aeroengine material during white layer formation, using micro-grids with a range of pitches, which are printed onto rather than etched into the material, is worthy of further investigation.

3.5 Summary of machining-induced white layers

It is apparent that the white layers formed in titanium and nickel superalloys can arise due to different mechanisms to those in some steels. Analysis of the physical properties, however, shows a considerable similarity between white layers in different materials regardless of the formation mechanism. All white layers are reported to be hardened, possess large residual stresses and contain small grain sizes, although they differ in chemical and phase composition depending on the formation mechanism. It is evident from the literature surveyed that there is still limited knowledge on the formation of white layers in titanium, particularly concerning the mechanisms involved and the resulting mechanical and metallurgical properties. Published research has not looked closely at a number of properties, such as elastic modulus and texture, which are fundamental to several non-destructive testing methods. An understanding of elastic modulus changes and the crystallographic texture in the white layer would certainly enhance the state-of-the-art and aid development of a suitable non-destructive inspection method. Additionally, quantifying the plastic strains involved in white layer formation, using the micro-grid method, will aid understanding of the formation mechanisms. This will contribute to the future development of machining tools or cutting strategies that may help reduce the likelihood of generating this anomalous feature.

4 Non-destructive methods for surface inspection

In this section, the published research in the field of non-destructive testing (NDT) of white layers will be discussed. However, NDT of white layers has been subjected to limited research, despite the considerable weight of research into machined surface integrity and the potentially catastrophic effect of the presence of white layer in a critical aeroengine component. This is possibly due to a lack of knowledge surrounding the properties of white layers. Due to the limited number of studies focusing on white layer detection, this chapter will also discuss other relevant NDT methods for surface inspection. For each technique, a description of the physical principles will be presented before discussing the literature. To conclude the chapter, an assessment of each NDT technique is made concerning its potential for detecting white layers.

4.1 The drive for NDT

In industry, white layers are currently identified through cross-sectional microscopy of cut-ups from a machined surface [160]. Typically, if optical microscopy inspection indicates a white layer thinner than 5 μm is present, then higher resolution SEM imaging is undertaken to verify this observation. This is because the detection of a white layer using optical microscopy is affected by the quality of the etching process used to reveal the microstructure. Edge rounding during cross-sectional sample-preparation can reduce the effectiveness of the etching process near the machined surface, therefore, higher resolution SEM imaging is typically used to verify the presence of thin white layers. However, the edge-effect in SEM imaging, wherein the brightness of the SEM image increases at the edge of the sample, due to the additional electrons released from the material around the edge, limits the thickness of white layers that can be identified from SEM to those greater than around 1 μm .

Microscopy, as a method for identifying white layers, is inherently costly, in terms of parts sacrificed, and time-consuming, due to the numerous process steps involved in preparing metallographic samples. At present, there is no widely researched and truly non-destructive method for reliably detecting the presence of a white layer in different alloys. Therefore, there is a high demand to be able to detect any white layer formed during machining using NDT techniques.

The advantages of white layer detection through NDT can be summarised as

- Enabling inspection of all components entering service, this contrasts the microscopy approach wherein a part must be sacrificed to validate the manufacturing route and it is then assumed all parts manufactured in the same way possess the same surface integrity.
- Significantly decreasing the time between machining and obtaining inspection results. NDT could give results within minutes rather than weeks. This allows for faster product development for tooling manufacturers and quicker process validation for aeroengine manufacturers.
- Allows inspection to be carried out in-process, either within the machining centre or in a purpose-built cell. In turn, this would allow for a traffic light system for machined parts, whereby parts that fail surface integrity inspection can be stopped before further manufacturing occurs, this saves machining and material costs.
- Enables data from surface integrity assessment to be added to the digital twin of a component in a smart factory, enabling more informed decisions to be made on the required actions for the component.
- Reduces the need to etch samples for microscopy using extremely harmful chemicals such as hydrofluoric acid.

4.2 Acoustic emission testing

Acoustic emission testing (AET) is based upon the physical principle that elastic (acoustic) waves are generated within a material when there is a re-distribution of stress fields. A schematic outlining acoustic emission monitoring during machining is shown in Figure 4.1. Due to the need for stress re-distribution, AET is inherently an in-process method. Signals in AET are generated over a wide range of frequencies from several kHz up to MHz frequencies, [68]. Typical features of AET signals used for condition monitoring include time-domain features such as the root mean square (RMS) of the signal and the count rate, as well as frequency-domain features such as the peak frequency and peak amplitude. Documentation on the standards for AET can be found from the International Standards Organisation (ISO) [161].

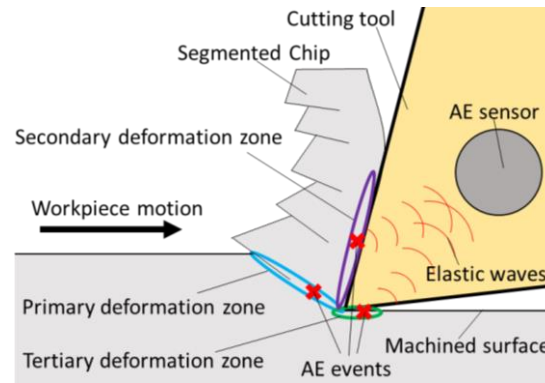


Figure 4.1 - Acoustic emission generation from the deformation zones during machining and the detection of elastic waves with an AE sensor.

AET is commonly used within the field of machining for tool condition monitoring [162]. This is because acoustic signals are generated through changes to the condition of the tool, such as rake face pitting and flank face abrasion [163]. Research has also been undertaken to investigate combined workpiece and tool condition monitoring [164]. It was shown that AET could be used to both monitor the condition of a tool and simultaneously detect when anomalous surface features were generated on the workpiece surface. This holistic condition monitoring approach is possible because acoustic emission (AE) energy can increase when workpiece surface anomalies such as laps and smeared material are generated during machining. This is due to the large redistribution of stress fields associated with these examples of severe plastic deformation.

There have been attempts [68] to monitor white layer formation during the turning of hard steels using AET. It was proposed that the RMS and frequency of the AE signal could be used as key indicators for the onset of white layer generation in the machined surface. It was reported that, due to the brittleness of the white layer compared with the bulk material, the RMS of the AET signal detected when generating a white layer was as much as 100% greater. The increased brittleness leads to reduced damping and consequently larger amplitude vibrations. Through the use of feedback loops, AET could in theory be used to monitor for white layer formation in-process and intelligently adjust the machining conditions to eliminate the generation of the anomalous surface.

It should be noted that surface roughness, tool edge sharpness and white layer formation are all reported to affect the AE signal but these effects are not trivial to isolate for every AE signal feature, as shown by Guo and Ammula [68]. Additionally, AE is generated from several sources, including the primary, secondary and tertiary shear zones, as shown in Figure 4.1. It has been reported [165] that deformation in

the primary and secondary shear zones contribute a greater proportion to total acoustic wave emission than the tertiary shear zone, however, the tertiary shear is critical in the formation of a white layer with worn inserts. Therefore future work should aim to decompose the AE signal so that only tertiary shear zone AE is monitored. This will greatly aid the development of AET for white layer detection.

AET is a mature in-process NDT method that would be comparatively cheap to conduct further research into, because of the accessibility of the technique. It is apparent from the research from Guo and Ammula [68] that there is merit for further work into the use of AET for white layer detection.

4.3 Eddy current testing

In eddy current testing (ECT), electromagnetic induction is used to detect anomalous features in a conducting material by measuring changes in electrical impedance. Eddy currents are induced in the near-surface of a test piece by a primary alternating magnetic field generated by the test probe, as shown in Figure 4.2. The opposing secondary magnetic field, which arises due to the induced eddy currents in the test piece, then interacts with the primary magnetic field, resulting in a change in test probe electrical impedance. Any change in the measured impedance can then be correlated to changes in one of three main factors, electrical permeability, electrical conductivity and lift-off. Lift-off in ECT refers to the change in the impedance signal when the distance between the test probe coil and the surface of the test piece changes. It is an undesirable effect that reduces the sensitivity of ECT for detecting changes in permeability or conductivity [166].

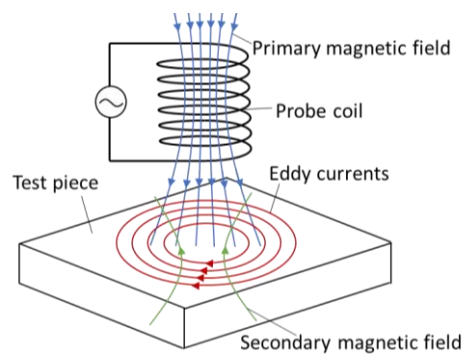


Figure 4.2 – A schematic showing eddy current induction from a probe coil.

The skin depth (δ) in ECT is the depth at which the strength of the induced eddy currents decay to 36.8% of their strength at the surface and is defined by Eq. 4.1, re-arranged from [167].

$$\delta = 50 \sqrt{\frac{\rho}{f \mu_r}} \quad \text{Eq. 4.1}$$

Here, δ is the skin depth in mm, ρ is the resistivity in $\mu\text{ohm/cm}$, f is the test coil alternating current frequency in Hz and μ_r is the magnetic permeability in the test material, relative to a vacuum. At a test coil frequency of 10 MHz, the high-end of most commercial high-frequency ECT systems [168], the skin depth in Ti-6Al-4V would be 200 μm [169]. Thus, the white layer would fill only a small proportion of the interaction volume and so the signal to noise ratio would be reduced, compared to inspection at higher frequencies. A test coil frequency greater than 100 MHz would be required to restrict the skin depth to the typical thickness of a white layer (10 μm or smaller). It should also be noted that the penetration depth in ECT is usually considered to be 3δ , as the material beneath the inspection depth can still have significant effects on results [167].

ECT is typically used to detect cracks in metallic components [170] and extensive standards for this application can be found from the ISO [171]. Due to the large number of microstructural features that can influence the measured impedance, ECT can also be used for macroscopic grain texture measurements [172] and to obtain residual stress profiles [173]. This may be useful for white layer detection because white layers have already been associated with extreme surface residual stresses and a different crystallographic texture to the bulk material.

Researchers [173] have investigated the application of ECT for determining residual stress profiles in shot-peened nickel superalloys. This was achieved through the measurement of the change in conductivity at different inspection frequencies. The residual stress profiles in machined surfaces with white layers have been measured, via XRD and hole drilling, to occur over a smaller depth than those from shot-peened samples (several hundred μm rather than mm [174]). Despite this, it is conceivable that the ECT residual stress-profiling technique may be transferable to machined surfaces.

There have been attempts [175] to carry out a similar ECT residual stress profiling technique in titanium but the change in impedance due to residual stresses was of the order of 10^{-5} to $10^{-4} \Omega$. Impedance variation due to temperature was 10 times greater than this. It is, therefore, apparent that this technique is not suitable for application to titanium alloys due to the low signal-to-noise ratio. This poor signal-to-noise ratio is primarily due to the less favourable electro-elastic behaviour of titanium-alloys compared to

nickel superalloys, which results in only small conductivity changes for different stress states [176]. Additionally, it has been shown [176] that there is variation in conductivity caused by the orientation of the HCP crystal structure in titanium α grains which would increase the noise in the signal when attempting to measure residual stress. In one study [172], this orientation phenomenon was used advantageously to characterise macroscopic grain texture in Ti-6Al-4V after different heat treatments. However, it should be noted that the grains could only be distinguished when larger than 1 mm in diameter, such as in a beta-annealed microstructure. This is significantly larger than the grains found in many polycrystalline aeroengine titanium alloys, which often have small grains to provide increased strength [177], and is over 1000 times larger than the nano-scale grains found in white layers. Characterisation of grains smaller than 1 mm is limited by the achievable diameters of the current class of probes [172].

More advanced forms of ECT including pulsed, swept-frequency and multi-frequency ECT have been trialled in a study [142] into the non-destructive detection of metallurgical machining features. In the tests, various probes were trialled, including a flexible magnetic carpet probe that facilitated better contact by allowing the probe coils to follow the contours of the test piece surface, thereby reducing lift-off effects. Results showed that pulsed, swept and multifrequency ECT were not appropriate methods for the detection of metallurgical machining damage, this was largely due to the effect of surface topography features such as feed marks left by the passage of the cutting tool as it advances across the workpiece. The effect of topography features on ECT is to mask any metallurgical changes within the machined surface. However, these advanced ECT techniques were sensitive to topological features such as lapped material on the workpiece surface which resulted in probe lift-off changes [142].

The detrimental effect of surface roughness on impedance measurements has been reported in other studies and is a major barrier in adapting ECT for inspection of the white layer. In one study [178] a 30% reduction in conductivity was measured for a surface with 13 μm RMS roughness compared to 1.5 μm RMS. In addition to roughness, there is also a detrimental compounding effect due to the similarity of the lift-off and conductivity curves in low conductivity aeroengine alloys such as Ti-6Al-4V, as illustrated in Figure 4.3. These curves are the trace formed on the impedance plane for changes in each variable. It can be seen from Figure 4.3 that a change in lift-off can appear as an apparent reduction in conductivity for an alloy such as Ti-6Al-4V, thus increasing the chances of a false positive indication, i.e. detecting an anomalous feature in a surface with none present. Another key issue with ECT is electrical noise during

the inspection. At high frequencies, the capacitance of the cable connecting the ECT probe to the electrical circuitry can be affected by interaction with the surroundings and this can result in additional noise in the impedance measurement. An ECT system has been developed [179] which mounts the electrical circuitry in the test probe and this equipment has been shown to facilitate the detection of smaller artificial defects in a test piece than a conventional system, due to an improved signal to noise ratio. Currently, the only solution to the issue of surface roughness is to polish the surface being inspected to produce an ideal, flat surface.

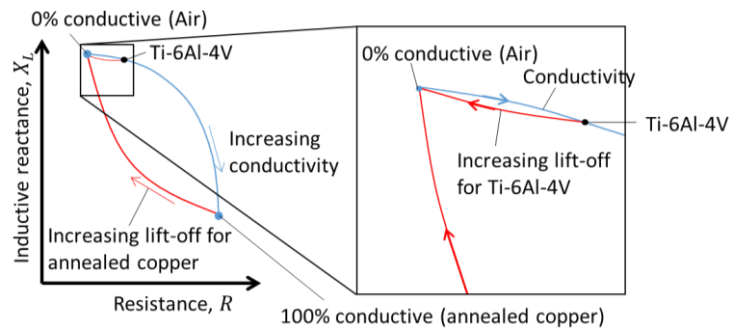


Figure 4.3. Conductivity and lift of curves on the impedance plane, after [180].

Eddy current inspection is a mature technology that would have a low cost to implement in its basic form due to the prevalence of equipment and knowledge in industry. It should be noted that high-frequency methods, which are more relevant to white layer detection, are at lower technological maturity and would have a higher cost to implement. Detection of white layers in nickel superalloys using ECT residual stress profiling should be the subject of future study due to the success of the technique for inspecting shot-peened surfaces

4.4 Ultrasonic techniques

In ultrasonic inspection techniques, the propagation of elastic waves is typically used to detect and size anomalous features such as cracks, but the phenomenon can also be utilised to determine underlying microstructural features [170]. The technique is readily accessible and is widely documented in industrial standards [181]. Concerning the detection of a white layer, surface acoustic wave (SAW) techniques are more suitable than conventional bulk ultrasonic testing wherein un-guided waves are used [182].

SAWs are elastic perturbations that propagate across the surface of a material but are confined to the near-surface region due to their rapidly decaying amplitude with depth [183]. Particles disturbed by a SAW oscillate in an ellipse in a plane perpendicular to the free surface of the material and parallel to the

propagation direction as shown in Figure 4.4. The penetration depth is typically considered as one wavelength beneath the surface, as here the energy of the wave is $1/e$ (36.8%) of that at the surface [184]. SAWs consist of a transverse wave and a longitudinal wave with identical propagation velocities. This SAW velocity, v_{SAW} , is defined by Eq. 4.2, for an isotropic medium [183], where ν is the Poisson's ratio and v_t is the velocity of a bulk transverse wave, which increases with elastic modulus. Aeroengine materials are anisotropic so the SAW velocity depends on crystal orientation due to the consequent variation in elastic constants.

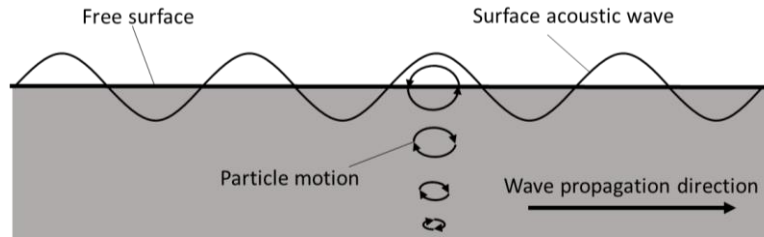


Figure 4.4 – A schematic of a surface acoustic wave and the decaying amplitude of particle motion with depth

$$v_{SAW} = \frac{0.87 + 1.12\nu}{1 + \nu} v_t \quad \text{Eq. 4.2}$$

In anisotropic materials, the wave velocity depends on the specific crystallographic plane parallel to the free surface on which the wave propagates and additionally the direction in which the wave propagates on this plane [185]. The underlying mathematical relationships are complex and beyond the scope of this study but in brief, involve solving the wave equation with a specific set of boundary conditions [185]. These conditions include the elastic constants for a particular crystal orientation and consideration of the traction-free surface on which the wave propagates. A traction-free surface cannot support stress perpendicular to the plane of the surface [186]. Essentially, through using known material elastic constants the SAW velocity on a particular crystallographic plane in a specific direction can be calculated.

HCP materials, such as α -titanium, show significant variation in elastic modulus with respect to the c-axis and this causes a variation in SAW velocity. For a SAW propagating on the basal plane of a HCP titanium crystal, the velocity is higher relative to propagation on other crystallographic planes and is directionally independent. Therefore, the velocity surface, which describes the angular dependence of velocity on propagation direction for a particular plane, is circular and has a large diameter [187], as shown in Figure 4.5 (a). By contrast, for other HCP planes, the velocity surface is elliptical i.e. the velocity changes with direction, as shown in Figure 4.5 (b), or can change rapidly for a small range propagation direction, leading

to velocity surfaces that are more complex than a simple ellipse in which the fastest and slowest directions are perpendicular. For the HCP prismatic planes, the velocity surfaces are identical as, acoustically, the HCP crystal behaves like a cylinder. This similarity between the two planes means the exact orientation of a HCP crystal cannot be determined in certain situations using SAWs, an issue not encountered for cubic crystals.

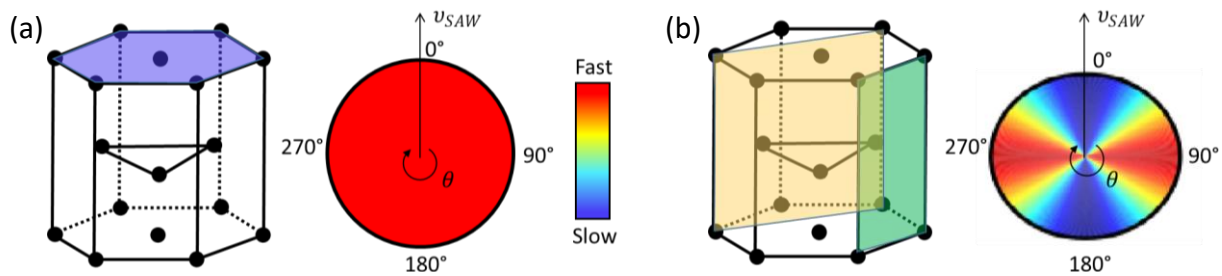


Figure 4.5 - Schematics showing (a) the basal plane and corresponding velocity surface and (b) the prismatic planes and their corresponding velocity surface, after [187].

In SAW techniques a surface wave is introduced on the surface of a test piece by a piezoelectric device [142] or a laser [188]. The propagation speed of the SAWs depends on the density and elastic stiffness constants of the local region of material [185], which are related to the elastic modulus and crystal structure orientation. Any change in these properties within a material can be detected by measuring a change in the velocity of the SAW [189].

Ultrasound is attenuated, even in ideal (anomaly free) materials, due to the spreading out of the wave. In non-ideal materials, there is also additional attenuation due to scattering and absorption [190] and it is these two attenuation mechanisms that are fundamental to SAW NDT methods. Scattering is the reflection of a wave in directions other than the original direction of propagation, whereas absorption is the conversion from sound to another form of energy. At material grain boundaries, ultrasound is always scattered to some degree due to changes in acoustic impedance [191]. Ultrasound attenuation can, therefore, be used as the basis of a technique for evaluating the grain size in polycrystalline materials, as shown by Li et al. [190] in AISI 304 steel. It was suggested [190] that the degree of attenuation is greater for smaller grain sizes as there are a higher number of grain boundaries to scatter the waves.

The backscatter of SAWs in machined Ti-6Al-4V has been investigated [142] for the detection of machining-induced damage. In this case, damage referred only to a workpiece with poor SI and so may have contained a white layer and a swept grain region. An angled probe ($\sim 30^\circ$) was used to introduce

surface waves on the titanium part submerged in a water tank and the ultrasonic signal from the backscattered waves was measured. It was found that the incident probe angle at which the backscattered signal is maximum, was as much as 1° lower for a damaged surface. This was attributed to the dependence of peak SAW generation angle on SAW velocity, as highlighted by Eq. 4.3 [142] and shown schematically in Figure 4.6.

$$\theta_{SAW} = \sin^{-1} \left(\frac{v_{l,water}}{v_{SAW}} \right) \quad \text{Eq. 4.3}$$

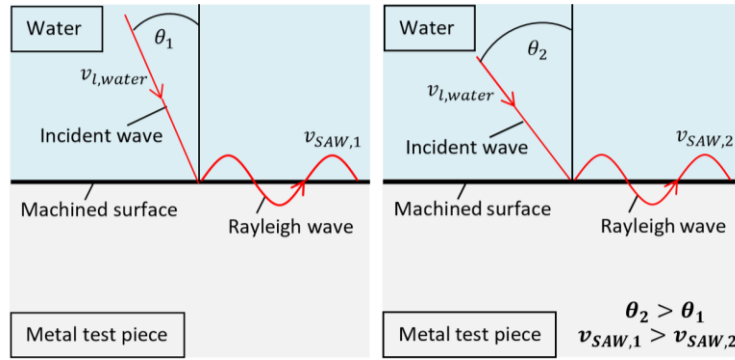


Figure 4.6. A schematic showing the dependence of the incident beam angle on SAW speed.

In Eq. 4.3, θ_{SAW} is the incident ultrasound wave angle required to generate a SAW, $v_{l,water}$ and v_{SAW} are the velocity of longitudinal ultrasound wave in water and the velocity of a surface wave in the test piece respectively. Patton et al. [142] proposed that this technique could detect damage due to changes in the elastic modulus of the surface region. Further investigation at higher frequencies, where penetration depth is smaller, could improve the capability to specifically detect a white layer rather than simply identifying broader machining damage. Due to the findings of Patton et al. [142], an investigation into the elastic modulus changes in the white layer and swept grain regions of titanium could reveal key differences between these features and aid the development of an NDT method to specifically target white layer.

In the same study [142], the use of ultrasonic microscopy for the detection of general machining damage was also investigated. As a SAW wave propagates it loses energy into the surroundings and a portion of this acoustic energy propagates through the water surrounding the submerged test piece and can be detected by a transducer. The amplitude of the radiation from the so-called leaky SAW was found to have no correlation with the presence of metallurgical anomalies such as a white layer or swept grain, as the effect of surface topology was overriding [142]. The technique was instead found to be sensitive to SI topology features such as redeposited material.

In addition to attenuation, the non-linearity (distortion) of waves can be used for NDT. In one study [192], non-linear ultrasound was utilised to characterise the hardness of heat-treated steel. Here the change in the density of dislocations and precipitates present in the material, due to ageing heat treatments, were found to directly impact the non-linearity parameter of the SAW, a common measure of wave distortion. It is predicted that, based on results presented here, both non-linear ultrasound and attenuation of ultrasound have the potential for use as a technique for white layer detection. This is due to the ultra-fine grain structure, in addition to SPD, associated with the anomalous surface feature. It is important to consider that the technical maturity and accessibility of these methods is lower than a method such as conventional ECT, partly due to the greater complexity of these ultrasonic techniques.

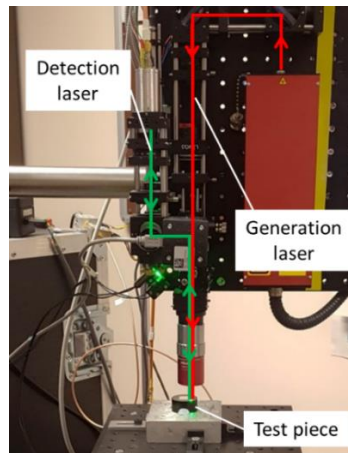


Figure 4.7 – The SRAS set-up with SAW generation and detection laser paths highlighted.

Spatially resolved acoustic spectroscopy (SRAS) is an ultrasonic inspection technique in which laser-generated SAWs are used to determine the grain orientation in a material through measurement of acoustic velocity. The laser-generated SAWs propagate across a small region of the sample and are detected by a continuous detection laser as shown in Figure 4.7. By determining the velocity of the surface waves in multiple directions and comparing these measurements to known relationships between crystal orientation and the wave propagation speed, a map can be produced showing the grain structure and orientation of the crystal lattice [193]. Further information on these crystal relationships can be found from Farnell [185].

SRAS scans in multiple orientations, when combined, can give comparable information to EBSD scans. This is at high scan rates of 1000 points per second and without the need to destructively section all samples in SRAS due to more generous size constraints. However, the spatial resolution in SRAS (100 μm)

is inferior to EBSD (several nm) and currently, SRAS samples must be polished smooth [194], which means the technique is not truly non-destructive. Although the capability of the current class of equipment is limited by sample roughness [193], techniques to circumvent this issue are under investigation [195], so SRAS may soon offer a lower resolution, truly non-destructive alternative to EBSD. The lower spatial resolution of SRAS is due to restrictions on the size of the generation patch that can be used, a smaller patch gives improved resolution but results in greater laser intensity which can lead to the burning of the sample [196]. However, the white layer is generally a continuous surface layer so a large spatial resolution is not critical and SRAS may still be able to detect a general change between the anomalous material and conventional material.

It is more appropriate to consider the depth resolution of SRAS when considering white layer detection because decreasing the wavelength of the laser-induced surface waves will allow smaller thicknesses of material to be examined. The depth resolution could, therefore, be tailored to increase the signal-to-noise ratio of any measurable difference associated with the white layer. The size of the detection laser spot in SRAS is 8 μm which, due to how the waveform is measured, dictates that detection of wavelengths smaller than 16 μm is not feasible [196]. Thus the current depth resolution limit is 16 μm , which is small enough that white layers may still form a significant portion of the interaction volume.

Early SRAS research focused on orientation measurements in cubic, single-crystal materials [188] but has since been expanded to show that the technique is capable of evaluating grain orientation in polycrystalline materials [193]. One study [196] has shown that crystallographic texture misorientation angles within coarse-grained Ti-6Al-4V and Aluminium could be evaluated to a standard deviation of less than 1.5° . Misorientation angles describe the difference in crystallographic orientation between two crystals. Whilst this small misorientation is comparable to the capability of XRD, Smith et al. [196] noted that this is still 3 times greater than for EBSD. There has been no study into the use of SRAS for evaluation of machined surfaces, however, based on the theory behind the technique and results summarised here, there is scope to trial this technique for white layer detection. The cost to implement SRAS, compared to a more established technique like ECT, would be higher due to the low technical maturity. The technique, at this stage, is still laboratory-based.

4.5 Barkhausen noise inspection

Below their Curie temperature, all ferromagnetic materials, are composed of magnetic domains in which the magnetic moment vectors of all atoms are aligned [197], as illustrated in Figure 4.8 (a). The orientation of these magnetic domains within a material is random when in an unmagnetised state. Adjacent magnetic domains are separated by a domain wall over which the magnetisation direction is rotated from one domain to the other. Domain walls are typically classed as 180° or 90° (non- 180°) domain walls, according to the angular difference between the magnetic moments in each domain. Under the application of an external magnetic field (H), the magnetic domain walls within the material will move and rotate such that the overall effect is magnetisation (B) in the direction of the applied magnetic field. Favourably orientated domains grow at the expense of other domains, as illustrated in Figure 4.8 (b).

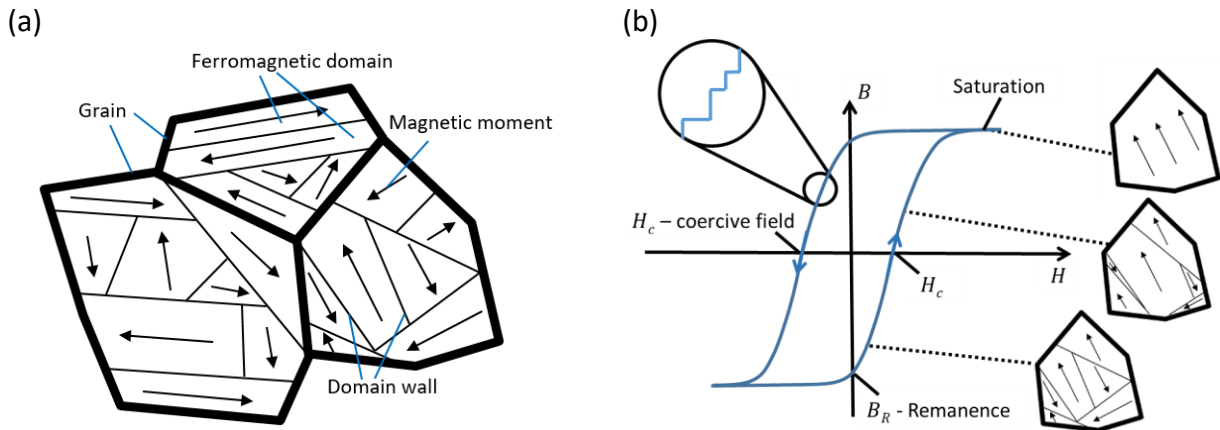


Figure 4.8 – Schematics of (a) the domain structure in a ferromagnetic material in the absence of an external magnetic field and (b) a B - H curve highlighting the discontinuous change in magnetisation with variation in magnetic field strength in addition to the domain wall motion during magnetisation.

Domain and domain wall structure is fundamentally the result of the minimisation of the sum of five energies outlined in Eq. 4.4 [198]. Consider a material composed of a single domain, as shown in Figure 4.9 (a), there is an energy associated with maintaining the magnetic field with a large distance between north and south poles. If the material was to split into two domains with opposing orientation, the energy associated with the magnetic field would decrease due to the reduced spatial extent of the magnetic fields as a result of the pole proximities [199]. This energy is the magnetostatic energy and it drives the presence of magnetic domains in a material. However, there is competing energy, the wall energy, associated with the surface energy of the domains. Smaller domains result in a larger domain wall surface energy and as such, an equilibrium state is reached between the reduction of magnetostatic energy and an increase in wall energy with an increasing number of domains [200].

$$E_{total} = E_{wall} + E_{exchange} + E_{magnetostatic} + E_{magnetoelastic} + E_{anisotropic} \quad \text{Eq. 4.4}$$

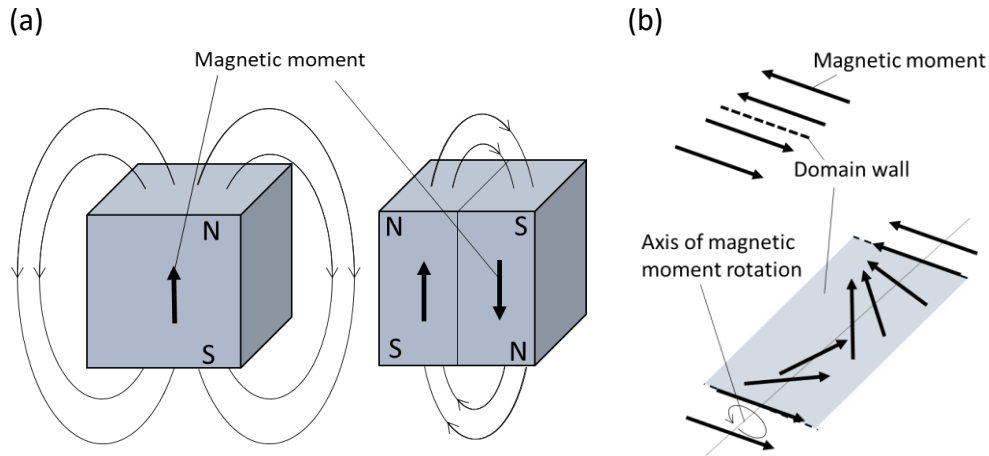


Figure 4.9 – Schematics illustrating (a) the reduction of magnetostatic energy through increasing the number of domains and (b) the rotation of magnetic moments for an infinitely small domain wall and a domain wall several atoms thick, after [199].

Domain walls are typically several tens or hundreds of atoms thick due to the balancing of exchange energy and magnetoelastic energies. Exchange energy is associated with the interaction between a pair of adjacent atoms with a different spin and therefore different magnetic moments. This energy increases with the square of the angle between the atomic spins, consequently, the energy associated with a 180° rotation between adjacent atoms is greater than the sum of energies associated with a large number of small rotations totalling 180° [200]. Thus, domain walls contain a large number of atoms over which the magnetic moment rotates, as shown in Figure 4.9 (b). However, there is also energy associated with the orientation of magnetic moments relative to the easy axis of the crystal. It has been shown [199] that there is a crystallographic direction along which magnetisation occurs for smaller changes in magnetic field strength and this direction is termed the easy axis of the crystal. Maintaining magnetisation away from the easy axis requires energy, termed the anisotropic energy. The rotation of magnetic moments over a domain wall involves orientations away from the easy axis, therefore anisotropic energy drives a reduction in domain wall thickness.

The final energy, critical to domain structure, is magnetoelastic energy which arises due to inverse magnetostriction. In a magnetic field, ferromagnetic materials change their dimensions due to magnetically induced strain. In the reverse effect, the application of stress to a material will cause a change in its magnetisation [199]. The sign of the magnetostriction parameter dictates the sign of the magnetisation change. For example, with positive magnetostriction, magnetisation increases under the

application of tensile stress, due to the growth of favourably orientated domains parallel to the applied stress. It can be seen, therefore that domain and domain wall structure is an equilibrium between these five competing effects. Changes in domain structure arise from the minimisation of the sum of these five energies as a direct consequence of an unbalancing caused by, for example, the application of an external magnetic field or the application of stress.

Changes in domain structure are achieved through domain wall motion and rotation. Rotation typically occurs closer to magnetic saturation and often requires greater energy input for a smaller return in magnetisation. The greatest barriers to rotation are typically the forces arising from magnetic moments non-parallel to the easy direction. Wall motion, by contrast, is often associated with the high gradient portion of the $B-H$ curve [199] and is influenced by inclusions and residual stress.

When a domain wall encounters a large inclusion, domain structures called spike domains are formed which increase the surface area of domain walls but reduce the magnetostatic energy, resulting in a minimum in the energy of the system, as shown in Figure 4.10 (a). When the domains are in this minimum energy state they are said to be pinned. As the domain wall continues to move past the inclusion, it stretches these spike domains in the direction of motion, increasing their wall energy (Figure 4.10 (a)). This increase in energy continues until the wall energy becomes too large and the moving domain wall snaps from the spike domains wall resulting in a jump in magnetisation (Figure 4.10 (c)). For inclusions smaller than the domain wall thickness, the magnetostatic energy is small and so wall energy effects dominate. If the domain wall contains an inclusion, its wall energy is reduced, therefore additional energy is required to continue the motion of the wall and this energy is greater for a compressive residual stress.

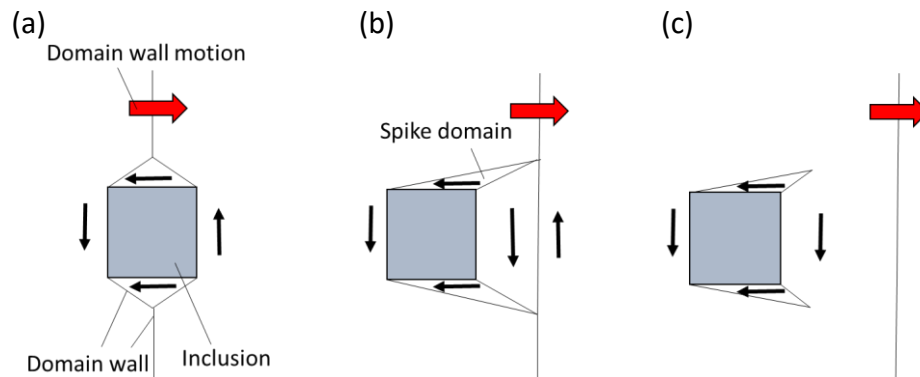


Figure 4.10 – Schematics of the motion of a domain wall away from an inclusion. (a) A domain wall bisected by an inclusion, (b) extension of the spike domains, increasing wall energy and (c) snapping of the spike domains causing a jump in magnetisation as the domain wall moves to the right.

Residual stress at the micro-scale, due to dislocations, and at the macro-scale affects domain wall motion due to magnetostriction and wall energy effects. When a 90° domain wall moves, the direction of magnetisation in the material encompassed by the motion is changed and this results in distortion of the material due to magnetostriction [199]. The interaction of the magnetostriction distortion with the local stress distribution typically counteracts domain wall motion. In materials such as iron-carbon alloys i.e. steels, the magnetostrictive constant is positive, so additional energy is required for domain wall motion under a compressive residual stress state [201].

For 180° domain walls, there are no magnetostriction effects as the magnetic moments are parallel yet opposite, however, stress anisotropy is introduced which changes the wall energy. For compressive residual stresses, additional energy is required to overcome this stress anisotropy for domain wall motion, whereas for tensile residual stress, stress anisotropy reduces the energy required.

There is a time-lag between an increase in the magnetic field applied to a ferromagnetic material and the material response in the form of increased magnetisation [202]. This is because domain walls lose energy when they become pinned, due to their adaptation to the strain field surrounding the pinning feature [203], as is illustrated in Figure 4.11 and pinning necessitates the input of additional energy for the domain wall to move. It has been shown [204] that overcoming this pinning is time-dependent as indicated by the relationship described in Eq. 4.5, describing the mean waiting time (τ), which is the average time for domain walls to overcome de-pinning.

$$\tau \propto e^{E_{WM}/k_B T} \quad \text{Eq. 4.5}$$

Here, E_{WM} is the activation energy for domain wall motion, k_B is the Boltzmann constant and T is the temperature. It can be seen that the time for which domain walls are pinned increases with the activation energy, and thus the residual stress state. There is therefore a greater lag between application of a magnetic field and domain wall motion if this activation energy increases.

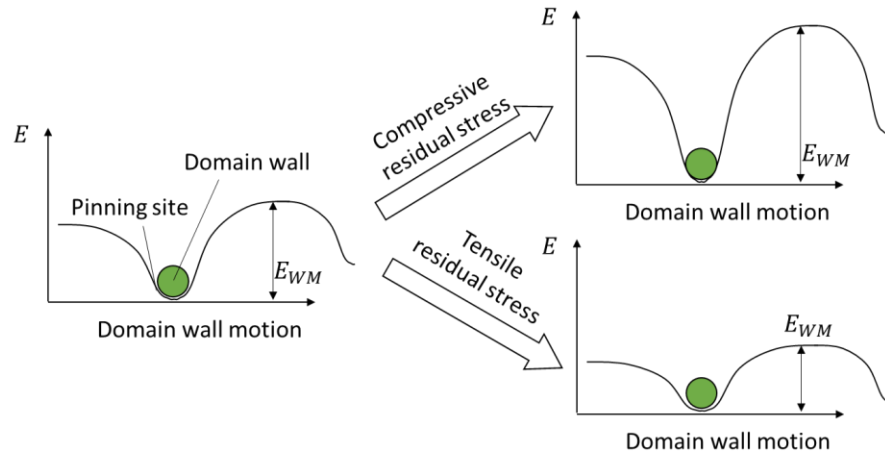


Figure 4.11 – A schematic of the energy well created around a pinning site and its change with residual stress state for a material with a positive magnetostrictive constant.

The Barkhausen effect is the discontinuous change in magnetisation of ferromagnetic material in a varying magnetic field due to sudden, finite changes in the size of ferromagnetic domains through the irreversible movement of magnetic domain walls [205] caused by the factors outlined above. Domain wall motion is detected as a change in current or voltage in a pick-up coil, in the case of magnetic Barkhausen emission (MBE), due to changes in magnetic field strength [206]. Alternatively, in magneto-acoustic emission (MAE) testing an acoustic emission sensor is used to detect elastic waves caused by stress generation during domain wall motion [197].

In Barkhausen noise (BN) testing, an alternating magnetic field is applied to the sample and as the magnetic field varies, the magnetic moments within the sample attempt to align with the field but, due to domain wall movement restrictions (e.g. dislocations), the magnetisation occurs in a step-wise manner. This magnetisation “noise” is measured by a pick-up coil through spikes in induced current or voltage [206]. A typical BN testing probe is shown schematically in Figure 4.12. Only ferromagnetic materials, such as some steel alloys, can be tested with BN equipment; nickel superalloys and titanium alloys are paramagnetic and therefore not applicable to BN testing. Standards for the BN inspection technique can be found from SAE-International [207].

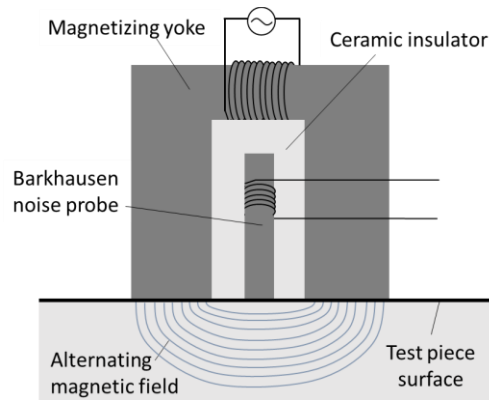


Figure 4.12 - A schematic of a typical Barkhausen noise inspection probe.

The volume of material that can be examined using BN testing methods is limited by the penetration depth δ of the electromagnetic waves, similar to eddy current inspection [199] and is also described by Eq. 4.1. The skin depth in BN is considered as the depth at which the eddy currents decay to $1/e$ of their surface strength.

Due to the dependence of penetration depth on frequency, BN inspection can be tailored to near-surface inspection using high frequencies. In MBE, the penetration depth can be further restricted using high-frequency filters, which limits the BN that can be detected to that generated close to the surface. This is because high-frequency BN that is generated at greater depths is attenuated through eddy current damping before reaching the surface. By contrast, the depth of inspection of MAE is much greater, O'Sullivan et al. [208] estimated that MAE could be used to a depth of 7-8 mm in ferritic stainless steel compared to just 0.1 mm for MBE. This is because acoustic emissions, which are only released from the motion of non- 180° domain walls, are not restricted by eddy current damping and so may propagate freely through much larger volumes of material. In this study, MBE was chosen due to its greater sensitivity to surface defects, which makes it inherently more suited to the detection of a white layer.

The most widely researched application of BN inspection is in the verification of case-hardening depth in steels [209], however the technique has also been used for the detection of grinding burn, a thermal defect introduced through grinding [210], as well as for the determination of residual stresses [211]. More recently the technique has been specifically applied to the detection of a white layer formed in milled steel [212], wherein increased tool wear during machining resulted in the appearance of a second peak in the BN envelope measured at different magnetic field strengths, as shown in Figure 4.13. Higher tool wear promotes white layer formation so this observation was attributed to increasing white layer thickness.

Stupakov et al. [213] developed a BN sensor in-house and investigated the use of different magnetisation waveforms for surfaces machined using tools with different levels of wear. They were able to rank samples, using BN, in terms of the associated level of tool wear and consequently the white layer thickness.

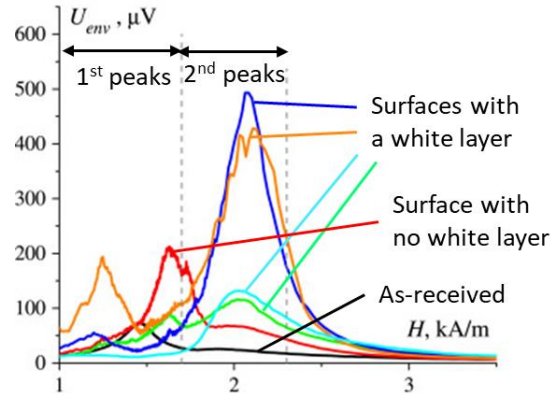


Figure 4.13 - Plots of the BN envelopes for surfaces with a white layer and the as-received material, after [195].

The ability to rank samples by increasing tool wear was attributed to a number of factors [213]: a harder white layer than the softer dark layer beneath, a favourable orientation of the martensitic matrix in the white layer and an increasing volume of retained austenite with a thicker white layer. It is worth considering that although Stupakov et al. [213] measured residual stress profiles for each surface, the effect of the residual stresses on the measurement was dismissed as inconspicuous. This was due to the hardened material being studied, a reduced inspection depth and compensation of the compressive sub-surface by a tensile spike at the near-surface. However, the maximum filter frequency used (120-200 kHz) would not usually restrict the BN to within the white layer, as suggested by Stupakov et al. [213], this is because BN is subject to the same skin depth restrictions experienced in ECT. A high-frequency filter restricts the depth from which BN can be detected by the pick-up coil. The effect of residual stress and the restriction of the inspection depth to within the white layer is therefore worthy of further investigation. It is apparent that significant progress has been made towards the reliable detection of machining-induced white layers using Barkhausen noise. However, a more thorough study to determine how the physical properties of the machined surface are facilitating this detection is required to enhance the overall understanding of this inspection technique. Additionally, the white layers studied by Stupakov et al. [213] were formed by the thermal mechanism, evidenced by the dark layers present beneath them, so it would also be appropriate to investigate the technique on a white layer formed by the SPD mechanism.

The application of BN for evaluating the case depth of case-hardened steels [209] is possible due to the difference in magnetic properties of a magnetically soft phase such as cementite (the core) and a magnetically hard phase such as martensite (the case). The measured BN is therefore affected by the difference in phase composition between the case hardened layer and the core material. Early research in this area focused on simple BN measures, such as the RMS of the BN signal, using purpose-built equipment [209]. More recently [214], magnetic voltage sweeps have been utilised on a commercially available BN analyser, such as the one shown in Figure 4.14, to successfully order a set of case-hardened axles based on the depth of the hardened region, showing that custom, laboratory level equipment is not always necessary for highly specific applications. BN has also been used for evaluating hardened layer thickness in grind-hardened surfaces using RMS measurements based on the same underlying microstructural changes [215]. In this grind-hardening study, it was found that the RMS of the BN signal decreased with increasing hardening depth, as would be expected due to the larger percentage of magnetically hard material in the inspection volume. Similarly, Liu et al [216] and Wilson et al. [217] were able to successfully distinguish between different heat treatments in grade 91 steel tubes and pipes using BN and coercivity measurements. In this case, the microstructure of the steel, both in terms of the phases present and the precipitate density, influenced the NDT measurement.

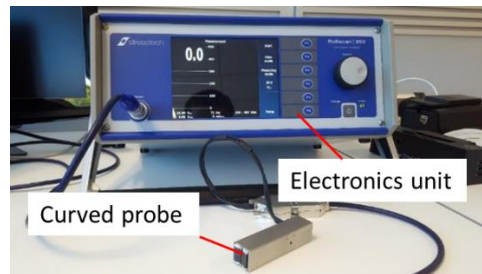


Figure 4.14 - A commercial Rollscan 350 Barkhausen noise analyzer

Quantification of residual stresses in structural steel to within ± 25 MPa has been achieved using BN inspection [206]. It is important to note that this technique was only effective within a narrow calibration region of -300 MPa to 150 MPa. The potential for measuring large tensile residual stresses was limited by a plateau in Barkhausen noise energy above 150 MPa, whereas for compressive stress the Barkhausen noise showed a linear relationship up to the calibration limit. The technique may, therefore, be suitable for measuring larger compressive stresses with appropriate calibration. Due to the large compressive

stresses that have been associated with white layer formation via the SPD mechanism, it may be possible to use BN for their detection.

Magnetizing voltage sweeps have also been used for residual stress evaluation [211]. For these sweeps, the maximum slope of the magnetizing voltage sweep divided by the magnetizing voltage was reported to correlate linearly with tensile stress, a closer relationship than that which has been measured for the RMS of the BN signal [218]. The detection of tensile stresses using sweep measurements may allow identification of machining-induced white layers formed via phase transformation.

It has been demonstrated [219] that qualitative evaluation of grain size using BN testing is achievable. Larger measured peak values in pick-up coil voltage have been attributed to smaller grain sizes. This is because a larger total grain boundary area, as found with smaller grains, restricts domain wall motion and consequently results in greater “noise” when domains overcome pinning. It may, therefore, be possible to use this technique to distinguish between the nanoscale grains in the white layer and the larger grains in a machined surface with good SI.

On-line measurements of elongation in skin passed steel samples have been carried out using BN [220]. Both the BN signal and hysteresis loop properties, such as the coercive field and the differential permeability, were found to change with percentage elongation. These changes in measured values were attributed to an increase in damage to the microstructure at greater elongation. Based on this research into in-process inspection, the adoption of BN within a machining centre may be achievable.

BN is a mature technique that would have a low cost to implement compared to other NDT techniques that are less established. The specific research into the application of BN for white layer detection by a handful of authors means that there are already excellent foundations for further work in developing this technique. However, the major restriction on the adoption of BN for inspection of white layers is the limitation of the technique to steel alloys within aeroengine materials.

4.6 X-ray diffraction methods

X-rays are a high-energy and thus small wavelength region of electromagnetic radiation. Electromagnetic waves with a frequency of 3×10^{16} Hz to 3×10^{19} Hz and a wavelength of 10 nm to 0.1 nm are classified as x-rays [146]. To generate x-rays, electrons are accelerated towards a target metal in an x-

ray tube using a high voltage, typically of the order of 10's of thousands of Volts. When the electrons impact the target metal cathode they decelerate rapidly, releasing x-ray radiation and heat energy in all directions. This x-ray radiation is composed of continuous (broadband) x-ray radiation and characteristic (monochromatic) x-rays. Characteristic x-rays are generated when incident electrons excite an electron in an atom of the target metal to a higher energy shell. An electron in one of these outer shells of electrons then immediately falls to the lower energy level to fill the vacancy left by the excited electron. Energy is released during the transition in the form of an x-ray with energy and wavelength corresponding to the energy difference between the shells [146]. Characteristic radiation, therefore, possesses a specific wavelength and is the most useful in conventional x-ray diffraction methods. To reduce the intensity of the continuous radiation and the characteristic radiation associated with unwanted electron transitions, a metallic filter is placed in the path of the x-ray beam to absorb a significant proportion of these x-rays. The material used depends on the target metal as well as the characteristic radiation selected.

X-rays penetrate only a limited depth into a material due to energy loss through collisions with atoms in the material [146]. The intensity of x-rays at a particular depth, $I(x)$, relative to that at the surface, I_0 , can be described by Eq. 4.6.

$$\frac{I(x)}{I_0} = e^{-\mu x} \quad \text{Eq. 4.6}$$

Here, μ is the linear attenuation coefficient, which is related to the number of atoms per unit area and the probability of an x-ray being scattered by them and is calculated from tables of mass attenuation coefficients by multiplying by the material density [221]. For the three aeroengine alloy groups, penetration depths for 90% attenuation of x-rays are less than 25 μm , hence, XRD is inherently a surface inspection method, ideal for white layer inspection. The actual penetration depth for XRD is smaller than the penetration of x-rays as the incident beam is not typically orthogonal to the surface and x-rays must leave the material on the diffracted leg to be detected.

X-rays incident on a sample under inspection result in the radiation of additional x-rays as well as scattering the original beam. Tightly bound electrons are oscillated by the incident x-rays and the acceleration and deceleration of these electrons produces x-ray emission at the same wavelength as the incident x-rays, this is called coherent scattering. In incoherent scattering, outer electrons scatter the incident x-ray beam, causing a reduction in wavelength due to the transfer of energy. If an x-ray quantum

was guided onto a singular atom, there would be a scattering of the x-ray in a random direction. However, when a beam of x-rays produced in an x-ray tube is guided onto the surface of a material with a regular crystal lattice, the x-rays are scattered only in particular directions due to constructive and destructive interference, related to the crystal lattice dimensions. This constructive coherent scattering forms the basis of all XRD methods [146].

Consider a parallel beam of monochromatic x-rays (A and B) incident on a crystal lattice as shown in Figure 4.15 (a). If ray A, with incidence angle θ , is scattered by the top plane of atoms AP1 through an angle 2θ and ray B, with the same angle of incidence and angle of scattering is scattered by a lower plane of atoms AP2, then there will be a difference in the distance travelled by those waves. In the example in Figure 4.15 (a), ray B has travelled a distance of $2d\sin(\theta)$ further than ray A to get to the same point X2. If $2d\sin(\theta)$ was equal to an integer multiple of the x-ray wavelength ($n\lambda$), then ray A and ray B would be in-phase and would constructively interfere, as highlighted in Figure 4.15 (b), resulting in a wave with a larger amplitude. This relationship is the basis of Bragg's law [222]. By contrast, if the two x-rays were out-of-phase by exactly half a wavelength then they would destructively interfere and cancel each other out. In a large crystal lattice with many atomic planes, for every scattered x-ray, there will typically be another x-ray scattered from an atomic plane at a greater depth which will provide destructive interference, except for the instance when $2d\sin(\theta) = n\lambda$. Thus x-rays are only diffracted when Bragg's law is met and as such, if the interatomic spacing or angle of incidence changes, there is no diffraction. From these examples, we can see the key principles of XRD.

- Diffraction occurs due to the interaction of x-rays with a regular crystal lattice
- Diffraction only occurs when $2d\sin(\theta) = n\lambda$, this is Bragg's law.
- When Bragg's law is met, constructive wave interference occurs
- When Bragg's law is not met, destructive interference occurs

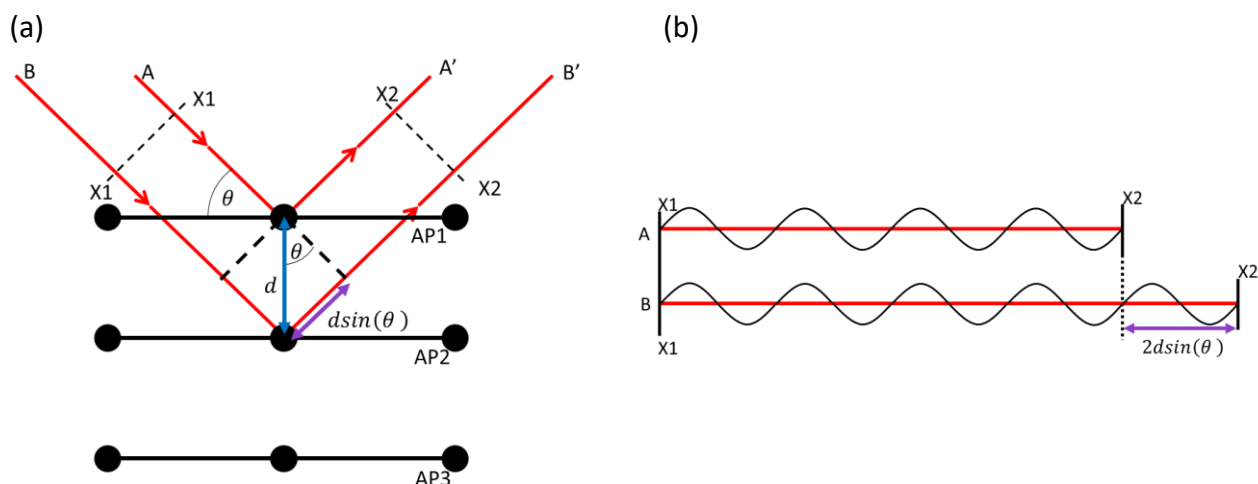


Figure 4.15 – Schematic of (a) x-ray diffraction from a regular array of atoms and (b) the difference in distance travelled for two parallel waves diffracting from two adjacent atomic planes is highlighted.

If the intensity of diffracted x-rays were plotted against the diffraction angle 2θ then peaks in intensity would be expected at angles that satisfy Bragg's law and no intensity expected elsewhere, as shown in Figure 4.16 (a). However, in real-world XRD, peak broadening occurs such that diffracted x-rays are measured at angles close to the theoretical diffraction angle and there is background radiation which gives rise to a low level of diffracted x-rays measured at all angles, as shown in Figure 4.16 (b). This is due to both instrument and sample peak broadening effects.

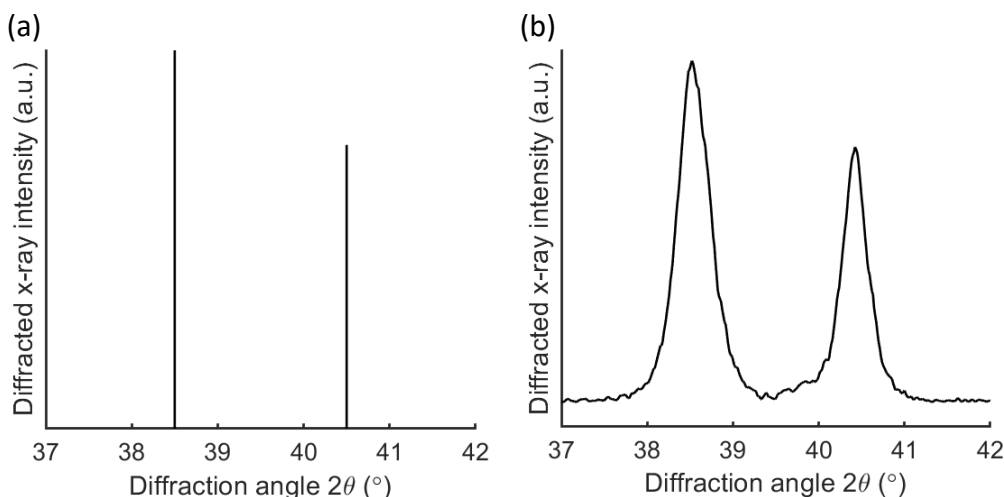


Figure 4.16 - (a) A theoretical XRD pattern and (b) an experimental XRD pattern

Instrument peak broadening arises due to the non-parallel, non-monochromatic nature of a real x-ray beam, the finite size of the x-ray source, the diffracting volume in a sample and the arrangement of slits in the beam path. A real x-ray beam contains x-rays that are converging and diverging as well as those which are parallel, this means that at a particular diffractometer angle of incidence there are always x-rays

at angles slightly above and below this angle. These x-rays may be at the Bragg angle for diffraction and so can lead to a measured intensity at diffractometer angles away from the theoretical diffraction angle. As outlined previously, an x-ray beam is composed of desirable characteristic radiation, which possesses specific wavelength, however, continuous radiation is present at a reduced level even after filtering. An x-ray beam, therefore, contains radiation at a continuous range of wavelengths so for any instrumental angle of incidence there will be a portion of the radiation that has a wavelength that satisfies Bragg's law. The slits, x-ray source and diffracting volume result in geometrical variances between x-rays and the crystal lattice such that diffraction occurs slightly away from the theoretical angle.

The effect of sample peak broadening arises due to the finite size of a crystal lattice, which extends only as far as material grain boundaries, as well as distortion of this lattice. When a grain, and thus the extent of the crystal lattice, is of the order of 10's or 100's of nm, there are only a limited number of atomic planes to provide destructive interference. For example, in titanium, which has a HCP structure, the lattice dimension for the c-axis is 0.469 nm [223]. This means, in a 50 nm grain, for example, there are approximately 213 atomic basal planes as there is a plane of atoms at the mid-point of the c-axis i.e. 2 planes per unit cell.

If the diffraction angle 2θ satisfies Bragg's law, then for x-rays at a small angle away from this e.g. $2\theta + \Delta\theta$ there is only partial destructive interference for small-grained material. This is because for a small change in angle, and therefore a small change in the path length of x-rays diffracting off different planes, there are not a sufficient number of atomic planes in a small grain for every x-ray to be cancelled by another, resulting in a diffraction pattern similar to that shown in Figure 4.16 (b). Consider now that $\Delta\theta$ is such that the x-ray (Z) scattered by the last atomic plane in the grain is $n+1$ wavelengths out of phase with the wave incident on the first atomic plane (A). At the mid-point in the lattice, there would be a plane that scatters an x-ray (M) $n/2 + 0.5$ wavelengths out of phase, i.e. half a wavelength out of phase, so destructive interference occurs. In this case, diffraction from all atomic planes in the top half of the grain cancels with those in the bottom half, as illustrated in Figure 4.17 (a) and (b). However, if $\Delta\theta$ was such that x-ray Z, scattered by the last atomic plane, was between n and $n+1$ wavelengths out of phase with x-ray A, then the plane that results in scattering exactly half a wavelength out of phase would lie past the mid-point of the grain. Consequently, there would not be total destructive interference for all scattered rays, as shown in Figure 4.17 (c) and (d), so peak broadening would be observed.

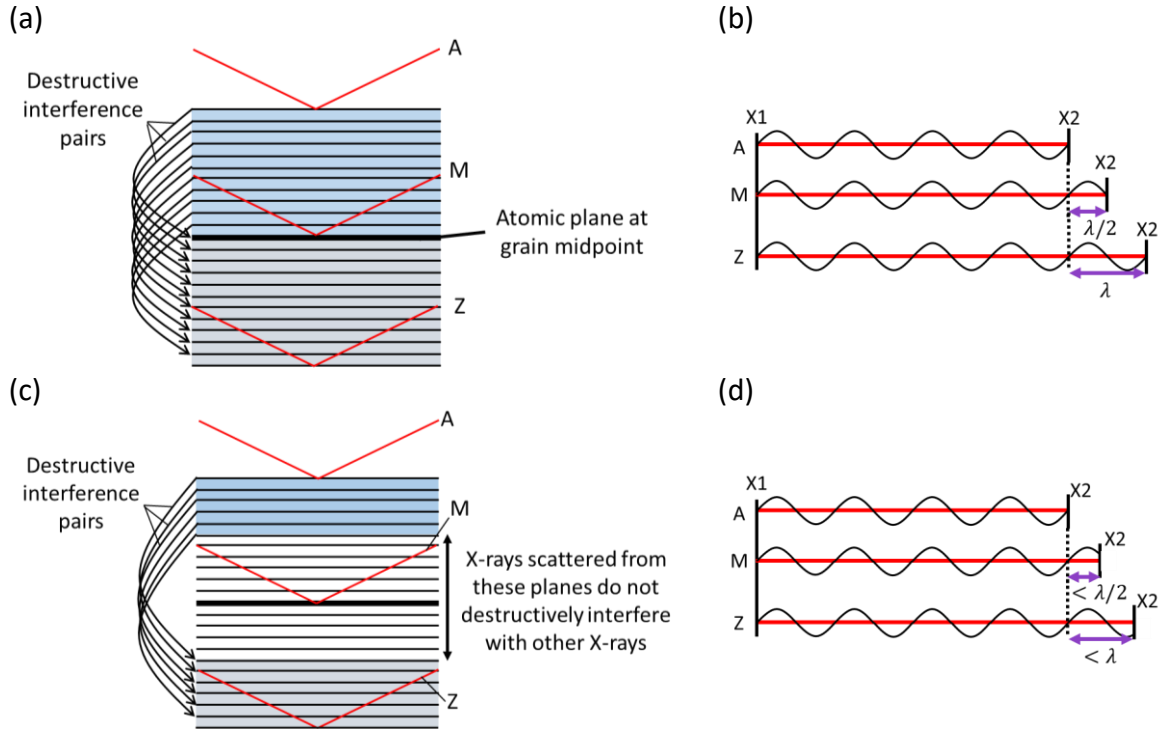


Figure 4.17 – Illustrations of (a) the case of atomic plane pairs resulting in total destructive interference and (b) the phase difference between particular rays. A comparable illustration (c) of the case of incomplete destructive interference and (d) the new phase differences for the same rays.

The relationship between crystal size and peak broadening (PB) is described by the Scherrer equation (Eq. 4.7) [224] where θ is the Bragg angle, D is the diffraction domain size, λ is the x-ray wavelength and k is a constant typically taken as 0.9. The Scherrer equation is only applicable if there are no strain broadening effects arising from inhomogeneous lattice strain.

$$PB_{cs} = \frac{k\lambda}{D\cos(\theta)} \quad \text{Eq. 4.7}$$

The diffracting domain size D is often equivalent to the grain size in polycrystalline materials, however, for SPD material produced by processes such as equal channel angular compression and high pressure torsion it has been shown D can be six times smaller than the grain size measured by TEM [225]. The reason for this discrepancy is the presence of dislocation cells within grains [226]. As a solid material is deformed and dislocation density increases, dislocations arrange into cell boundaries to minimise their energy [227]. These cells have low angle grain boundaries so there can be insufficient contrast to resolve them through TEM or SEM, however, their misorientation is sufficient that it causes XRD peaks broadening and it is the cell size which is measured by the Scherrer equation in this case [226].

Uniform strain in a lattice changes the interatomic spacing for all planes, thus shifting the associated XRD peak to a different angle, as outlined previously when describing XRD residual stress measurements. However, for non-uniform lattice strain, the change in interatomic spacing with depth beneath the surface results in diffraction away from the theoretical value for a uniform crystal. Peak broadening due to lattice strain, ε , is proportional to $\tan(\theta)$ as described in Eq. 4.8, and so is greater at high angles of diffraction. The relative effects of strain and crystallite size broadening, $\tan(\theta)$ and $1/\cos(\theta)$ respectively, at different Bragg angles are highlighted in Figure 4.18. It can be seen that for low angles, the relative effect of crystallite size broadening is more dominant than inhomogeneous lattice strain effects.

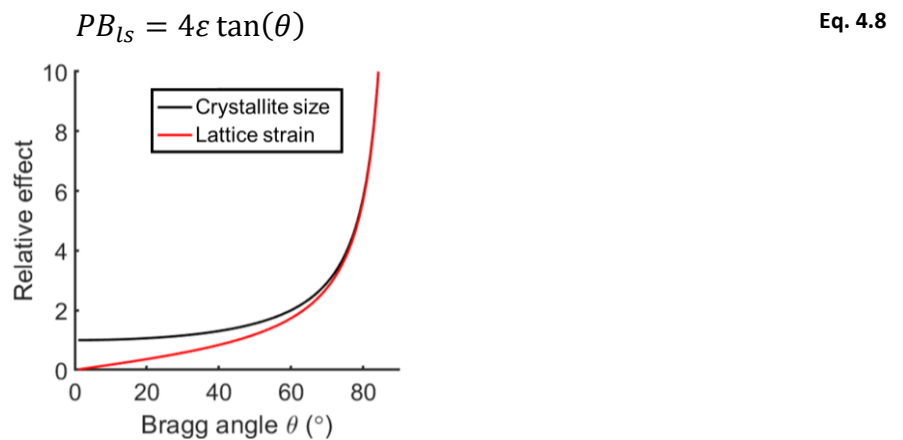


Figure 4.18 – The relative effect of crystallite size and lattice strain effects highlighting illustrating the more dominant effect of crystallite size at low Bragg angles.

Despite the interaction of x-rays with the near-surface (typically less than 10 μm [146]) of a metallic sample being non-destructive, the XRD measurements process is often destructive due to the sectioning required to fit the sample within the constraints of the diffractometer, as highlighted in Figure 4.19. It has already been identified that XRD phase analysis can distinguish between a steel surface with a thermally formed white layer and a good machined surface and additionally, machined titanium can exhibit a change in XRD peak height. The broadening of XRD peaks has been identified in rail steel when a white layer was present on the surface [228]. This was attributed to a smaller grain size within the white layer as it has been established that XRD peaks broaden when diffracting domain size is small (<200 nm) and there is inhomogeneous lattice strain [146]. It is worth noting that in the investigation of rail steel, the XRD method was merely used a way of measuring grain size rather than for non-destructive assessment. There is therefore a need for further investigation into using XRD as a method for the detection of white layers, as the technique is sensitive to small grain size, crystallographic texture and residual stress.

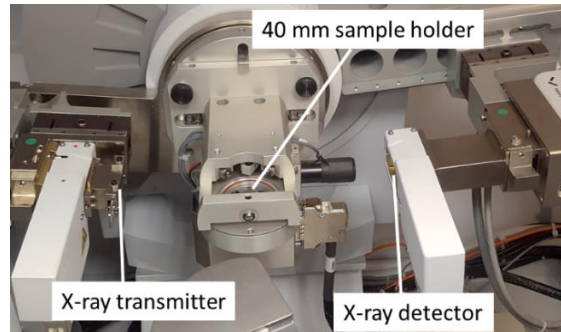


Figure 4.19 – A typical powder diffractometer for laboratory-based XRD highlighting the small sample size restriction.

XRD equipment which can facilitate larger samples, and thus negate the requirement for destructive sectioning, already exists [229], however, such equipment is typically high-cost and would require removing the part from the machining centre and mounting in the diffractometer. Considering aeroengine parts, such as discs, are often large and heavy, moving them between machines is not a desirable solution. Portable XRD equipment has been developed in recent years, particularly for the application of in-situ measurement of historical objects [230]. These systems, such as that shown in Figure 4.20, often overcome the usual complexity of incorporating a goniometer to facilitate measurement at a range of diffraction angles, by employing a fixed x-ray transmitter and a plate detector, which allow for x-ray detection over a wide range of angles without a goniometer [230]. Alternatively, a different form of XRD, energy-dispersive XRD (EDXRD) can be used [231] to improve the transferability of the technique. In EDXRD, as opposed to conventional angle-dispersive XRD, the diffraction angle is kept constant and broadband x-rays are used [232], so in this method, the x-ray wavelength is changed to identify different crystallographic planes. EDXRD has been shown to exhibit insensitivity to sample morphology when back-reflected (high diffraction angle) geometry is used [231], this property is favourable for inspecting a wide range of component shapes with minimal set-up change.

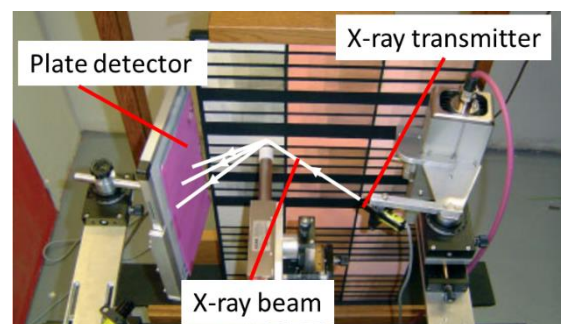


Figure 4.20 – Portable XRD equipment with a plate detector, after [230]

It is important to note that XRD comes with additional health and safety constraints compared to traditional NDT methods like ultrasonic testing, due to the ionizing radiation used. X-rays can cause significant harm to humans if dosage limits are exceeded. For this reason, XRD machines are typically highly shielded to prevent x-rays leaving the confines of the machine. Consequently, to use XRD within a machining centre would require the implementation of shielding into the machine, increasing the costs for the technique above the basic equipment costs. Standards for various applications of XRD can be found from the ISO [233], with regulations for x-ray health and safety set by governments [234].

4.7 Optical scattering

The phenomenon of the reflection of light waves from a surface forms the basis of optical scattering NDT. Incident light waves on the surface of a perfectly flat material are reflected at an angle equal to the incidence angle. By contrast, for a rough surface, the beam is scattered over a range of angles [235], as outlined in Figure 4.21. Optical scattering techniques are typically used for surface roughness measurements and it has been shown [236] that an optical system can be used for in-process monitoring of surface roughness during machining. The use of optical scattering methods for detecting surface roughness is covered in a series of standards [237].

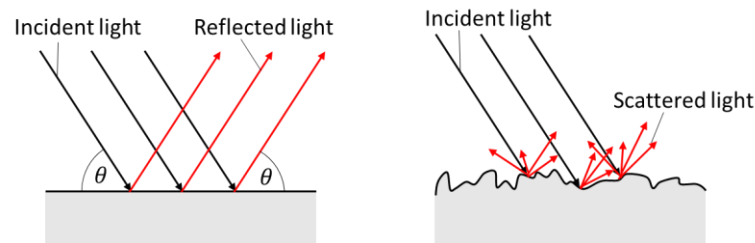


Figure 4.21 – Reflection of light from a smooth surface (left) and scattering of light from a rough surface (right)

In one study [238], an attempt was made to utilise optical scattering for the detection of a white layer on the surface of a hard turned steel. It was measured that the degree of optical scattering was greater from a surface machined with a worn tool compared to a new tool, particularly at high cutting speeds. It is claimed [238] that the increase in scattering is due to the presence of a white layer rather than an increase in surface roughness, which was similar across all the samples tested. This indicates that optical scattering may be appropriate as a test for white layers, however, only 4 samples and one type of alloy were investigated so further study is necessary to instil greater confidence in the presented results. Similar to AET, optical scattering would have a low cost to implement, due to the high technical maturity and

availability of equipment. However, the link between the physical properties of the white layer and the physical phenomenon behind the technique is not clear and requires further study.

4.8 Positron annihilation spectroscopy

Positron annihilation spectroscopy (PAS) is a technique in which positron-electron annihilation events within a test piece are used to determine the proportion of crystallographic defects, such as dislocations, that are present within the material. Due to their positive charge, positrons are repelled to the regions within a material which are furthest from atomic nuclei, such as vacancy sites and dislocations in the crystal lattice [142]. In these regions positrons annihilate with valence (outer shell) electrons, which have much lower momentum than inner shell electrons. This annihilation event produces two gamma-ray photons, as shown in Figure 4.22, which exit the material and can then be detected. Thus, in a test piece with a large number of vacancies and dislocations, positrons will tend to annihilate with valence electrons, whereas for a test piece with a more ideal crystalline structure, the annihilation of positrons will be with a greater proportion of higher energy, inner shell electrons. The spectrum of gamma-rays produced in the annihilation event is used to determine the nature of the collision [239].

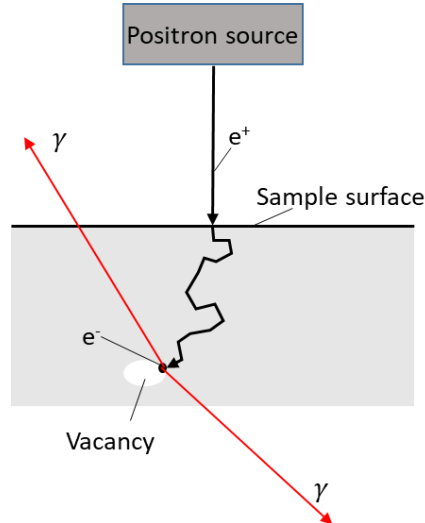


Figure 4.22 – Schematic of positron annihilation during PAS, resulting in the emission of two gamma-ray photons.

It has been shown that PAS can be used for the detection of general machining damage in Ti-6Al-4V [142] and stainless steel [239]. The ability to distinguish between damaged and undamaged samples was attributed to the increased number of dislocations in the near-surface, caused by deformation during machining. A higher number of dislocations changed the nature of positron annihilation events and broadened the gamma-ray spectrum. No attempt has been made to separate machining damage into

individual SI features such as a white layer and swept grain. It is also worth noting that PAS has been used to identify damage in a worn track [240], one of the common situations to find a white layer outside of machining. It should be noted that the implantation depth of positrons (the average depth to which they reach) in metals, varies significantly for different positron sources. For example, implantation depth is only 29 μm in steel for a ^{22}Na positron source [239] whereas it is 159 μm for a $^{68}\text{Ge}/^{68}\text{Ga}$ source, as such, appropriate choice of the positron source is necessary to tailor the inspection depth to the desired level. It is clear from the results presented in the literature, that PAS is inherently a surface inspection technique which may be suitable for white layer detection if the effect of other SI features can be removed and an appropriate positron source can be chosen which results in a small implantation depth in the workpiece material.

It is worth considering that in a white layer where SPD plus dynamic recrystallisation has occurred, there may be a lower concentration of crystallographic defects in the white layer due to recovery. As such further study is necessary to distinguish the effects of the white layer and the swept grain region. A major barrier to any implementation of PAS is the extra precautions that must be in place to protect the operator due to the ionizing gamma-radiation released during positron annihilation. Due to the lower technical maturity of PAS, compared to more established methods such as ECT, and the lack of industry standards, this technique is less accessible and would have an increased cost to implement.

4.9 Dye-penetrant inspection

In dye-penetrant inspection (DPI), a dye-penetrant, usually fluorescent in colour, is applied to the material and, through capillary action, is drawn into cracks in the test piece due to a combination of wall adhesion and surface tension [241]. After a dwell time, the excess penetrant on the surface is removed, leaving only the penetrant in the cracks. Using a developer, the penetrant is then drawn out of the cracks allowing the locations of the defect to be identified by the re-appearance of the penetrant. DPI is already widely used in aeroengine manufacturing to check for cracks in a broad range of components, however, the technique is not sensitive to any physical property of white layers.

4.10 Magnetic particle inspection

Magnetic particle inspection (MPI) is based on the phenomenon of magnetic flux leakage around a discontinuity, such as a crack, in a material [242]. The discontinuity is detected by introducing small

magnetic particles such as iron filings onto the surface of a magnetised test piece, the particles are then attracted to the area of flux leakage, highlighting the flaw location. MPI has an advantage over dye penetrant in that it can be used to detect both surface and slightly subsurface discontinuities, however, the technique is limited to ferromagnetic materials. The white layer is simply a region of different microstructure, rather than a mechanically separated section of material, therefore, MPI could not be used to detect white layers.

4.11 Thermography

Heat conduction in a solid material forms the basis of thermographic NDT methods. Anisotropy and discontinuities in the material disturb the flow of heat into the surrounding material, therefore a non-uniform flow of heat can be used to indicate the presence of an anomaly [243]. In another form of thermography, the level of blackbody radiation emitted by a uniformly heated material can be used to detect anomalous near-surface features [244], as a higher temperature is recorded above a discontinuity.

Laser thermography has been used as a non-destructive technique to identify surface-breaking defects in aluminium and steel [244]. The inspection method consists of a pulsed laser used to heat a spot of material. The consequent flow of heat, radially away from the spot as well as the temperature of the spot are both monitored and used to infer the presence of defects. Similar to DPI and MPI, thermography is more suited to the detection of discontinuities, however, if the white layer possessed significantly different thermal properties compared to a surface with no anomalous features, then it may be possible to separate between surfaces with and without white layer. It should be noted that significant temperature rises caused by heating during thermography could be detrimental to the surface integrity of the workpiece, due to the introduction of thermal SI features such as residual stresses [245].

4.12 Ranking the surface NDT methods

A critical analysis of all NDT methods discussed in this paper is presented in Table 4.1. The techniques have been assigned a usefulness score (**U**) based on the sensitivity of the physical phenomenon behind the technique to a physical property of the white layer. A technique based on a physical phenomenon that is not sensitive to the physical properties of white layers will score 1, whereas a technique with high sensitivity to one or more properties of white layers will score highly (5). A practicality score (**P**) was also assigned to each NDT method. This score was derived from the feasibility of using the technique for a

range of different materials and to carry out inspection in-process as part of a machining centre. A technique which has no material restrictions and is not reliant on a vacuum or large equipment would score highly (5). It should be noted that the **U** and **P** scores are aimed at guiding an individual or organisation that does not already possess considerable expertise and equipment resources in one particular field of NDT.

The product of these **U** and **P** scores were calculated and used to provide a quick measure of the potential of each technique in the application of white layer detection. **U*P** can be used to rank the techniques and identify which methods should be investigated further.

Table 4.1 – A summary of NDT methods and their applicability to the detection of white layers. The techniques are ranked from most to least appropriate according to their U*P score.

NDT Technique	Features detectable	Material limitations	U /5	P /5	U*P /25
Ultrasonic SAW testing	Cracks, Grain size, grain orientation	No limitations	5 This inherently near-surface technique can be used to detect grain orientation, differences in grain size and dislocation density, all useful for white layer detection.	4 No material limitations and due to the current automation of SRAS, SAW inspection may be transferable to a machining centre. Inspection depth can be tailored using SAW frequency.	20
X-ray diffraction	Diffraction domain size, grain orientation	No limitations	5 XRD can be used to detect grain orientation and differences in diffraction domain size, both useful for white layer detection. X-ray penetration depth is of the order of white layer thickness.	4 No material limitations and portable systems already exist. Health and safety precautions that would be required due to ionizing radiation limit a very high score.	20
Barkhausen noise inspection	Residual stress, hardened surface layers, grain size, grinding burn, white layer	Ferromagnetic materials	5 BN may detect white layers through association with large residual stresses, small grain sizes and hardness changes.	3 BN is suitable only for some steels. In-process monitoring may be possible. Improvements would be required in the resolution capability.	15

Table 4.1 continues on the next page

Table 4.1 continued

Positron annihilation spectroscopy	Lattice defects such as dislocations and vacancies	No limitations	4 PAS is very good for detecting severe plastic deformation which is fundamental to one of the formation mechanisms for white layers.	3 There are no material limitations but there would be difficulty implementing the technique due to the ionizing radiation produced and the lack of lack of portability.	12
Eddy current inspection	Cracks, Residual stress changes, grain texture, conductivity changes	Electrical conductors	4 ECT can be used for the detection of extreme residual stresses, changes in conductivity and grain texture. It has been shown that white layers often possess extreme residual stresses and have a refined grain structure. Further study is needed to investigate a change in conductivity in the white layer.	2 Residual stress profiling is applicable only to superalloys. Grain texture measurements are only suited to titanium alloys. Conductivity measurements can be carried out in all materials. Penetration depth can be improved through higher frequencies but this adds noise, particularly with rough surfaces. In-process monitoring may be possible.	8
Acoustic emission testing	Laps, smeared material, white layer*	No limitations	2 AET may be able to detect white layer formation from RMS measurements but there is difficulty in isolating other contributing factors to the measured signal.	4 AET is an inherently in-process inspection method but would require calibration for every change in tooling and cutting parameters.	8
Optical scattering methods	Roughness, white layer*	No limitations	1 There is no clear direct link to the physical properties of the white layer.	5 No material limitations and there is potential to implement in a machining centre.	5
Thermography	Thermal anisotropy, discontinuities	No limitations	1 Limited evidence of a direct link to the physical properties of the white layer.	4 No material limitations and the potential for on-machine inspection, although heating a component may alter the SI.	4
Dye-penetrant inspection	Surface breaking cracks	Non-porous materials	1 The technique is only suitable for crack detection.	4 There are material limitations but online inspection is unlikely.	4
Magnetic particle inspection	Surface breaking and slightly sub-surface cracks.	Ferromagnetic materials	1 The technique is only suitable for crack detection.	3 The technique is limited to steels and automated inspection would be difficult.	3

* Only one study claiming the detection of a white layer with this method.

4.13 Summary

In this chapter the NDT methods suitable for surface inspection were introduced and their underlying physical phenomena outlined. The current state-of-the-art is limited as only a small number of studies have investigated the non-destructive detection of white layers. However, aside from the Barkhausen noise work, these studies do not link results from inspection back to the physical properties of the white layer. There is therefore a gap in the literature for furthering the understanding of the link between the physical phenomena behind NDT techniques and the physical properties of white layers in titanium and other aeroengine alloys and the subsequent development of an appropriate testing method.

Based on the physical properties of white layers already reported in the literature, a preliminary assessment of surface non-destructive evaluation methods has been made in this chapter. SAW, XRD and Barkhausen noise methods appear to be the most suited to white layer detection and the theory behind these methods was discussed in more detail. However, further characterisation work, identified in Chapter 3, must first be undertaken to fully understand white layers, particularly in titanium, and make a more informed prediction of a viable approach to implementing these three NDT methods. The next chapter describes the methods employed in the generation and characterisation of white layers in aeroengine alloys to fill the gaps identified in the literature.

5 Generation and characterisation of machining induced white layers

This chapter describes the methods employed in the generation and characterisation of machining-induced white layers in three aeroengine alloys, Ti-6Al-4V, IN-718 and SCMV, selected based on their relevance to aeroengine applications. It was highlighted in the literature review that further work was required to expand the understanding of the formation and physical properties of white layers in titanium alloys. A nickel superalloy and a steel alloy have been investigated concurrently to produce samples to aid the development of a non-destructive testing method. It has already been identified that NDT techniques will interact with these materials in different ways, most noticeably between ferromagnetic and paramagnetic alloys, so it was necessary to investigate alloys with different mechanical and physical properties.

5.1 Materials

5.1.1 Titanium-based alloy Ti-6Al-4V

Mill-annealed Ti-6Al-4V in the form blocks all cut from the same cross-rolled plate was used for white layer generation. This alloy was selected due to its widespread use in aeroengines. In cross-rolling, as opposed to unidirectional rolling, the material is rolled in one direction on the first pass, then on the subsequent pass, the rolling direction is perpendicular to the first. This is repeated for the desired number of rolling passes. The specimens for the machining trials were sectioned so that their longest axis lay parallel to the final roll direction (FRD), rather than the cross-rolled direction (CRD), as shown in Figure 5.1 (a). The microstructure of the as-received material consisted of 10-20 μm sized primary α -grains with residual grain boundary β , as highlighted in Figure 5.1 (b). The volume fraction of the residual β -phase was estimated to be 10-20% using particle analysis techniques on Fiji ImageJ software [246]. The chemical composition and mechanical properties of Ti-6Al-4V are summarized in Table 5.1 and Table 5.2.

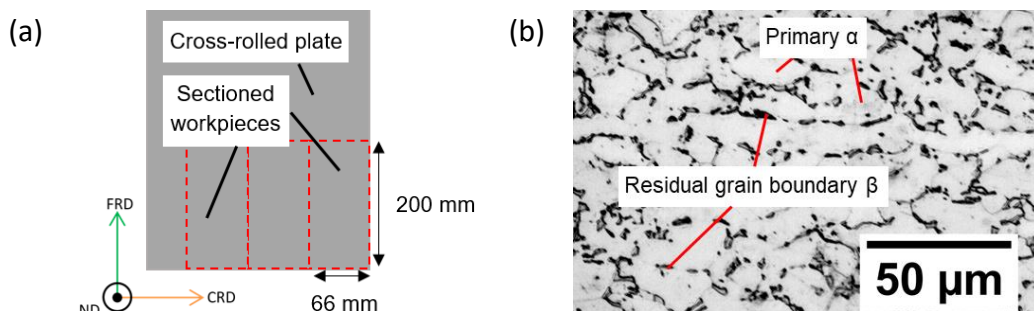


Figure 5.1 – Schematic of the workpieces obtained from a cross-rolled plate (a) and a cross-sectional micrograph of the as-received material (b).

Table 5.1 - Minimum and maximum alloy additions in Ti-6Al-4V [11].

Element	Al	V	Fe	O	C	H	N	Y
Wt%	6	4	0.3	0.2	0.08	0.01	0.03	50ppm

Table 5.2 – Mechanical properties of Ti-6Al-4V, after [11].

UTS (MPa)	$\sigma_{0.2\%}$ (MPa)	Hardness (HV)	Density (g/cm ³)
900-1160	830	340-360	4.33

To better understand the effect of initial texture on the formation of white layers, titanium workpieces with alternative crystallographic textures, including strongly textured unidirectional (U/D) rolled plate and comparatively un-textured titanium produced from powder feedstock via field-assisted sintering technology (FAST) [247], were machined. In FAST a direct current is applied to the mould allowing rapid heating of the powder which consolidates under high pressure through diffusion bonding. Figure 5.2 (a) and (b) show the equiaxed grain structure of the U/D plate and the lamellar microstructure of the FAST titanium, respectively. Additional U/D material was also used for trials investigating strain measurement in machined surfaces.

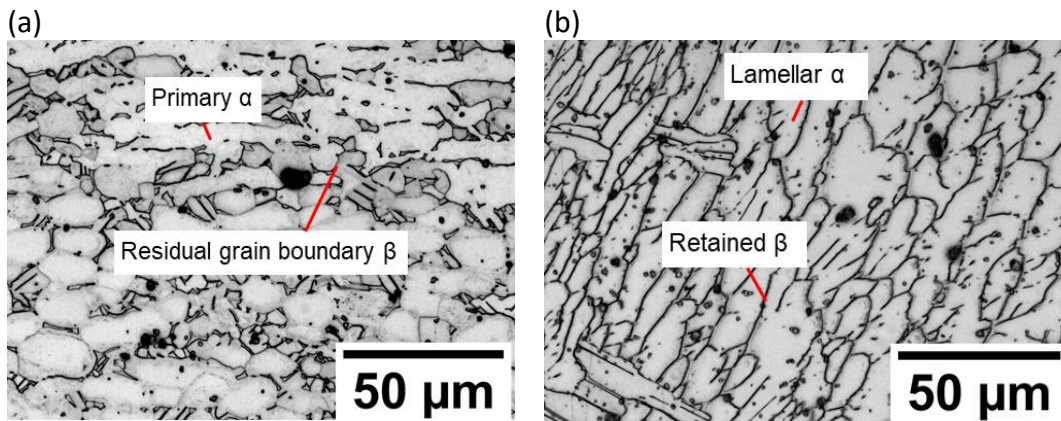


Figure 5.2 – Micrographs of the bulk microstructure in the (a) U/D plate and (b) FAST titanium.

5.1.2 IN-718

IN-718, a common aeroengine disc alloy, was used for generating white layer samples in a nickel superalloy. The microstructure of the as-received material consisted of FCC γ' and γ'' within a FCC γ matrix, as shown in Figure 5.3 (a). The orthorhombic δ -phase was visible at the grain boundaries. The chemical composition and mechanical properties of IN-718 are summarized in Table 5.3 and Table 5.4.

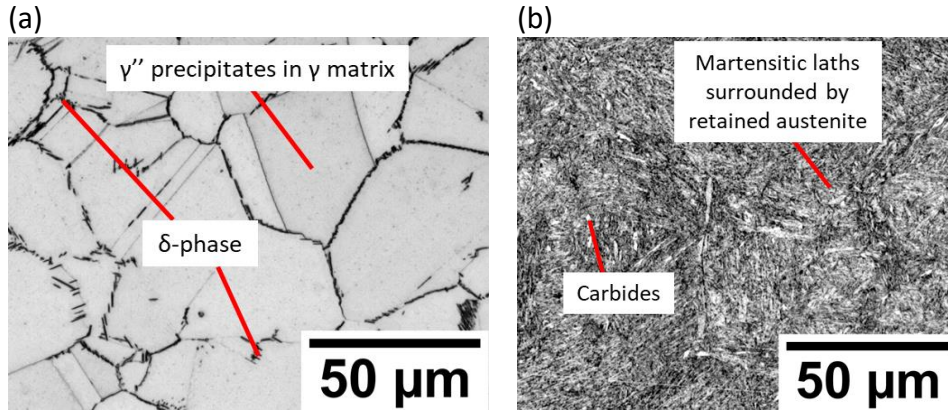


Figure 5.3 – Micrographs of the bulk microstructure in (a) IN-718 and (b) SCMV.

Table 5.3 – Composition of IN-718, after [20].

Element	Ni	Fe	Cr	Nb	Mo	Al	Ti	C
wt%	52.5	18.5	19	5.1	3.0	0.5	1.01	0.08

Table 5.4 – Mechanical properties of IN-718, after [248].

UTS (MPa)	$\sigma_{0.2\%}$ (MPa)	Hardness (HV)	Density (g/cm ³)
1310	1110	332	8.19

5.1.3 SCMV

SCMV was used for white layer generation in a steel alloy due to its application as an aeroengine shaft alloy. The tubes used in the machining trials were all cut from a single low-pressure turbine shaft. The microstructure of the as-received material was martensitic, with high aspect ratio martensite laths surrounded by retained austenite and the presence of fine carbide precipitates, as shown in Figure 5.3 (b). The chemical composition and mechanical properties of SCMV are summarized in Table 5.5 and Table 5.6.

Table 5.5 – Composition of SCMV, after [28].

Element	C	Cr	Mo	Va	Si	Mg	Fe
wt%	0.35-0.43	3.0-3.35	0.80-1.10	0.15-0.25	0.10-0.35	0.40-0.70	bal

Table 5.6 – Mechanical properties of SCMV, from MSRR6119 [30].

UTS (MPa)	$\sigma_{0.2\%}$ (MPa)	Hardness (HV)	Density (g/cm ³)
1470-1670	1220-1370	450-520	7.8

5.2 White layer generation

Machining trials were carried out to generate surfaces with a white layer in addition to surfaces that would pass SI inspection, to build a database of samples that could be used for characterisation of white layers and development of an NDT method for their detection. Milling was selected for the machining of Ti-6Al-4V and IN-718 since a flat surface would make subsequent NDT measurements easier, as part access and probe contact are more difficult for non-planar surfaces. However, for the SCMV material, turning was used, as parts manufactured from SCMV are used solely as shafts in aeroengines and therefore turning is the only relevant choice. Additional orthogonal milling trials, for measuring strain in the machined surface during white layer formation using the micro-grid method were carried out in Ti-6Al-4V. Orthogonal milling was selected for these trials to facilitate plain strain conditions for the plane on which the micro-grids were printed, this is explored further in section 5.2.3.

5.2.1 Square-shoulder milling trials

Ti-6Al-4V and IN-718 were milled on a Mori-Seiki Nv5000 α 1 vertical machining centre using a Seco Tools AB shoulder-milling cutter (R217.69-2020.0-12-2AN) and coated tungsten carbide inserts (XOEX120408R-M07 MS2050). Although the tool holder could hold two inserts, only one cutting insert was for the trial to simplify the cutting force analysis and negate the effect of tool run-out. A Kistler 9139AA multicomponent force dynamometer was used to measure the forces during cutting at a sampling frequency of 1000 Hz, chosen to provide at least 10 measurement points during one tool rotation. Hocut 795B coolant was used in these trials at a pressure of 6 bar.

A number of the inserts were ground, by Seco Tools AB, on their flank face to result in two different levels of flank wear ($V_B = 0.5$ mm and 0.85 mm), as shown in Figure 5.4 (a) and (b), to facilitate the controlled generation of white layers in both alloys. The artificial wear resulted in increased forces during cutting, particularly in the thrust direction. Previous studies have shown that as a tool wears and a white layer is formed, the forces increase, with the thrust force increasing the most [75, 249]. It should be noted that the wear on the ground inserts was beyond the 0.2 mm average flank wear criterion typically used to define tool-life in industry, however, the inserts were used exclusively for generating samples with thick white layers. The grinding was performed such that the resultant wear land would contact the workpiece surface leading to frictional contact between the flank face and the workpiece. This was designed to cause rubbing of the workpiece surface without chip formation, during the second half of the tool rotation, as

shown in Figure 5.5. After grinding, the inserts were re-honed to provide a nose radius of 800 μm and a cutting-edge radius of approximately 30 μm , in line with the off-the-shelf inserts. The cutting-edge radius was verified before the cutting trials using an Alicona InfiniteFocusSL microscope to ensure consistency across all trials. The z-position of the tool in the machining centre was corrected for the trials with worn inserts, to guarantee a consistent depth of cut despite the removal of insert material during the production of the wear land.

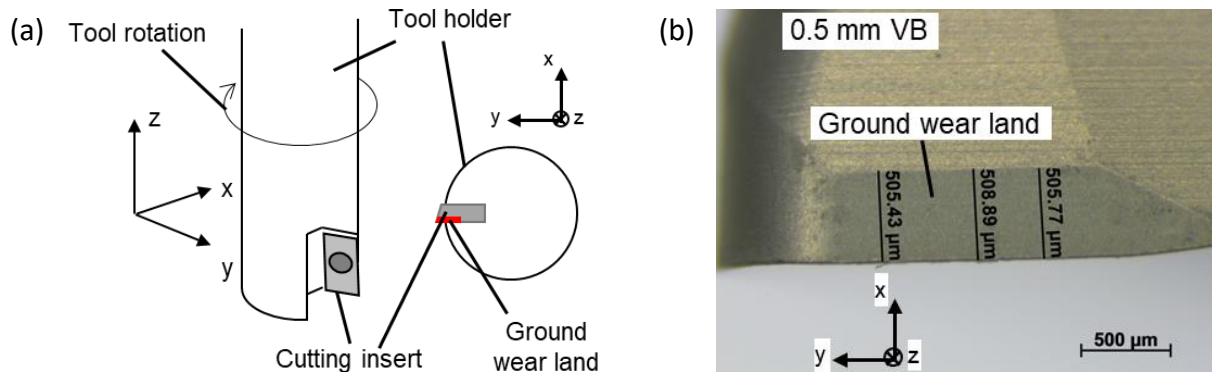


Figure 5.4 – A schematic (a) outlining the area on the cutting insert which has been ground to produce an artificially worn region and a macrograph (b) of the ground wear land for a 0.5 mm worn insert.

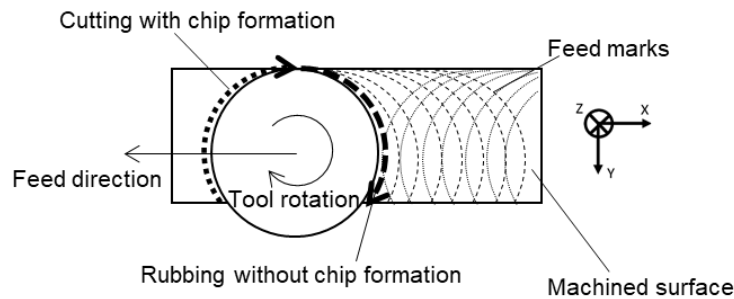


Figure 5.5 – A schematic of the cutting operation highlighting the rubbing of the workpiece by the worn cutting insert which occurs during the second half of the tool rotation.

For both the titanium and nickel trials, a range of cutting speeds (20-200 m/min) were imposed in an attempt to achieve machining conditions with dominant thermal effects (higher speeds) and mechanical effects (lower speed) on the workpiece material. These correspond to an increased likelihood of generating white layers through the PT and SPD mechanisms, respectively, as reported by Hosseini et al. [250]. Slower cutting speeds were necessary when machining with the artificially worn inserts to reduce their failure rate under the high temperatures and forces caused by the frictional contact of the artificial wear regions on the workpiece. Full cutting conditions for all machining trials are given in the appendix, however, key parameters are given when presenting any results from specific samples in the subsequent chapters.

A schematic of the designed cutting setup for square-shoulder milling of Ti-6Al-4V is shown in Figure 5.6 for the 200 x 66 x 20 mm workpieces. The feed was varied between 0.06 and 0.12 mm/tooth and the radial depth of cut was kept constant at 16 mm. Two levels, 2 mm and 0.5 mm, were implemented for the axial depth of cut to represent roughing and finishing cuts respectively. The cutting trials with new cutting inserts were conducted under both dry and wet conditions, wherein the coolant was applied under a conventional flood-cooling regime, to enhance and suppress thermal effects respectively. The trials using ground inserts could only be carried out with coolant, due to excessive heating when machining dry that led to tool failure. The workpieces were mounted directly to the force dynamometer using two M10 bolts through pre-drilled holes.

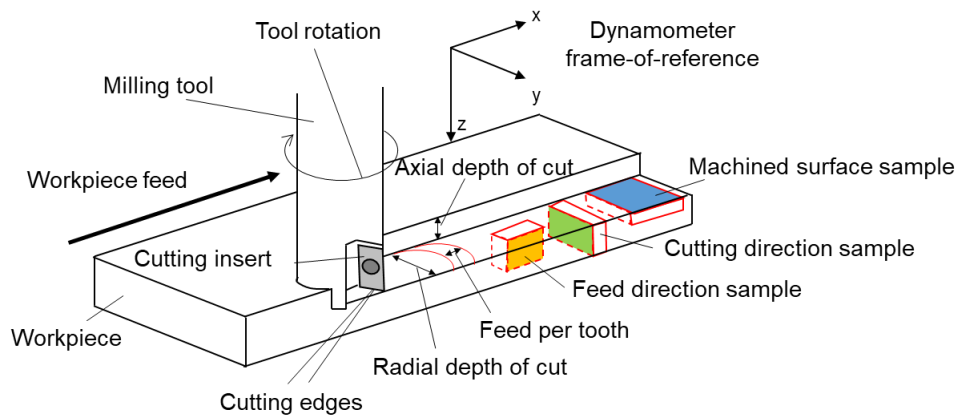


Figure 5.6 - Schematic of the square shoulder milling operation for the titanium workpieces.

In the small-scale effect-of-texture study in Ti-6Al-4V, a square shoulder milling operation was carried out using identical cutting parameters to the white layer generation trials but with a constant cutting speed of 40 m/min. The workpieces were smaller than the previously described titanium trials, so the cutting set-up resembled that shown in Figure 5.7, with the workpiece (50 x 20 x 20 mm) held on a machine-bed vice mounted on the dynamometer. The unidirectional rolled workpieces were machined on three different faces, rolling direction (RD), normal direction (ND) and transverse direction (TD), as these each exhibit different textures relative to the surface being machined. The FAST titanium was machined on the axial face and the circumferential face, relative to the circular billet from which the workpieces were sectioned. These orientations are highlighted in Figure 5.8.

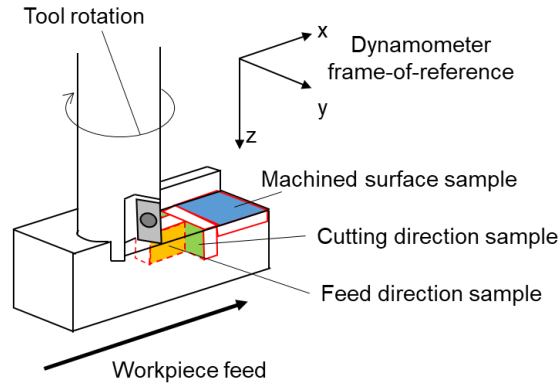


Figure 5.7 – Schematic of the square shoulder milling operation for small-scale effect-of-texture-study in titanium as well as machining trials using IN-718 workpieces.

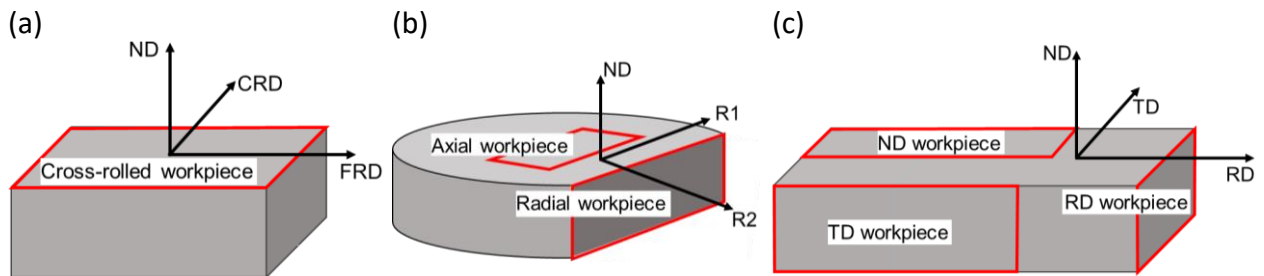


Figure 5.8 – Schematics of the workpiece orientations for the effect-of-texture trials showing (a) the cross-rolled plate, (b) the FAST workpiece and (c) the U/D plate. The surfaces machined for different workpiece orientations are highlighted in red.

A similar cutting set-up as shown in Figure 5.7 was used for the IN-718 machining trials, with a lower feed, compared to Ti-6Al-4V, of 0.03 mm/tooth and a single axial depth of cut of 0.5 mm used, due to the tendency for this superalloy to strain-harden which led to tool failure at more aggressive cutting conditions. After each cut, the workpiece (55 x 25 x 25 mm) was rotated 90° about the x-axis and machined on a new face.

5.2.2 Turning trials

Turning of a SCMV tube (127 OD x 105 ID x 137 mm) was carried out on a Cincinnati Hawk 300 lathe using a PDJNL2020K15JETL tool holder in combination with DNMG 150608 coated carbide inserts, both provided by Seco Tools AB. A schematic of the experimental setup is shown in Figure 5.9. Hocut 795B was used as the metal working fluid and was delivered at a high pressure of approximately 70 bar. Cutting forces were recorded with a Kistler 9121 lathe dyno, mounted behind the tool holder, at a sampling rate of 20,000 Hz. This sampling rate was selected to encapsulate any high-frequency dynamic effects during machining, for example chatter, based on experiences during the milling trials.

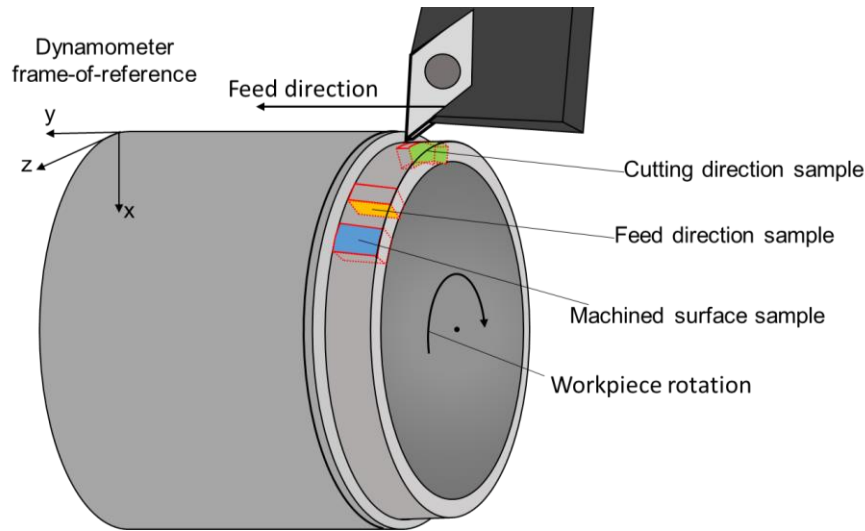


Figure 5.9 – A schematic of the SCMV turning operation.

Similar to the milling trials, a number of the turning inserts were pre-ground by Seco Tools AB to introduce artificial wear at levels of 0.15 mm, 0.25 mm and 0.5 mm on the flank face, as shown in Figure 5.10 (a) and (b). The increased number of artificial wear levels employed in these trials, compared to the milling trials, were selected to encourage the generation of a broader spectrum of thinner white layers with different thicknesses, based on experience gained from the milling trials. To ensure the contact between the ground wear land and the workpiece required for rubbing, the wear land was ground at an angle greater than 6° from the flank face as shown in Figure 5.10 (c). Thus, when the insert was mounted in the tool holder, which had a rake angle of -6° , the ground wear land could still rub the surface, accounting for any additional rotational deflection under the action of cutting forces and the workpiece curvature. The x-position of the tool in the machining centre was again adjusted for the trials with worn inserts to ensure a consistent depth of cut across all trials. The cutting edges were re-honed following grinding to approximately $80\text{ }\mu\text{m}$ and verified with measurement on an Alicona Infinite SL microscope. For the trials, only inserts with cutting edge radii between $72\text{--}82\text{ }\mu\text{m}$ were selected in an attempt to minimise the effect of cutting edge radius on the results. To generate a broad spectrum of surfaces for characterisation and NDT, cutting speeds between $60\text{--}180\text{ m/min}$ and tool wear levels between $0\text{--}0.5\text{ mm}$ were used. The depth of cut and feed were held constant at 0.5 mm and 0.15 mm/rev respectively.

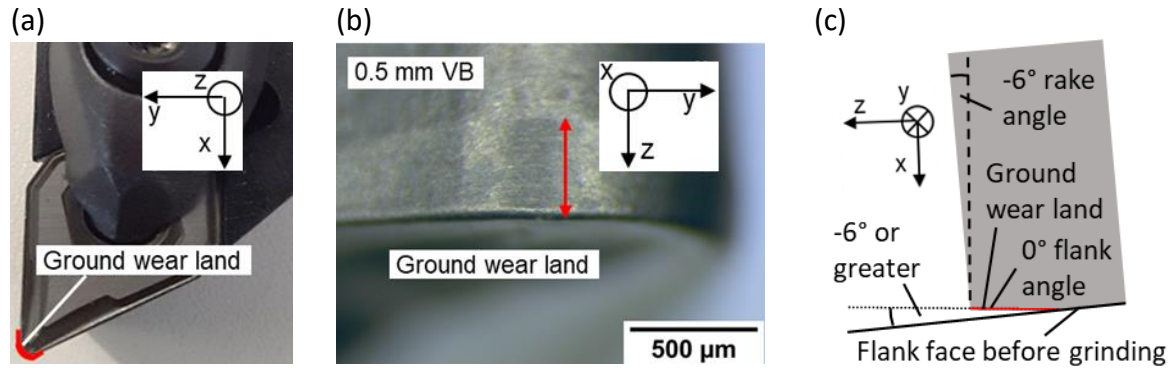


Figure 5.10 – Macrographs of (a) the insert in the tool holder with the ground wear land highlighted, (b) a 0.5 mm ground wear land and (c) a schematic demonstrating the angle at which the artificial wear was ground into the flank face to ensure rubbing with the workpiece surface.

5.2.3 Orthogonal milling

To measure the strain in the machined surface during white layer formation, orthogonal milling of Ti-6Al-4V was carried out on a DMG Mori NVX5080 machining centre using a Seco Tools R220.17-0040-16 tool holder, modified for single-point milling, and TPUN160308 HX coated carbide inserts. Hocut 795B was again used as the metalworking fluid and was delivered at 6 bar. A number of inserts were ground by Seco Tools AB, on their clearance face, to produce an artificial 0.5 mm wear land with the cutting edge re-honed to 85 µm in half of these inserts and left at 10 µm for the remainder. Insert grinding was used as the desired level of control over the tool geometry could not be achieved using naturally worn inserts. This variation in cutting edge allowed a controlled investigation into its effect on surface integrity and strain as the impact of cutting edge radius was purposely blocked in the machining trials described earlier in this chapter. An insert worn naturally through repeated cutting was also used to facilitate a comparison with the artificially worn inserts. The different inserts used in the trials are summarised in Table 5.7. Cutting speeds were selected to mirror those from the previously described milling trials in Ti-6Al-4V. To ensure variation in uncut chip thickness over the tool engagement, from smaller than, to larger than the cutting edge radius, a feed of 0.5 mm per tooth was selected. This corresponded to a maximum uncut chip thickness of 0.105 mm.

Table 5.7 – The cutting inserts used during the orthogonal milling trials.

Insert type	Wear land (mm)	Cutting edge-radius (µm)
Unworn	0	30
Artificially worn	0.5	10-15
Artificially worn and re-honed	0.5	80-85
Naturally worn	0.265	137

To allow orthogonal milling, i.e. milling on a single plane with the cutting edge perpendicular to the cutting velocity, a special workpiece jig was used to hold two 25.5 x 24.5 x 4 mm plates such that at the interface between the two plates, the region on which grids were printed was under plane strain conditions. The cutting set-up is illustrated in Figure 5.11 (a), (b) and (c). Both climb and conventional milling were used to compare the effects of thick-to-thin and thin-to-thick chip formation respectively.

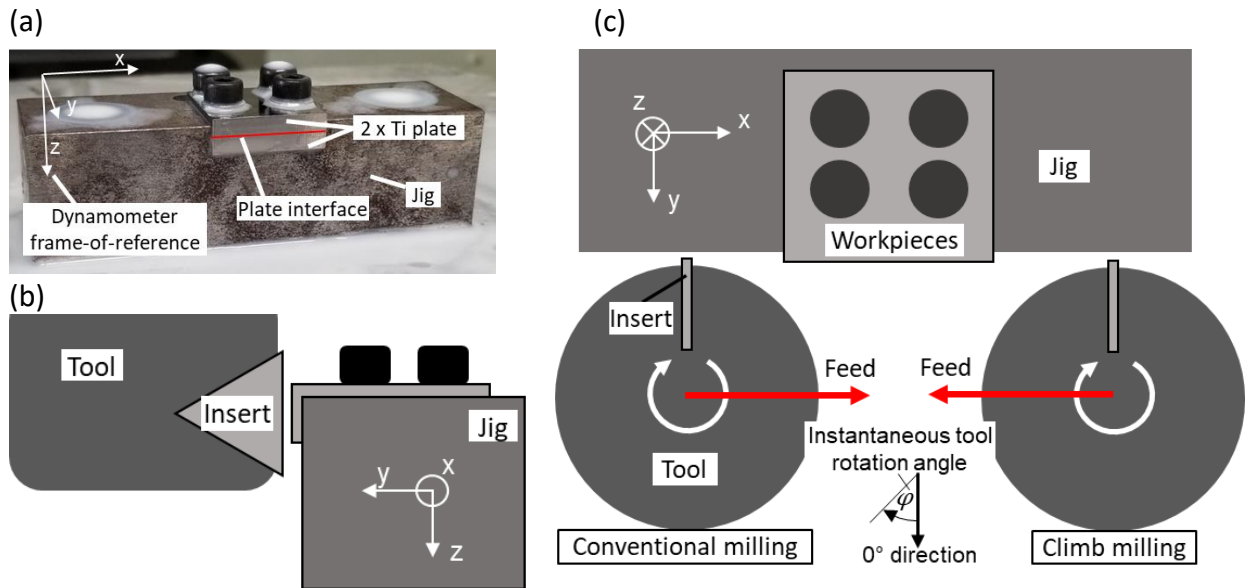


Figure 5.11 - (a) The workpiece and jig mounted in the machining centre. Schematics of the cutting operation in (b) the y-z plane and (c) the x-y plane.

5.2.4 Cutting force analysis

Cutting forces were recorded during all machining trials to help understand the results from destructive characterisation, particularly changes in crystallographic texture. By monitoring the cutting forces during the full cut, the uniformity of the surface integrity could be predicted and used to aid the selection of destructive cut-ups and NDT regions. When forces remained constant over the length of the cut, subsequent optical microscopy indicated that the surface integrity was unchanged over the length of the machined surface. Therefore, in this case NDT could be carried out at any point on the machined surface and the region under inspection would be representative of the whole test piece. Measurement of the out-of-plane forces during orthogonal milling could be used to ensure plane strain conditions were maintained.

During turning, the dynamometer used to measure forces was located behind the tool holder meaning cutting forces were measured in the cutting tool frame-of-reference. By contrast, in milling, the

dynamometer was situated beneath the workpiece so geometrical relationships were used to convert from the dynamometer frame-of-reference (F_x and F_y) to a cutting tool frame-of-reference (F_c and F_r). A cutting tool frame-of-reference is beneficial as it more clearly shows the dependence of cutting forces on the instantaneous immersion angle (φ) during milling. The dynamometer frame-of-reference and cutting tool frame-of-reference are shown in Figure 5.12. Both force pairs, F_x and F_y as well as F_c and F_r lie on one plane and are orthogonal so the relationship stated in Eq. 5.1 applies.

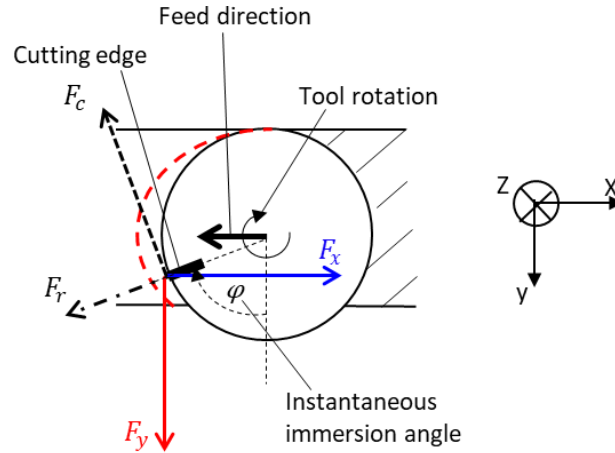


Figure 5.12 – Schematic of a square-shoulder milling operation highlighting the relationship between the cutting and radial forces, which have a cutting tool frame-of-reference, and the x and y forces measured by a dynamometer.

$$F_{resultant}^2 = F_x^2 + F_y^2 = F_{cutting}^2 + F_{radial}^2 \quad \text{Eq. 5.1}$$

By using the instantaneous immersion angle, equations for forces in the cutting and radial directions can be stated, as in Eq. 5.2 and Eq. 5.3. Note the minus signs arise due to the axis convention outlined in Figure 5.12.

$$F_c(\varphi) = -F_x \cos(\varphi) - F_y \sin(\varphi) \quad \text{Eq. 5.2}$$

$$F_r(\varphi) = -F_x \sin(\varphi) + F_y \cos(\varphi) \quad \text{Eq. 5.3}$$

From classical models for the mechanics of oblique cutting, equations for the prediction of cutting forces during milling can be stated [42], as in Eq. 5.4, Eq. 5.5 and Eq. 5.6, as a function of the instantaneous chip thickness t_i , which is itself a function of the tool immersion angle φ .

$$F_c(\varphi) = K_{tc} a_p t_i(\varphi) + K_{ce} a_p \quad \text{Eq. 5.4}$$

$$F_r(\varphi) = K_{rc} a_p t_i(\varphi) + K_{re} a_p \quad \text{Eq. 5.5}$$

$$h(\varphi) = t_1 \sin(\varphi) \quad \text{Eq. 5.6}$$

K_{cc} and K_{rc} are cutting coefficients for shearing forces in the cutting and radial directions, K_{cc} and K_{rc} are the cutting coefficients for ploughing forces in the cutting and radial directions. Ploughing forces arise due to the effect of a small chip thickness in relation to the cutting edge radius (t_1/r). With a small t_1/r a chip does not form, instead, the workpiece material is ploughed underneath the tool.

It can be seen from Eq. 5.4, Eq. 5.5 and Eq. 5.6, that the cutting and radial forces are dependent only on the immersion angle, except for small chip thicknesses. These cutting tool frame-of-reference forces are easier to visualise than the forces measured directly by a force dynamometer, which continuously vary in magnitude and sign due to the curved cutting path on the x-y plane. These differences are immediately apparent in force-time plots, as shown in Figure 5.13 (a) and (b).

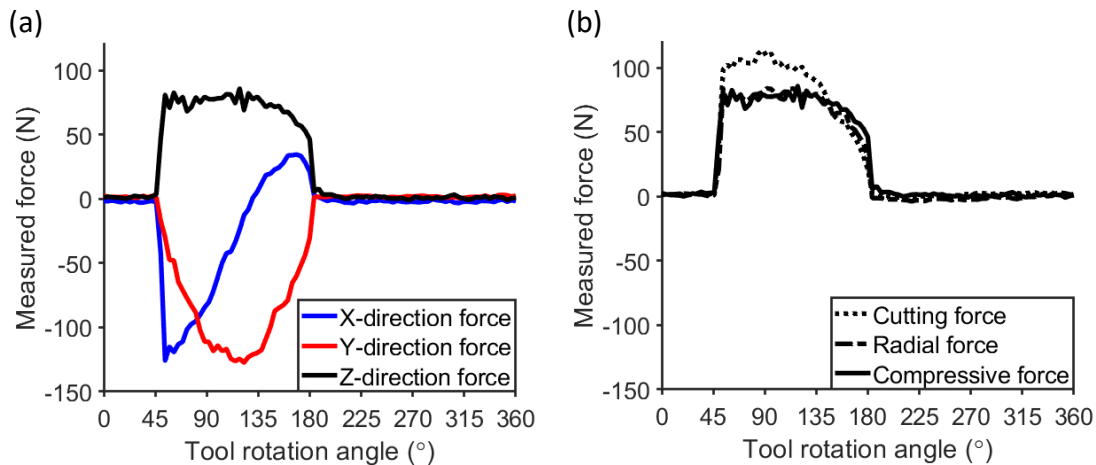


Figure 5.13 – Cutting forces during one tool rotation from (a) the dynamometer-frame-of-reference and (b) the cutting tool frame-of-reference.

5.2.5 Surface roughness

Surface roughness measurements were undertaken on all machined surfaces to allow the effect of surface topography on NDT methods to be quantified. Measurements, using a Mitutoyo Surftest SJ-210 portable surface roughness profilometer, were taken parallel to the feed directions for both the milled and turned surfaces. A sampling length of 0.8 mm and an evaluation length of 4 mm were used in the measurement of average surface roughness (R_a), the average deviation of the surface profile from the centreline according to the BS1134 standard [251]. On each surface, five measurements were taken and averaged.

5.3 Methods for machined surface characterisation

5.3.1 Sample preparation

Following machining, workpieces were sectioned into smaller samples to facilitate characterisation work. Three types of samples were obtained, machined surface, cutting direction (CD) and feed direction (FD), as shown previously in Figure 5.6, Figure 5.7 and Figure 5.9 for each material. Cutting and feed direction samples were used for characterisation of the cross-section, with both directions being necessary to build a complete picture of surface integrity, which can vary across a surface. Machined surface samples were used for the characterisation of the top surface. The Ti-6Al-4V and IN-718 workpieces were sectioned, in the first step, using a manually operated Buhler Abrasimet equipped with Silicon Carbide cut-off blades. Due to their larger size, the SCMV workpieces were initially sectioned using wire electrical discharge machining to reduce the sample size. Following preliminary sectioning, all materials were reduced to their final dimensions using a Struers Secotom-50 precision sectioning machine. A silicon carbide cut-off wheel was used for Ti-6Al-4V and IN-718 whereas an aluminium oxide cut-off wheel was used for SCMV, as per manufacturer recommendations.

Following sectioning, feed and cutting direction-samples were mounted in PolyFast conductive resin using a Buehler SimpliMet 3000 mounting press. A conductive mounting medium was used so that all samples could be examined in a SEM if needed. To produce a smooth surface for characterisation, samples were ground and polished using a Buehler Automet automatic polishing machine. Grinding using papers with successively finer grits (P400, P800, P1200, P2500 all for 1 min) was undertaken to remove the deformation introduced by sectioning. Samples were then polished using a Struers MD Chem polishing pad in combination with colloidal silica suspension for 15 minutes to achieve a mirror finish suitable for microscopy. A final 2 minutes of polishing with water only, was used to clean the colloidal silica from the polished surface. A separate polishing pad was used for each alloy and samples were typically polished in groups of four or six samples of the same material.

To reveal the microstructure for optical microscopy, polished samples in each material were etched using a chemical etchant. For Ti-6Al-4V, Kroll's reagent (HF (40% vol) 2 ml, HNO (69% vol) 6 ml, H₂O 92 ml) was used as this reagent preferentially attacks the alpha phase, providing contrast between the two phases in titanium. Kroll's reagent is particularly toxic and requires special consideration relative to other

etchants, however, it is the only etchant which reliably etches titanium alloys. IN-718 was etched using Kalling's reagent (copper (II) chloride 5g, ethanol 100 ml, HCl (37%) 100 ml) to reveal grain boundaries. Nital (HNO₃ (68%) 2 ml, Industrial methylated spirit 98 ml) was used to reveal the grain structure in SCMV steel. The SCMV and IN-718 samples had to be etched immediately following the final polishing step otherwise a slow and inconsistent etch response was encountered.

In optical microscopy analysis and subsequent non-destructive testing, a pass/fail system is used to indicate:

- Anomalous surfaces that would fail SI inspection (i.e. the surface contains a white layer or critical levels of swept grain). ❌
- Surfaces that would pass SI inspection. ✅

5.3.2 Heat treatment for white layer grain growth

A small subset of Ti-6Al-4V samples were heat treated at 700 °C for 2 hours using a vacuum furnace. This heat treatment was designed to grow the fine grain structure, typically encountered in the white layer, to allow the α and β -phase content of the white layers to be inspected. A vacuum furnace was used to prevent oxidation of the titanium and inhibit alpha-case formation in the near-surface. A temperature of 700 °C was used as it lies below the β -transus temperature, so it prevents precipitation of the β phase.

5.3.3 Microscopy methods for microstructural observations

An Olympus BX51 microscope was used to capture optical micrographs of etched surfaces at 500x magnification, for consistency with industry inspection methods [160]. The micrographs, as shown in Figure 5.14 (a), were decomposed into three distinct sections including, the white layer, the deformed region beneath, termed the swept grain region, and the bulk, according to the criteria given by Puerta-Velásquez et al. [65]. The white layer region was taken as the region in which the grain structure was not resolvable for each of the alloys. The swept grain region was deemed to finish at the depth beneath the surface where grain distortion was indistinguishable from the bulk microstructure.

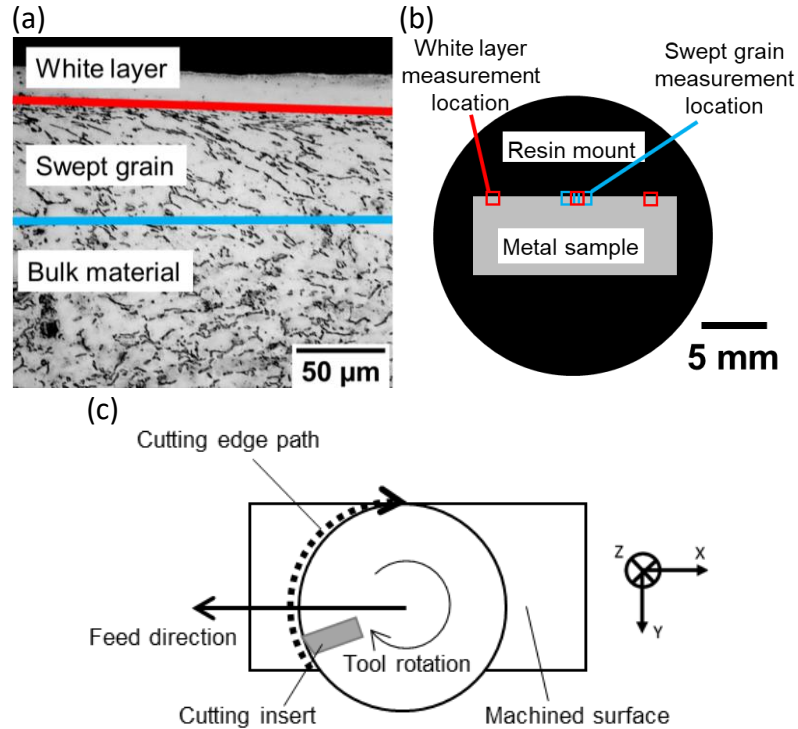


Figure 5.14 – A cross-sectional micrograph (a) of a Ti-6Al-4V machined surface outlining the three distinct microstructure regions and schematics that highlight (b) the measurement locations on a cross-sectional cutting direction sample and (c) the curved path of the cutting edge during milling.

The white layer thickness was calculated as an average of 15 measurements across 3 micrographs taken from different locations along each sample, as shown in Figure 5.14 (b). Within every micrograph, each thickness measurement was taken at the same 5 locations across the width of the image. To calculate the average white layer thickness for a particular machined surface, the values across FD and CD samples from the start and end of the cut were averaged. Due to the curved tool path in milling, as shown in Figure 5.14 (c), the direction of grain distortion changes across the width of the machined shoulder. Therefore, all the swept grain depth measurements were obtained from an average of 10 measurements at two locations near the centre of the cross-sectional sample, as shown in Figure 5.14 (b). At these locations, the tool pass direction and therefore the grain distortion was in the plane of the image. Similarly, for the turned SCMV surfaces, all swept grain measurements were carried out on CD samples, where grain distortion was again in the plane of the image.

High-resolution microscopy was carried out on samples from each material using an Inspect F50 field emission gun SEM (FEG-SEM). Due to the poor etch response of the white layer, highlighted in the literature review, FEG-SEM imaging was carried out on polished but un-etched samples using back-

scattered electrons for Ti-6Al-4V and SCMV. In these SEM images, the contrast between different grains of the same phase arises due to crystal orientation effects (channelling contrast) [252] and due to atomic number contrast for different phases [253]. In IN-718, imaging etched samples proved to be more successful. A small spot size and an accelerating voltage of 10 kV were used to enhance the resolution of the ultrafine grains within white layers in all materials. By reducing the spot size, the size of the electron beam incident on the sample surface is reduced. Similarly, using a 10 kV accelerating voltage rather than 20 kV, for example, reduces the energy of incident electrons, limiting their penetration into the sample. The combined effects of a smaller accelerating voltage and probe size improve the resolution by reducing the overall interaction volume from which electrons are detected. It should be noted that further reductions of accelerating voltage and spot size lowered the signal from the sample, decreasing the signal to noise ratio.

5.3.4 Nanoindentation measurements

To determine the hardness and elastic modulus variation below the machined surface, cross-sectional profiles were obtained for all three alloys using a Triboscope nanoindenter, through the Oliver & Pharr method [254]. A plot of a typical loading cycle during nanoindentation is outlined in Figure 5.15 with hardness is calculated using Eq. 5.7.

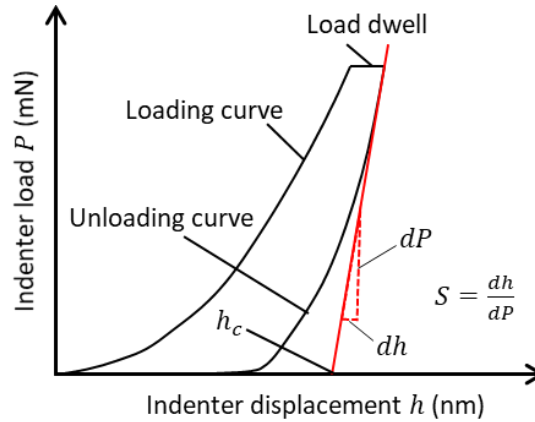


Figure 5.15 – A schematic of a typical load-displacement plot used in the Oliver and Pharr method, after [254].

$$H = \frac{P_{max}}{A_p} \quad \text{Eq. 5.7}$$

In Eq. 5.7 H is the nanohardness, P_{max} is the maximum indentation load and A_p is the projected area of the indentation. For an indenter with a regular geometry, the projected indentation area is a function

of the contact depth, h_c . Due to the wear of the indenter tip, which will change its shape with use, a new area function is calibrated regularly to ensure valid measurements.

Nanoindentation can also be used to measure the elastic modulus by first calculating the stiffness, S , of the unloading portion of an indentation loading cycle, as shown in Figure 5.15. The reduced elastic modulus, E_r , can then be calculated using Eq. 5.8.

$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \quad \text{Eq. 5.8}$$

Where A_c is the contact area of the indentation and, similar to A_p , is a function of the contact depth for a known indenter shape. To calculate the elastic modulus, E , from E_r , Eq. 5.9 is used.

$$\frac{1}{E_r} = \frac{(1 - \nu^2)}{E} + \frac{(1 - \nu_i^2)}{E_i} \quad \text{Eq. 5.9}$$

Here, E and ν are the Elastic modulus and Poisson's ratio of the material being tested and E_i and ν_i are the Elastic modulus and Poisson's ratio of the indenter material. To obtain the hardness profiles, a 5000 μN load was applied with a Berkovich indenter on mirror-polished cross-sectional samples using an 11 x 5 indentation array, 50 μm x 25 μm in size with eleven indentations in the depth direction, as shown in Figure 5.16 (a). A mirror-finish was required due to the sensitivity of nanoindentation to variations in surface height. A 40-second drift compensation measurement was carried out before every indentation. Hardness and elastic modulus values were averaged for each of the five indentations at every depth interval. An atomic force microscope (AFM) was used to position the indenter adjacent to the machined surface edge. An AFM image of a machined surface, post-indentation, is shown in Figure 5.16 (b), the top edge of the sample (the machined surface) is easily identified by a step-change in surface height, resulting from the higher material removal rates in the PolyFast mount, relative to the metallic sample, during polishing.

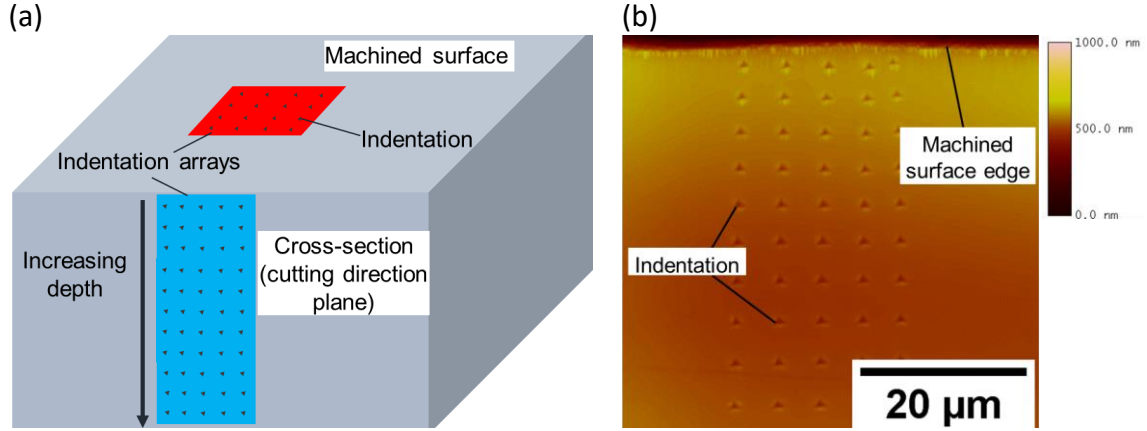


Figure 5.16 – (a) A schematic outlining nanoindentation measurements in the cross-section and on top of the machined surface. (b) An AFM image of a nanoindentation array in the cross-section of a Ti-6Al-4V sample.

Additional indentations to investigate the effect of crystallographic orientation on hardness and elastic modulus in the white layer in Ti-6Al-4V were undertaken using a 4 x 4 array on the machined surface, following mirror polishing, and averaged to obtain a single value. Similarly, bulk material hardness for all alloys was obtained by averaging indentations from a 4 x 4 array in the as-received material, at least 500 μm beneath the surface. In all indentations, the first indentation beneath the surface, as well as adjacent indentations, were kept a distance of three indentation diameters apart, as defined in ASTM E384 [255]. The indenter material constants, E_i and ν_i , were taken as 1141 GPa and 0.07 respectively. Values of 0.34 [169], 0.29 [256] and 0.30 [5] were used as the Poisson's ratio for the Ti-6Al-4V, IN-718 and SCM4V samples, respectively. Hardness and elastic modulus values were normalised with respect to the bulk material hardness in each sample, to illustrate the trends of these properties with depth beneath the surface. The white layer thickness in the vicinity of the indentation array was quantified after indentation.

5.3.5 X-ray diffraction phase analysis

To investigate whether phase transformation had occurred during white layer formation, phase analysis was carried out using x-ray diffraction by examining the location of the peaks in intensity in an XRD pattern, which plots intensity against diffraction angle. Peaks in the diffraction pattern arise due when Bragg's law, shown in Eq. 5.10, is met and they can be associated with a particular interatomic spacing which can be used to determine the corresponding phase from databases of known crystals.

$$2d\sin(\theta) = n\lambda \quad \text{Eq. 5.10}$$

A schematic of a typical x-ray diffractometer is shown in Figure 5.17, highlighting the three main components, the x-ray source, the beam optics and the x-ray detector. X-rays are generated in an x-ray

tube by accelerating electrons towards a target metal using high voltages. Electrons collide with the target material and their deceleration causes the release of x-rays, which then pass through the incident beam optics to strike the sample. The diffracted x-rays then pass through the diffracted beam optics before reaching the detector. Soller slits restrict the axial divergence of the beam thereby reduce diffraction peak asymmetry and divergence and anti-scatter slits are used to restrict the height divergence of the beam, which determines the illumination area of the sample and reduces instrument peak broadening. A monochromator is used to remove the $K\beta$ radiation, allowing only $K\alpha_1$ and $K\alpha_2$ radiation to pass through, due to their similar wavelengths. If $K\beta$, which has a different wavelength to the $K\alpha$ doublet, was not filtered out, it can be reasoned from Eq. 5.10 that there would be additional peaks in the diffraction pattern, which would provide no additional information. A charge-coupled device or a hybrid pixel detector is used to measure the intensity of the diffracted radiation.

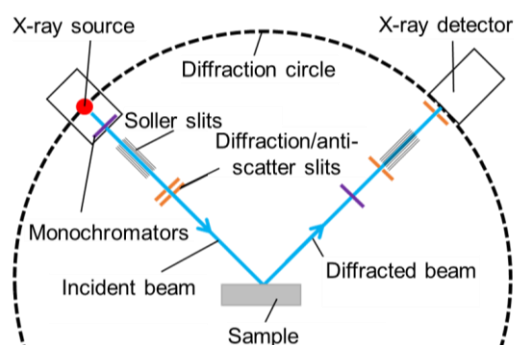


Figure 5.17 – A schematic of an XRD instrument in Bragg-Brentano geometry.

Metallurgical phases of the machined surfaces were analysed using a PANalytical X'Pert³ x-ray diffractometer. The measurements were conducted at room temperature on machined surface samples with and without white layers, as well as examples of the as-received material. Copper $K\alpha$ x-rays were used and the x-ray penetration depths (90% beam attenuation) for the range of diffraction angles imposed in each material, were calculated using AbsorbDX software [257] and are summarised in Table 5.8. The range of diffraction angles used for each material was selected based on the diffraction angles over which most high-intensity peaks are found [223]. The XRD measurements were carried out over an area of approximately 8 mm x 4 mm on the machined surfaces. Post-processing was used to correct for background intensity before PDF-4+ Sleeve+ indexing software [258] was used to fit empirical phase data from the International Centre for Diffraction Data [223] to the diffraction patterns to allow identification of different planes.

Table 5.8 – X-ray penetration depths over the range of diffraction angles used to obtain diffraction patterns in each aeroengine material.

Material	Diffraction angle range (°)	Penetration depth for 90% attenuation (μm)
Ti-6Al-4V	35-80	4.3-9.2
IN-718	40-100	3.4-7.6
SCMV	40-100	1.7-3.9

5.3.6 Crystallographic texture measurement

XRD pole figure measurements were undertaken on machined surface samples to investigate the effects of white layer formation on the resultant crystallographic texture of the material. A pole figure is a 2D representation of 3D crystallographic texture and to obtain a pole figure using XRD, a sample must be rotated in three axes to measure the intensity of x-rays for a particular crystal plane (i.e. a constant diffraction angle) at different sample orientations. By using a sufficient number of steps for each rotation angle, and measuring the pole figure for three non-parallel planes, a full picture of the crystallographic texture can be built up. Measurement on more than one non-parallel plane is necessary to describe the texture of a material, as a pole figure for a single plane shows only the direction of the normal to this particular plane and as such does not contain any information on the rotation of the plane around this normal axis. Consider a pole figure for the basal plane in a HCP crystal, as shown in Figure 5.18, the orientation of prismatic planes, which are perpendicular to the basal plane, could only be determined from additional pole figure measurements.

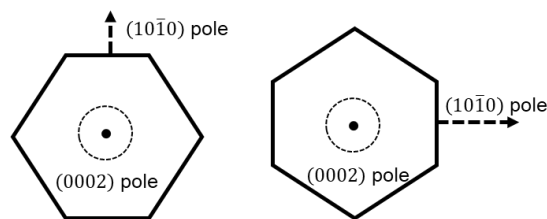


Figure 5.18 – Schematic highlighting the need for more than one pole figure to describe the crystallographic texture. In both images, the (0002) pole is in the same direction yet the (10 $\bar{1}$ 0) pole is in different directions. So the (10 $\bar{1}$ 0) pole could not be found from a pole figure measurement using the (0002) plane.

XRD pole figure measurements require a special type of diffractometer equipped with a 4-circle goniometer. This allows for the rotation of the sample in 3 axes, ψ , ϕ and ω as well as changing of the diffraction angle 2θ , as shown schematically in Figure 5.19 (a).

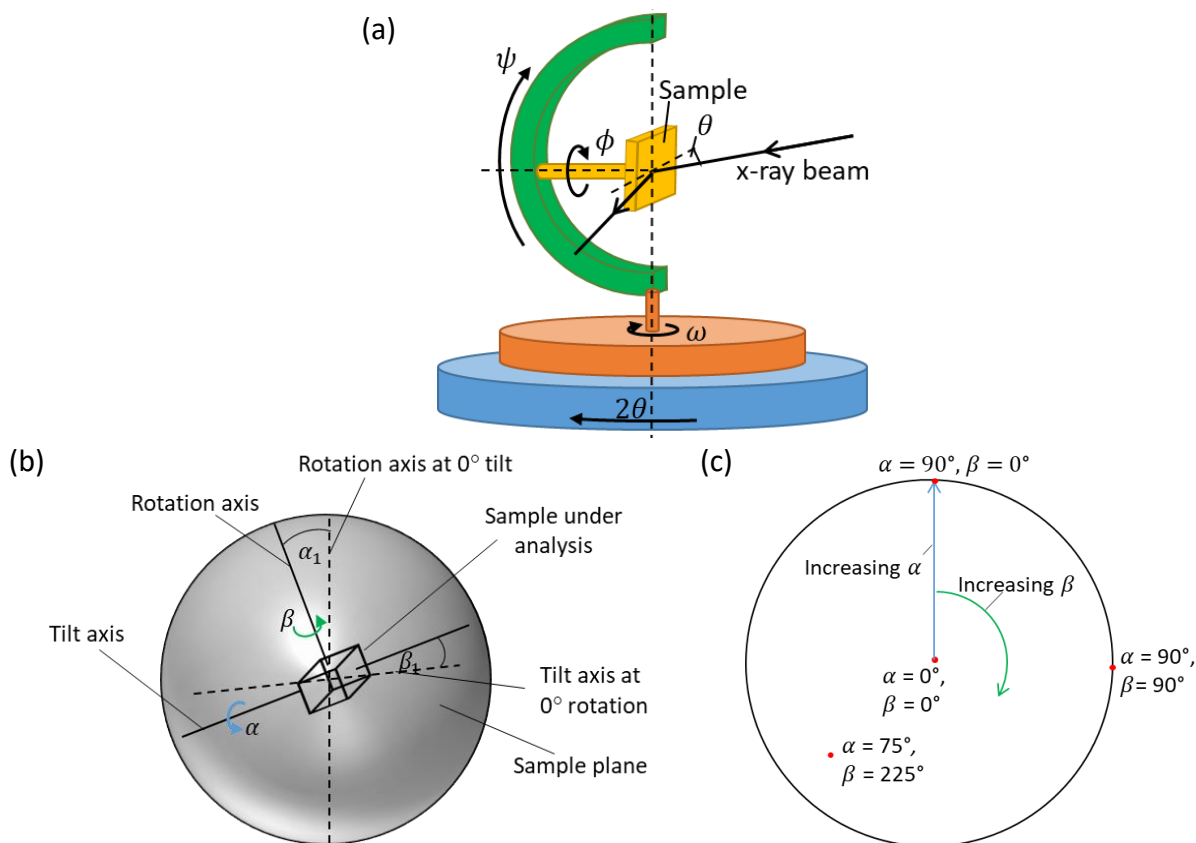


Figure 5.19 – Schematics outlining the process behind XRD pole figure measurements; (a) A four circle goniometer required to rotate the sample, (b) the sample frame-of-reference with pole figure angles outlined and (c) a pole figure plot highlighting three individual measurements at combinations of α and β .

The goniometer angles must be mapped to the sample frame-of-reference, to enable plotting of the data on a pole figure. The goniometer angle ϕ corresponds to the pole figure-angle β , which describes the rotation of the sample around an axis perpendicular to its surface. The angles, ψ and ω , combine to give the other pole figure-angle α , which describes the tilt of the sample around an axis parallel to the plane of its surface, as shown in Figure 5.19 (b). In a full pole figure measurement, the sample is rotated in ψ , ϕ and ω in the path of an x-ray beam at a set diffraction angle such that x-ray intensity measurement can be obtained for a range of values in α (0-90°) and β (0-360°). The intensity measurements are used to plot the pole figure, a circular plot where α is plotted as the radius of the circle and β as the rotation around the circle, as shown in Figure 5.19 (c).

There are two conventions for plotting a pole figure, equal-area and equal-angle projections. The physical interpretations of these two projections are illustrated in Figure 5.20 (a) and (b) respectively. In an equal-area projection, the size of features in a pole figure is preserved regardless of position, as shown in Figure 5.21 (b), thus for a material with a random texture, there will be a uniform distribution of points

on the pole figure. By contrast, the equal-angle projection (stereographic projection) preserves the shape, so a circular region of points appears circular no matter where it is situated in the pole figure, however it will have a larger area towards the circumference of the pole figure, as shown in Figure 5.21 (b). A randomly orientated crystal will have a greater density of points in the centre of a pole figure than at the edges, for an equal-angle projection, distorting the impression of texture. Texture distribution is the property of interest in this study, therefore, equal-area projections were the convention used.

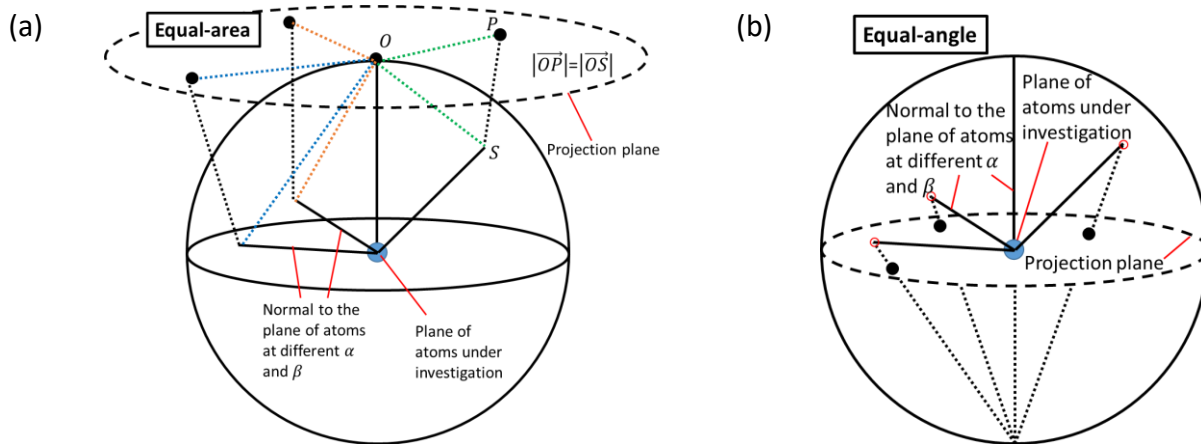


Figure 5.20 – Schematics outlining the physical interpretation of (a) an equal-area pole figure and (b) an equal-angle pole figure.

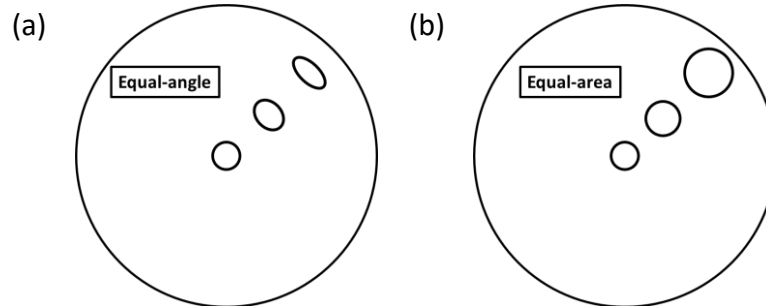


Figure 5.21 – Schematics outlining the effect of the projection on the size and shape of a circular region of points for (a) equal-area and (b) equal-angle projections.

A Bruker D8 Advance x-ray diffractometer equipped with copper $K\alpha$ radiation was used to obtain pole figures for machined surfaces as well as the as-received material in each alloy. The area under investigation in each case was 5 mm x 2 mm. For two machined surface samples in Ti-6Al-4V, the white layer was mechanically polished away before pole figure measurements took place to reveal the swept grain material beneath and allow investigation of the texture of this region. To ensure the white layer had been removed, material removal was quantified using a digital micrometer and cross-referenced against the known white layer thickness in each sample from optical microscopy measurements. A reference measurement was taken from a powder sample to correct the results for defocusing at high tilt angles and

background intensity. The MTEX MATLAB toolbox, as described by Bachmann et al. [259], was used to calculate the orientation distribution functions (ODF) from the corrected data and then plot recalculated pole figures. By calculating an ODF, the full pole figure for any crystal orientation could be plotted and the texture index (TI) calculated. The TI is the square integral of the ODF for a pole figure, as such, it is a numerical indicator of preferential crystallographic orientation in a material; a larger TI indicates a stronger texture.

5.3.7 Residual stress profiling

Profiles of residual stress with depth in the near-surface of machined surface samples were measured through XRD using the $\sin^2(\psi)$ method, with electropolishing used for material removal. Residual stresses impart micro-strains on the crystal lattice, which change the interatomic separation, d and, therefore, alter the position of the peaks in intensity in a diffraction pattern. Tensile stresses shift the peak to lower diffraction angles, whereas compressive residual stresses do the opposite.

In the $\sin^2(\psi)$ method, as described by Cullity [146], an XRD pattern is obtained in the vicinity of a particular crystallographic plane at different tilt angles, ψ and the interatomic spacing is determined at each tilt angle from the peak position. Due to the small depth of penetration of x-rays that confines them to a region close to the free surface, a condition of biaxial stress is assumed over the x-ray interaction volume. This means the stress and strain perpendicular to the surface are taken as zero, however, the stress and strain parallel to the surface are proportional to the change in lattice spacing relative to the unstressed sample. The $\sin^2(\psi)$ method removes the need for calibrating with a stress-free sample of every material, as would be required if only one tilt angle was used, as the stress in a particular direction, σ_x , is proportional to the change in lattice spacing with changing tilt angle, as described by Eq. 5.11.

$$\sigma_x = \frac{\partial d}{\partial \sin^2(\psi)} \left(\frac{E}{1 + \nu} \right) = m \left(\frac{E}{1 + \nu} \right) \quad \text{Eq. 5.11}$$

Where E is the Elastic modulus of the alloy when loading perpendicular to the lattice plane used to measure the change in lattice spacing, ν is Poisson's ratio of the material being measured and m is the gradient of a d vs $\sin^2(\psi)$ plot. A schematic of an XRD residual stress measurement is shown in Figure 5.22. During the measurement, the range of diffraction angles, 2θ , used to obtain the diffraction pattern for a particular peak is held constant and the sample is rotated through a series of tilt angles, positive and

negative relative to the initial position. At each location, the position of the peak in intensity associated with the target crystallographic plane is recorded and converted to a lattice spacing. From this, the d vs $\sin^2(\psi)$ plot can be used to calculate the residual stress.

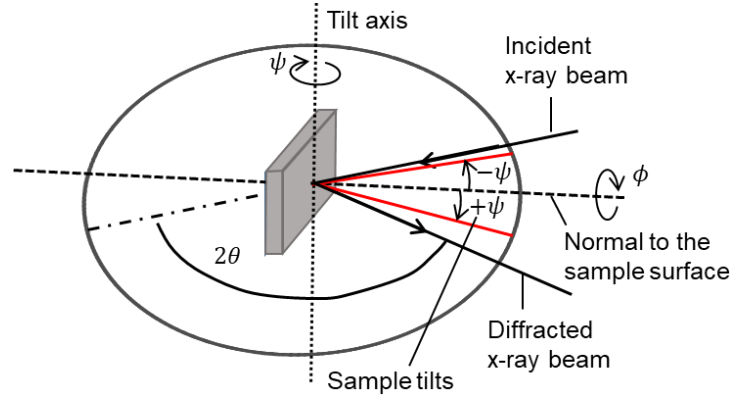


Figure 5.22 – A schematic of an XRD residual stress measurement. The diffraction angle 2θ is kept constant while the tilt angle, ψ , is changed.

Residual stress was measured for machined surface samples in the feed and cutting direction using a Proto iXRD portable diffractometer with a copper $K\alpha$ x-ray tube. Both cutting and feed direction measurements were taken, as residual stress can vary significantly between these directions [69]. Depth profiling measurements were achieved through successive material removal using the electropolishing technique with a salt and water electrolyte. Electropolishing was used as it facilitates the removal of material without altering the residual stress state through mechanical deformation, as could occur with mechanical polishing. The material removal was quantified with a digital height gauge. The $\sin^2(\psi)$ method with 11 tilt angles (ψ) between -19° and $+19^\circ$, was used to measure crystal lattice strain and, through calculation, residual stress in the feed and cutting directions, with the data captured from 2 x-ray detectors. The $\alpha(21\bar{3}3)$ peak and the $\alpha(211)$ peak were chosen for the measurements in Ti-6Al-4V and SCMV respectively, due to their high Bragg angle (approximately 140° and 155°) [223]. Fitzpatrick et al. [147] have shown that higher angles result in larger changes in diffraction angle for a given change in lattice spacing. Due to the prohibitively high cost of these measurements and the examples already highlighted in the literature, residual stress measurements were not obtained for IN-718.

Elastic constants used in the stress calculation for the $\alpha(21\bar{3}3)$ crystallographic plane in Ti-6Al-4V were $S_1 = -2.97 \times 10^{-6} \text{ MPa}^{-1}$ and $S_2/2 = 11.89 \times 10^{-6} \text{ MPa}^{-1}$, which correspond to an Elastic modulus of 112.1 GPa and a Poisson's ratio of 0.33. For measurements in SCMV, $S_1 = -1.42 \times 10^{-6} \text{ MPa}^{-1}$ and $S_2/2 = 5.67 \times 10^{-6}$

MPa⁻¹ corresponding to $E = 235$ GPA and $\nu = 0.333$, were used. Flat plate corrections were applied to account for the stress relaxation effect of layer removal, as outlined by Sikarskie [260]. In addition to stress measurements, peak broadening measurements were also obtained for each sample. The peak broadening was taken as the full width of the XRD peak at half of the maximum intensity (FWHM). An increase in peak breadth can be used as an indicator of a small grain size or inhomogeneous lattice strain over the depth of inspection [146].

5.3.8 Chemical profile analysis

Dynamic secondary ion mass spectrometry (DSIMS) was used to obtain chemical profiles in three Ti-6Al-4V machined surface samples at Loughborough Surface Analysis, two that contained a white layer and one sample with good SI. Measurements were not carried out in steels and nickel due to the prohibitive cost of these measurements and the EDXS research already published for these materials. DSIMS was selected for this investigation due to its extremely high sensitivity to most elements, which allows the detection of elements present in only 10 parts per billion [261]. This sensitivity is significantly greater than that of other techniques such as EDXS and x-ray photoelectron spectroscopy which can only achieve 100 parts per million [262] and 1000 parts per million [263] sensitivity respectively. EDXS is ill-suited to the detection of elements with low atomic numbers, e.g. C, N and O [262], all of which were important to this analysis due to their α -Ti stabilisation effects. Additionally, the x-rays detected during EDXS are generated over a large volume compared to the size of the electron beam, resulting in low depth resolution.

Secondary ion mass spectrometry is a surface analysis technique in which a beam of primary ions is focused onto the surface to be analysed. The interaction of these primary ions with the atoms in the test piece causes secondary ions to be ejected from the surface. These secondary ions are accelerated through a mass spectrometer to determine the chemical composition of the surface. By varying the magnetic field in the mass spectrometer, different atoms can be filtered out such that only ions of one element at a time can reach the detector.

In DSIMS, the beam of primary ions continuously removes material from the test piece and as such allows inspection at increasing depths [261]. A typical DSIMS instrument is shown schematically in Figure 5.23. By measuring the final depth of the crater created by the ion beam using a Dektak profilometer and recording the time at which each mass spectrometry measurement point is taken, a chemical composition

vs depth profile can be created. The depth reached beneath the surface during material removal is proportional to time, as the material removal rate is assumed constant for a largely homogeneous material [261]. This assumption would not be valid for a material constructed from layers of different materials where each material is only several micrometres thick, however, it is acceptable for these Ti-6Al-4V samples. It should be noted that data is only taken from the central $60\text{ }\mu\text{m} \times 60\text{ }\mu\text{m}$ of the crater, which has an overall size of $200\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}$. This is to ensure that only ions ejected from the base of the crater by the ion beam are detected rather than ions ejected from the sides, which would come from a different depth.

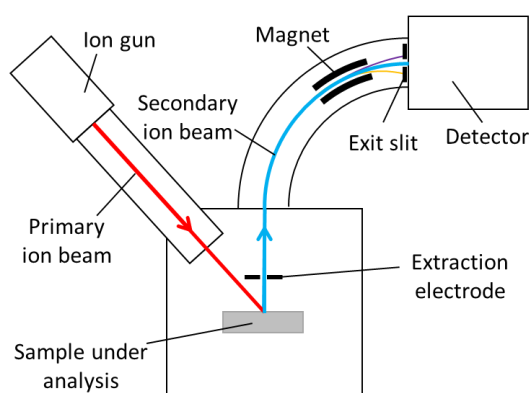


Figure 5.23 – A schematic of a DSIMS instrument highlighting the key features.

Due to the continuous material removal, as well as the nature of the mass spectrometer, only a subset of elements could be measured. This is because the magnetic field in the spectrometer has to be changed to detect each element, hence only a single element can be detected at any given point. The count rate for the desired element is captured and then the field is changed to allow detection of the next element. Consequently, the more elements selected for detection, the greater the interval in depth between measurements for the same element, lowering the depth resolution of the technique. The elements selected for analysis of the machined surface composition were Ti, Al and V, as they are the primary elements in Ti-6Al-4V, in addition to O, C and N as these are alpha-stabilizing elements, which readily diffuse into titanium. Co and W were also selected for detection due to the composition of the tungsten carbide cutting tool used to form the surface being analysed.

The intensity of a particular element does not equate to the exact composition of the alloy, however, it does allow the change in that element with depth to be quantified. To illustrate the change in total alpha

stabilizing content, an aluminium equivalent intensity was calculated using the weightings of the well-known relationship for weight percent aluminium equivalency [32, 264], as shown in Eq. 5.12.

$$A. E_i = Al_i + 10(O_i + C_i + 2N_i) \quad \text{Eq. 5.12}$$

Where X_i is the count rate for element X . This is not a true aluminium equivalency, as the intensity measured by DSIMS does not equate to composition, however it is adopted in this study simply to illustrate general trends. Following the DSIMS measurement, each sample was sectioned and the white layer thickness near the crater was quantified.

5.4 Micro-grid strain measurement

To facilitate strain measurement in the orthogonal milling (OM) samples, micro-grids were patterned onto the 25.5 x 24 mm OM workpieces using electron beam lithography (EBL) as highlighted schematically in Figure 5.24 (a). The workpieces were polished to a mirror finish on the face onto which the grid would be printed. Due to the size of the workpieces and issues encountered with severe edge-rounding when manual polishing, a custom adapter was designed and fabricated which allowed the samples to be polished using automated polishing machines. Following polishing, the samples were coated in AR-P0-6200.13, an electron sensitive resist. The samples were then transferred to an EBL machine where the resist on the samples was exposed to an electron beam, which traces the grid lines on the surface. The interaction of the electron beam with the resist changes the solubility of the resist such that, when the sample is subsequently immersed in a solvent, the exposed regions of resist (the grid lines) are removed.

In the next step, on one workpiece from each pair, thermal evaporation deposition was used to coat the sample in a 10 nm layer of titanium and then a 90 nm layer of gold. The titanium deposition enhances the adhesion between the gold and the titanium substrate. During preliminary trials when using only deposited grid workpieces, the grids peeled off, primarily due to a poor surface finish following polishing. Consequently, on the other workpiece from each workpiece pair in the full trials, a 30 nm coat of gold was sputtered onto the surface, in case the deposition method failed again. Gold was used for the micro-grids due to its large atomic number, which gives rise to a strong contrast on a back-scattered electron SEM image. Additionally, gold possesses a high melting point of 1064 °C [265], so it would only melt if extreme heating had occurred during machining. In thermal evaporation deposition, a metallic source is heated in a vacuum to cause the vaporisation of the material. The vapour cloud produced, cools on contact with the

substrate resulting in the deposition of a thin layer. A vacuum is used to ensure a more uniform layer is produced by reducing collisions between the gold and other atoms when travelling between the source and substrate. By contrast, in sputtering, gold is ejected from an electrode inside a vacuum chamber through bombardment with high-energy ions and then condenses on the substrate forming a thin film.

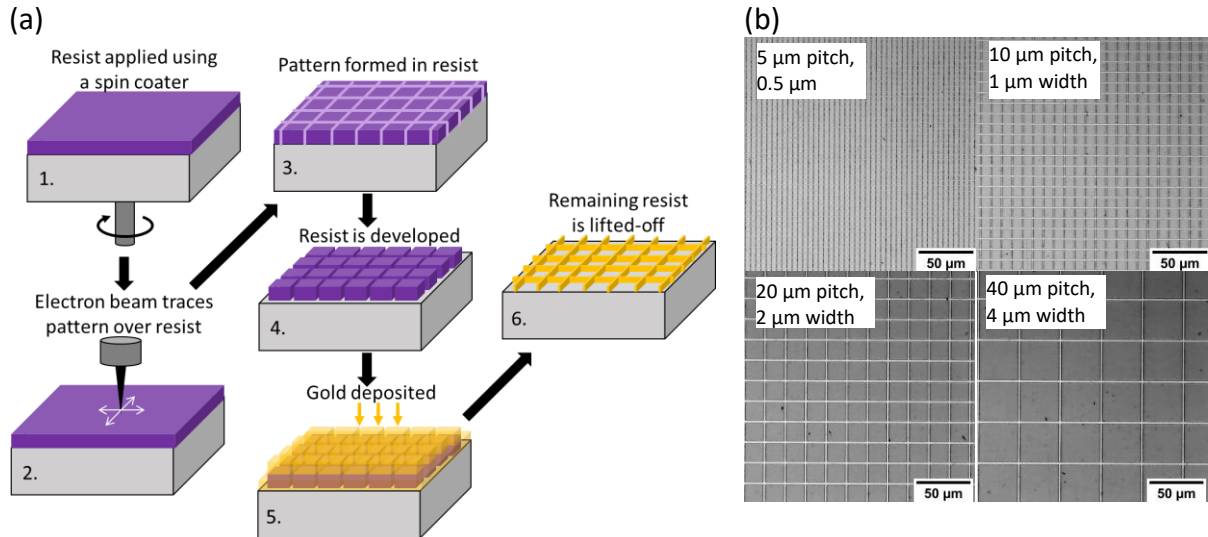


Figure 5.24 – (a) A schematic of the grid printing process and (b) micrographs of the final grids showing the four grid sizes used.

Following deposition and sputtering, the samples were submerged in n-methyl-2-pyrrolidone to lift-off the remaining resist. This process also lifts-off the gold directly above the resist leaving only the gold deposited in the channels produced by EBL, i.e. the grid lines. For these experiments, four different micro-grid sizes were used to ensure deformation could be captured at different length scales as illustrated in Figure 5.24 (b). The grids were arranged as shown in Figure 5.25 (a) with the final machined surface passing through the grids as indicated in Figure 5.25 (b). Note that when sandwiched in a workpiece pair a 5 μm grid would be in contact with a 40 μm grid. This was so that both high and low-resolution strain measurements could be captured, as it was not known how each of the grid sizes would respond to the deformation induced by machining.

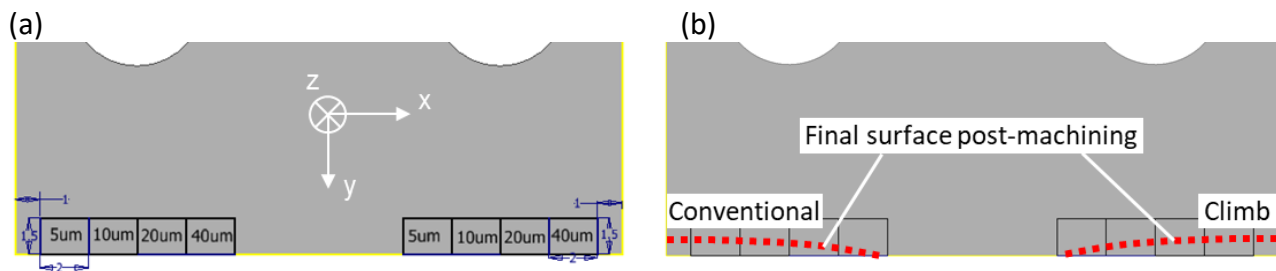


Figure 5.25 – Schematics of (a) the grid locations and (b) the final surface post-machining.

Edge rounding of the workpieces, despite the use of a custom jig to facilitate automation of the polishing process used to prepare the surface for EBL, meant that when the finished workpieces were mounted in pairs in the jig there was a gap between the plates, as shown in Figure 5.26 (a). To reach the plane strain region, the workpiece pair had to be machined using clean-up passes. To reduce the amount of material removal needed, an aluminium foil spacer was used to separate the plates at the rear of the jig, the clamping forces then resulted in plate contact nearer the machined surface, reducing the material removal required before each trial, as highlighted in Figure 5.26 (b). Note the size of the spacer is greatly exaggerated for illustration purposes.

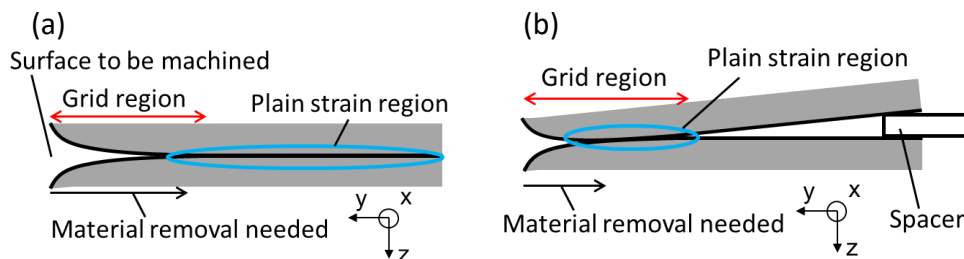


Figure 5.26 – Schematics of (a) the separation between the workpieces caused by edge rounding that necessitates considerable material removal and (b) the reduced material removal when a spacer was used.

Following machining, the strain in the orthogonally milled surface was calculated using the method outlined by Allais et al. [157] by comparing SEM images of deformed and undeformed grids. A full description of the underlying mathematical relationships used during the calculation, the image analysis process and validation of the MATLAB strain measurement script is provided in the Appendix, section 13.4.

5.5 Summary

In this chapter, the materials and machining methods employed in the generation of a set of surfaces for characterisation and non-destructive testing of white layers have been described. The characterisation methods used to measure the physical properties of machining-induced white layers have been detailed. The procedures employed for the measurement of the strain in a machined surface with a white layer have also been introduced. The following chapter presents the results from the tests described in this chapter and then discusses the key characteristics of white layers to guide the development of an NDT method for their detection. The results of this characterisation work in all three materials will be presented in parallel, in order, to facilitate direct comparison between the mechanical and microstructural properties of the white layers in each material.

6 The physical properties and mechanisms of white layer formation in aeroengine alloys

The results from the characterisation of machining-induced white layers in the three aeroengine alloys are reported in this chapter, alongside quantitative strain analysis of orthogonally milled surfaces containing a white layer. Results for each material are presented in parallel to facilitate comparison. Following this, a detailed discussion section will identify the key physical properties of the white layers in each alloy and propose formation mechanisms. Particular attention is given to Ti-6Al-4V, as there is little known about the characteristics and mechanisms of white layer formation in this alloy. Based on the findings of the characterisation work, viable NDT methods for detecting the white layers in each material are suggested.

6.1 Results

6.1.1 Microstructural morphology

Quantitative analysis of cross-sectional micrographs was used to measure the size of the white layer produced in Ti-6Al-4V, SMMV and IN-718. It can be seen from the optical micrographs in Figure 6.1 that surfaces with and without a white layer were produced in each alloy. The traffic lights represent the pass/fail result from SI inspection, as outlined previously.

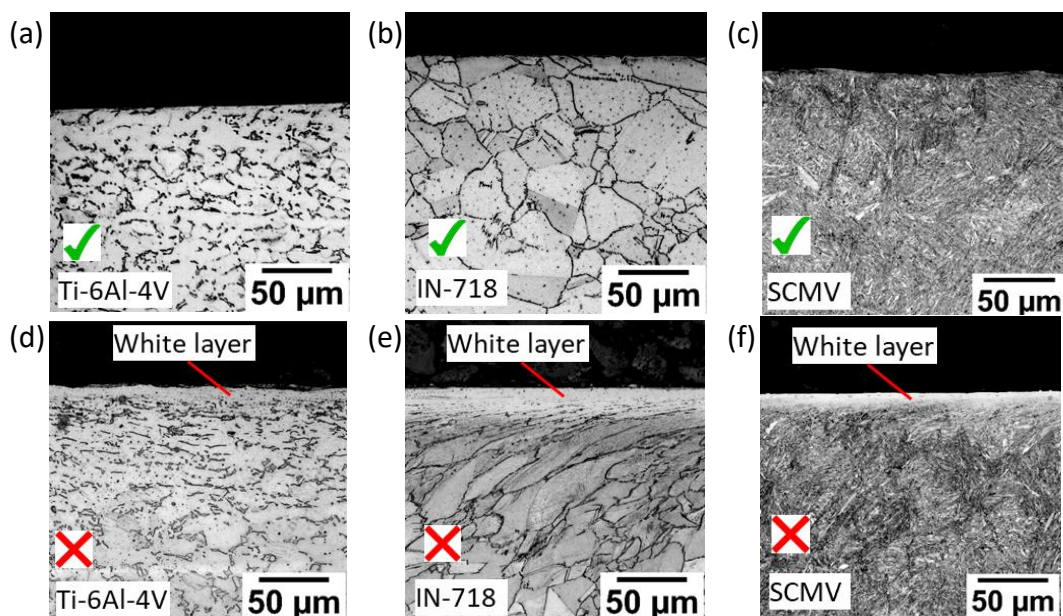


Figure 6.1 – Micrographs of surfaces that would pass surface integrity inspection: (a) Ti-6Al-4V (20 m/min, 0 mm VB) (b) IN-718 (20 m/min, 0 mm VB), (c) SCMv (60 m/min, 0 mm VB) and surfaces which possess a white layer and would thus fail inspection (d) Ti-6Al-4V (20 m/min, 0.5 mm VB) (e) IN-718 (20 m/min, 0.5 mm VB), (f) SCMv (60 m/min, 0.5 mm VB).

White layers were only produced when machining with worn inserts in Ti-6Al-4V and IN-718, however in SCMV a white layer was produced when machining with an unworn insert at high speed (180 m/min). A summary of the surfaces generated in each alloy, according to the white layer thickness, is shown in Table 6.1.

Table 6.1 – The number of surfaces with a particular white layer thickness, quantified through optical microscopy, in each material.

Material	Number of samples				
	No white layer	< 5 μm white layer	5-10 μm white layer	10-15 μm white layer	> 15 μm white layer
Ti-6Al-4V	5	1	11	4	1
SCMV	4	9	9	1	0
IN-718	5	0	0	1	3

From optical inspection, as shown in Figure 6.1, the white layer appears amorphous, i.e. grain structure cannot be resolved, however, higher resolution imaging shows this not to be the case. FEG-SEM micrographs of the white layer and swept grain regions formed during machining of a Ti-6Al-4V surface with a 0.5 mm VB worn insert are shown in Figure 6.2. The higher magnification micrograph shown in Figure 6.2 (b) illustrates the difficult-to-resolve ultrafine microstructure in the white layer, which is at the resolution limit of the FEG-SEM equipment. The microstructure of the white layer is more easily resolved in Figure 6.2 (c), at the transition between the white layer and swept grain regions. It can be seen that the white layer, in this location, consists of nano-scale equiaxed grains, with an average grain size of the order of 100 nm or smaller, measured using the Heyn lineal intercept method as described in ASTM E112 [266]. However, grains are likely to be even smaller in the region shown in Figure 6.2 (b), but this cannot be quantified due to resolution limitations. This sub 100 nm grain size in the white layer contrasts the 10-20 μm α -phase grain size and the elongated grain boundary- β in the bulk microstructure.

In the white layer, it is apparent that any retained- β is indistinguishable from the primary- α , whereas, in the transition and swept grain regions, Figure 6.2 (c) and (d) respectively, the β -phase can be resolved due to atomic number contrast in the micrograph, caused by the heavier elements that stabilise the beta phase. The grain sizes are larger in the swept grain region immediately below the white layer, as shown in Figure 6.2 (d) but were still measured to be smaller than 500 nm. The grain size rapidly increases with depth, through the region of the swept grain material immediately adjacent to the white layer, to reach

the bulk grain sizes. The white layers in IN-718 and SCMV were similarly found to possess a highly refined grain structure, as highlighted in Figure 6.3.

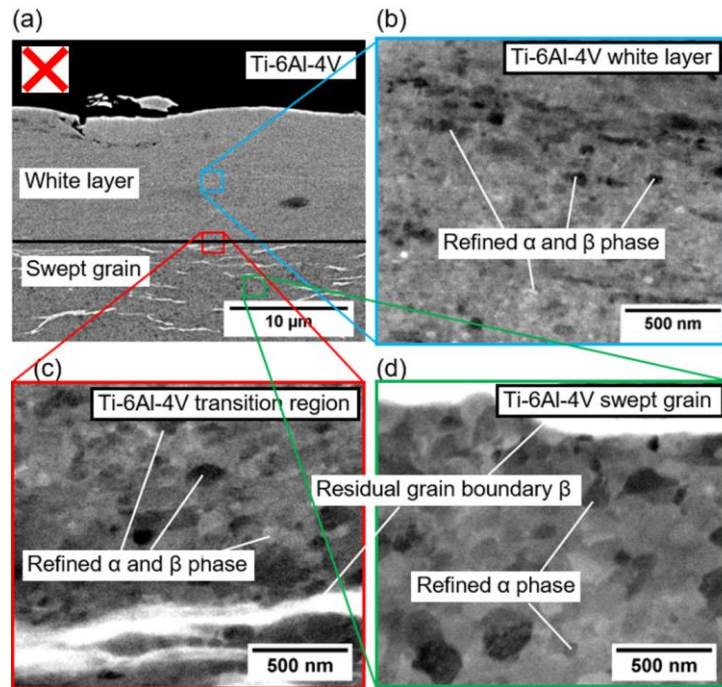


Figure 6.2 – Cross-sectional backscattered electron SEM images of an un-etched Ti-6Al-4V machined surface (80 m/min, 0.5 mm VB), sectioned perpendicular to the feed direction, showing the white layer and the swept grain region (a), and higher magnification images of the white layer (b), the transition region between white layer and swept grain (c) and the swept grain region (d).

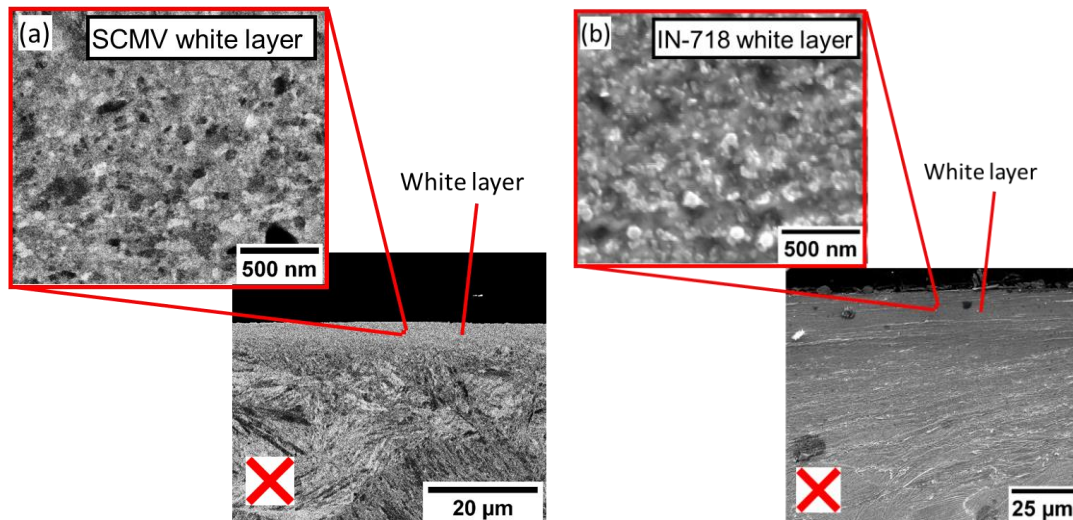


Figure 6.3 – Cross sectional backscattered electron SEM images of (a) an un-etched SCMV sample (60 m/min, 0.5 mm VB) and cross sectional secondary electron images of (b) an etched IN-718 sample (20 m/min, 0.5 mm VB).

To investigate the phase structure of the white layer in Ti-6Al-4V, a sample was heat-treated in a vacuum furnace at 700 °C for 2 hours to drive grain growth, with the corresponding micrograph presented in Figure 6.4. The heat treatment caused α -Ti grains to grow to up to several micrometres in diameter

within the white layer region. The presence of β -Ti in the heat-treated white layer, despite the thermal cycle taking place approximately 300°C below the β -transus temperature, is evidence that the white layer is composed of both the α and β -phases. It is therefore hypothesized that within the refined microstructure of the white layer region, the β -phase is found at the α -grain boundaries, despite the inability to resolve the phases prior to heat treatment in the as-machined white layer.

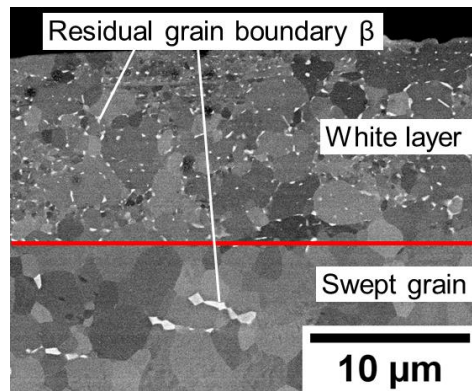


Figure 6.4 – Cross-sectional backscattered SEM micrograph of an un-etched Ti-6Al-4V white layer sample (20 m/min, 0.85 mm VB), which had been heat-treated below the β -transus to result in grain growth without β precipitation

6.1.2 Phase composition of the white layers

XRD patterns, as shown in Figure 6.5, were obtained from surfaces in both the machined and unmachined condition for each of the materials under investigation. It can be seen from Figure 6.5 (a) that only peaks corresponding to the α -phase can be resolved for all the Ti-6Al-4V surfaces examined. The absence of β -phase peaks is due to the small (10-20%) volume fraction of the β -phase, even in the as-received material, in addition to masking of the typically high-intensity $\beta(110)$ peak by the $\alpha(0002)$ peak. However, the larger $\alpha(0002)$ peak for a surface with a white layer cannot be correlated to an increase in the underlying $\beta(110)$ peak as there is no concurrent appearance of the $\beta(200)$ peak at 56° or the $\beta(211)$ peak at 70°. There is a significant difference in the relative intensity of the low angle diffraction peaks between all surfaces, indicating a change in the crystallographic texture due to machining.

It can be seen from the SCMV XRD pattern in Figure 6.5 (b) that there is no measurable increase in retained austenite in the surface with a white layer as only peaks corresponding to the martensitic α' phase are present. Similarly, for IN-718, Figure 6.5 (c), only peaks corresponding to the coherent $\gamma/\gamma'/\gamma''$ phases are present. It was, however, observed that the relative intensity of the (200) peak was different for a surface with a white layer, indicating that texture in the superalloy may have been altered by the machining process. These texture changes are investigated in the next section.

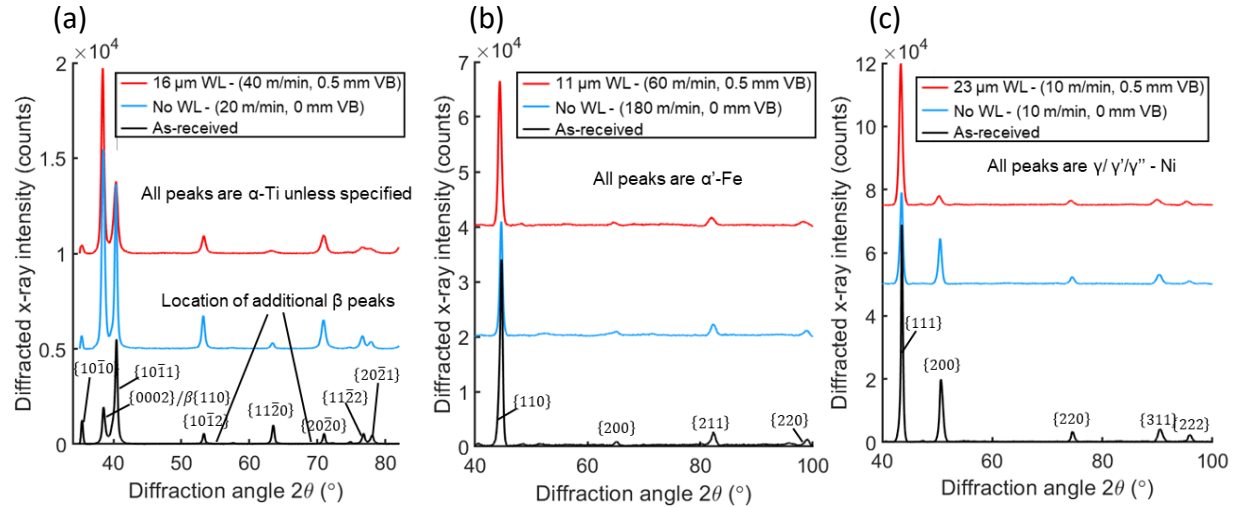


Figure 6.5 – XRD patterns for (a) Ti-6Al-4V, (b) SCMV and (c) IN-718, comparing surfaces with and without white layers (WL).

6.1.3 Crystallographic texture of the white layers with respect to the cutting forces

The XRD pole figures, shown in Figure 6.6, highlight the crystallographic texture of the as-received Ti-6Al-4V material. The pole figures show the multiples of random density (MRD) for the material under inspection, which is a measure of the likelihood of finding a crystal in a certain orientation. It can be seen that the basal (0002) poles are preferentially orientated in the FRD and the CRD.

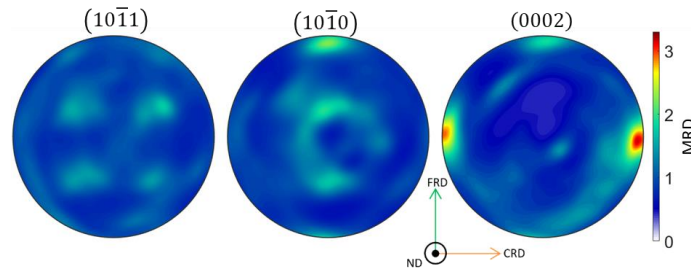


Figure 6.6 - ODF pole figure for the as-received Ti-6Al-4V material.

The pole figures for a Ti-6Al-4V surface with a white layer, Figure 6.7 (a), shows that the white layer exhibits a strong basal $\alpha(0002)$ compression texture. This is indicated by a high MRD value, in the ND of the $\alpha(0002)$ pole figure, compared to the value near 1 for the as-received material in Figure 6.6. By contrast, the swept grain region and a machined surface with no white layer both exhibit a shear texture, as presented in Figure 6.7 (b) and Figure 6.7 (c) respectively. It should be noted that the shear texture is towards the CD for the swept grain region and away for a surface with good SI.

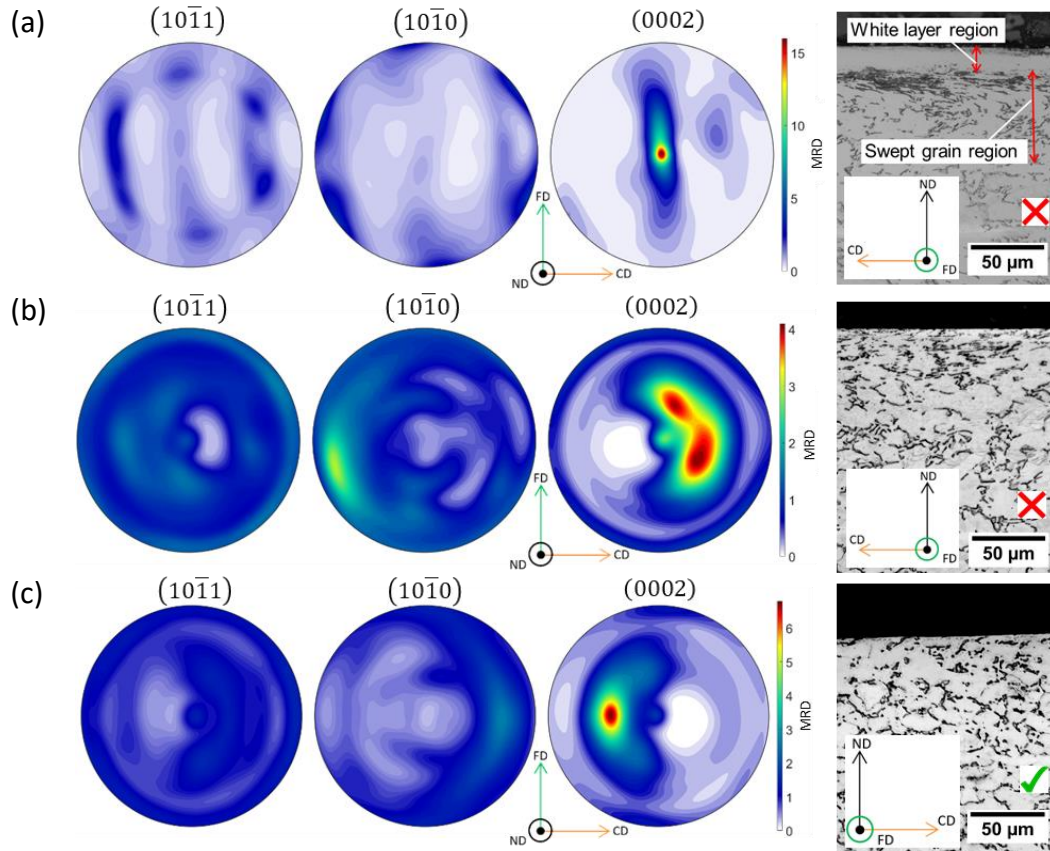


Figure 6.7 – Ti-6Al-4V ODF pole figures and corresponding micrographs for (a) a machined surface (80 m/min, 0.5 mm VB) containing a white layer of up to 20 μm in thickness, (b) a sample from the same machined surface where the white layer has been removed, through mechanical polishing to leave only the swept grain material and (c) a machined surface (40 m/min, 0 mm VB) where no white layer was present.

The compression texture in the white layer is supported by the machining forces measured during cutting with worn inserts, as shown in Figure 6.8 (a). The maximum compressive (z) force of the tool on the workpiece (in the normal direction) was over 2500 N for a surface with a white layer compared to only 80 N for a surface with no white layer, as shown in Figure 6.8 (b). The swept grain sample lacks a compressive texture, despite being subjected to the same machining process as the white layer directly above it. The shear texture arises due to shearing of the workpiece near-surface by the cutting tool, as evidenced by the grain distortion visible in the optical micrographs of the sub-surface, shown in Figure 6.7 (a) and (b), in which the residual β -grains are almost parallel to the surface, immediately below the white layer. However, it is clear that the white layer and swept grain regions are very distinct, in terms of crystallographic texture and microstructural morphology and this will be explored further in the discussion section 6.2.1.

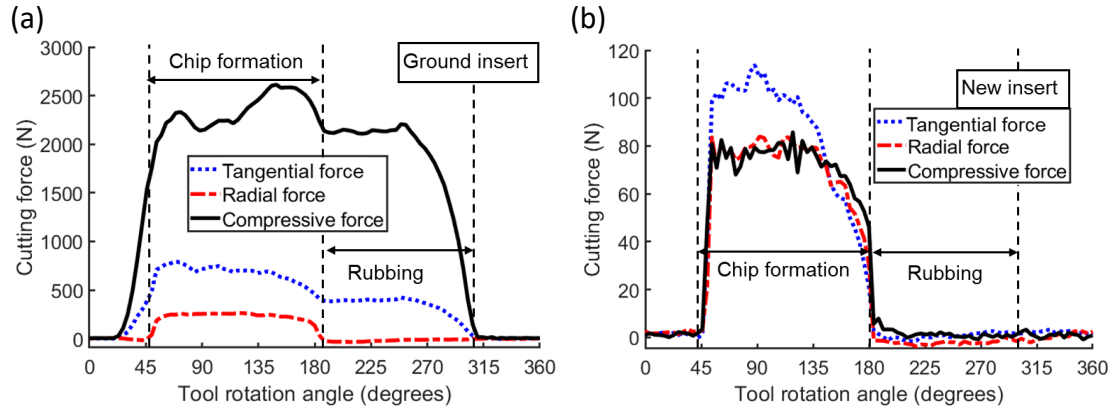


Figure 6.8 – Plots of the forces for Ti-6Al-4V during one tool rotation: (a) a surface on which a white layer was generated (40 m/min, 0.5 mm VB) and (b) a surface on which no white layer was produced (40 m/min, 0 mm VB).

The effect of starting workpiece texture on white layer formation in Ti-6Al-4V is summarised in Table 6.2 and illustrated in Figure 6.9. To demonstrate the preferential crystallographic orientation in a machined surface, relative to a random texture, a numerical indicator called the texture index (TI) is used with a larger TI indicating a stronger texture. The TI is the square integral of the ODF for a pole figure and the corresponding values related to the tested samples are highlighted in Table 6.2. It can be seen that TI of the white layer is much larger than the as-received material in all cases. Additionally, it should be noted from the pole figures that the strong basal texture is formed in the white layer, irrespective of the initial texture.

Table 6.2 – TI for the as-received and as-machined Ti-6Al-4V surfaces for different starting workpiece textures.

Surface	Texture index as-received	Texture index machined-surface
ND	1.26	3.49
RD	1.32	2.74
TD	1.31	4.00
CR	1.18	5.53
Radial	1.31	3.67
Axial	1.26	3.49

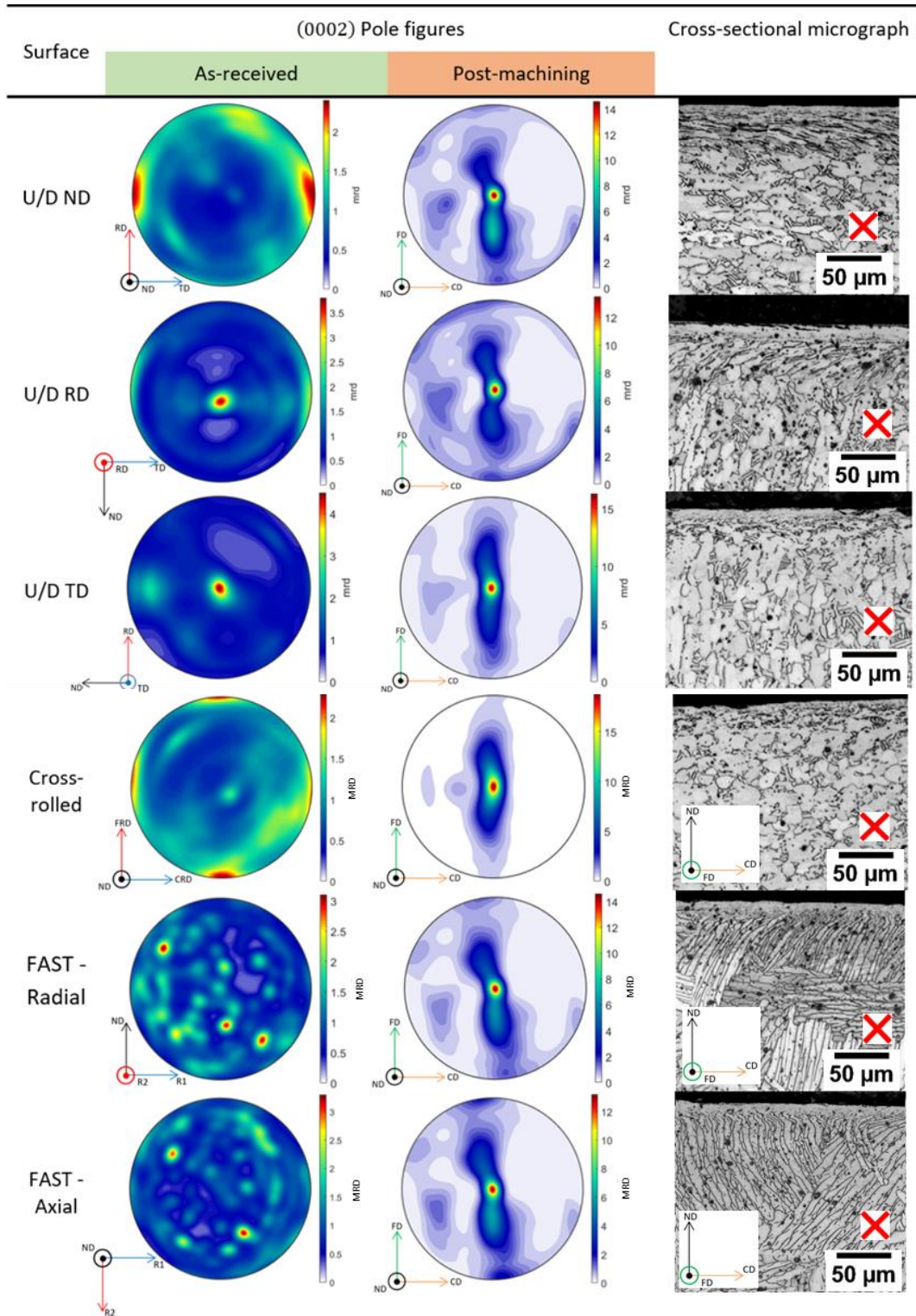


Figure 6.9 – (0002) ODF pole figures for as-received material and as machined workpieces (40 m/min, 0.5 mm VB), containing a white layer, alongside the corresponding cross-sectional micrographs of each surface post-machining.

Pole figures for the as-received material and machined surfaces in IN-718 are shown in Figure 6.10. It can be seen that the machined surfaces exhibit a different texture to the as-received material, however,

there is no significant change in texture between machined surfaces with or without a white layer. In both cases (Figure 6.10 (b) and (c)), a shear texture is present.

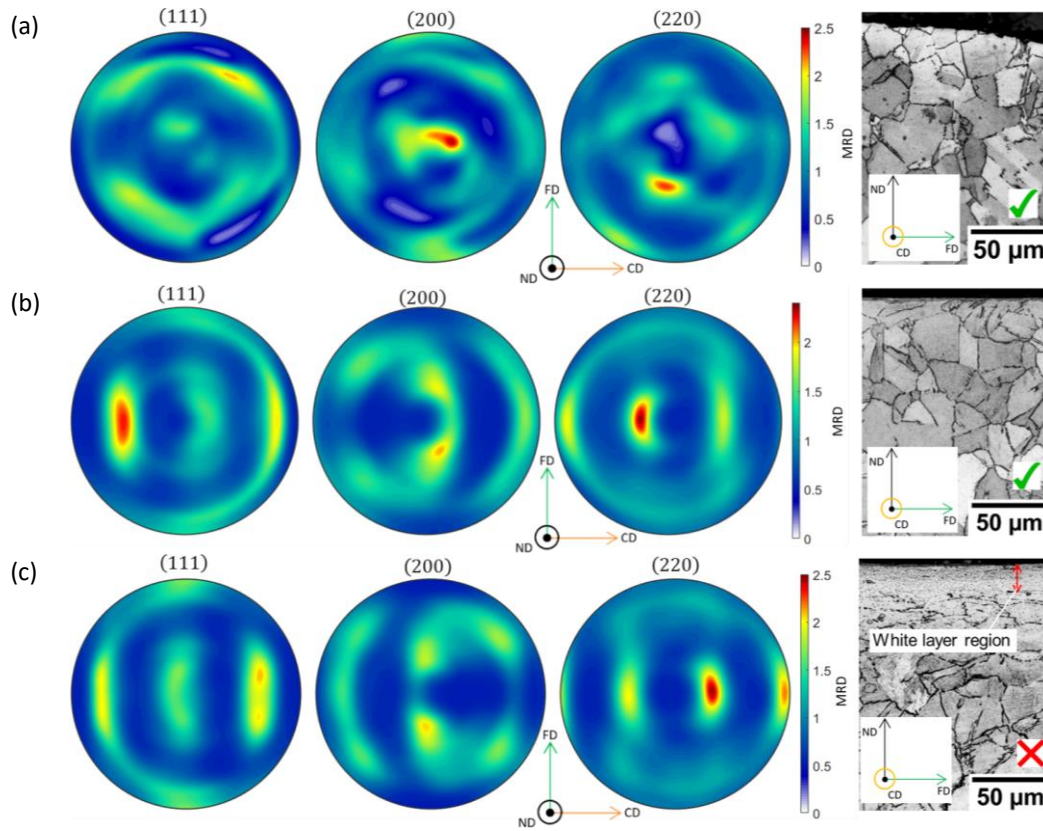


Figure 6.10 – IN-718 pole figures for (a) the as-received material and machined surfaces with (b) no white layer (20 m/min, 0 mm VB) and (c) a 40 μm white layer (10 m/min, 0 mm VB). The axes for the as-received material describe the directions of subsequent machining on the workpiece.

Plots of the typical forces measured during machining of IN-718 when generating a surface with and without a white layer are shown in Figure 6.11 (a) and (b), respectively. A large compressive force was measured when machining with worn inserts during the generation of a white layer, as was observed for the Ti-6Al-4V trials. It should be recognised that each region of material on the square shoulder milled surface was machined during multiple tool passes, due to the larger width of the cutting tool compared to the feed. This is even more apparent for the surfaces machined with the ground inserts as these tools rubbed the surface, even when a chip was not being formed, as evidenced by the large forces measured beyond 180° tool rotation in Figure 6.8 (a) and Figure 6.11 (a).

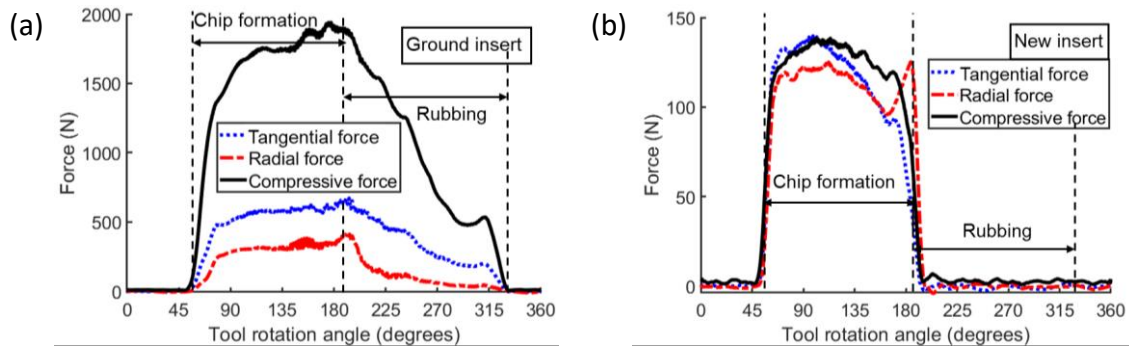


Figure 6.11 – Plots of the forces during milling of IN-718 for one tool rotation: (a) a surface with a white layer (20 m/min, 0.5 mm VB) (b) a surface with no white layer (20 m/min, 0 mm VB).

XRD pole figures for as-received and as-machined SCMV are shown in Figure 6.12. It can be seen that there is a shear texture in all cases, confirming that the as-received material had previously been machined. The cutting and feed directions indicated for the as-received material, Figure 6.12 (a), are those subsequently used in the cutting trials. It should be noted that there was a change in the strength of the crystallographic texture for a surface with a white layer, Figure 6.12 (c), compared to a machined surface with good surface integrity, Figure 6.12 (b), as shown by the increase in the MRD on the (200) plot, and a concurrent decrease in the (110) plot.

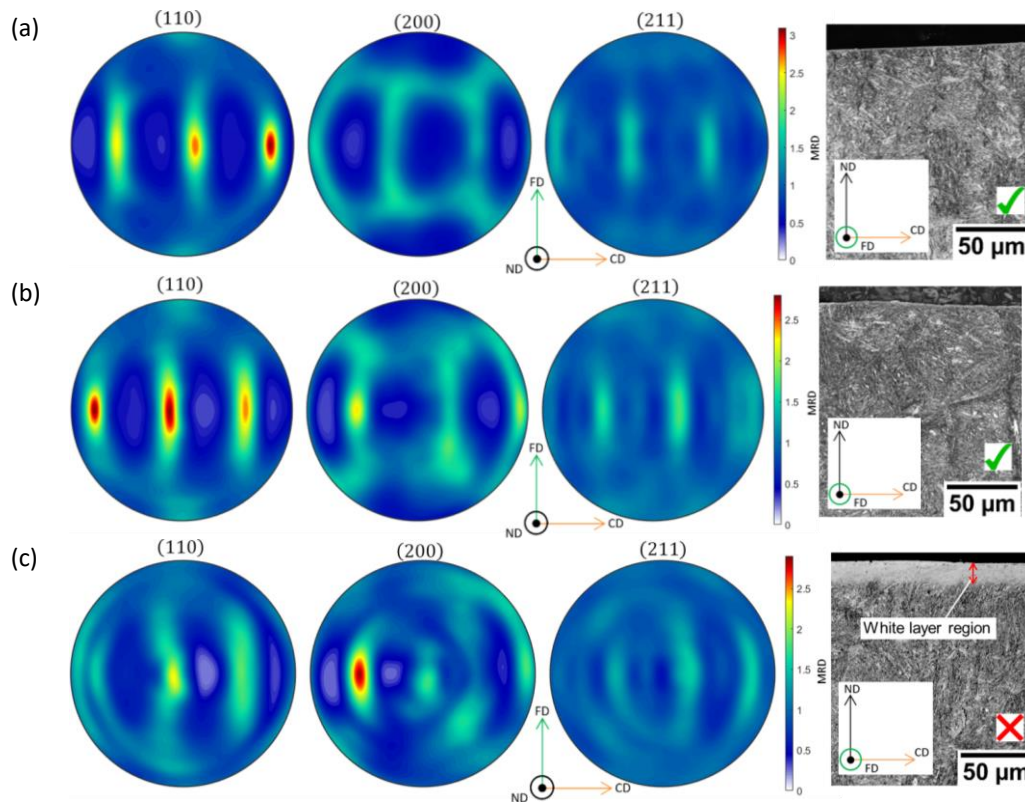


Figure 6.12 – SCMV pole figures for (a) the shaft material prior to these machining trials (b) a machined surfaces with no white layer (180 m/min, 0 mm VB) and (c) a machined surface with an 11 μm white layer (60 m/min, 0.5 mm VB).

Cutting forces during turning of SCMV with worn and unworn inserts are shown in Figure 6.13 (a) and (b) respectively. It can be seen that when generating a white layer with worn inserts, all forces are increased relative to those for a good surface and the compressive force, in particular, is again considerably larger compared to the tangential and feed forces.

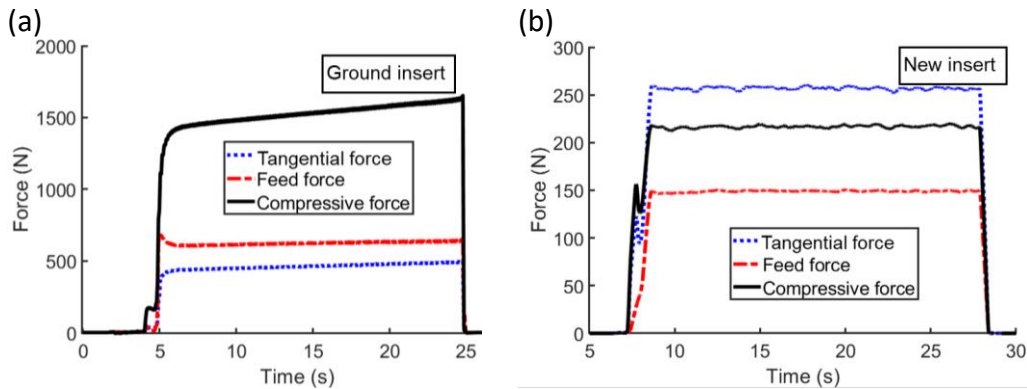


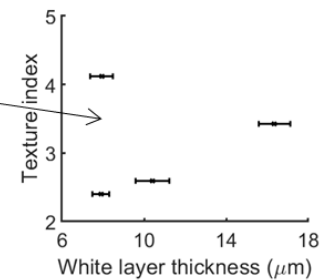
Figure 6.13 – Plots of the forces measured during turning of SCMV: (a) a surface on which a white layer was generated (180 m/min, 0.5 mm VB) and (b) a surface where no white layer was produced (180 m/min, 0.5 mm VB).

The texture index, texture-type and white layer thickness are summarised in Table 6.3 for all materials. In the case of Ti-6Al-4V, all samples having a white layer display a more ordered crystallographic structure (larger TI) than samples with no white layer at the surface, however, the TI is not proportional to the white layer thickness. By contrast, for IN-718, TI increases only slightly and in SCMV the TI decreases for surfaces with a white layer.

Table 6.3 – TI, texture type and white layer (WL) thickness of each type of sample and the dependence of TI on WL thickness in Ti samples.

Sample	TI	Texture type	WL thickness (μm)
As-received – Ti	1.25 (± 0.07)	Cross-rolled	0
As-machined – Ti	2.04 (± 0.14)	Shear	0
As-machined – Ti (WL)	3.14 (± 0.69)	Compression	10.6 (± 3.4)
As-received – Ni	1.44	Shear	0
As-machined – Ni	1.39	Shear	0
As-machined – Ni (WL)	1.49 (± 0.05)	Shear	28.6 (± 11.0)
As-received* – SCMV	1.76	Shear	0
As-machined – SCMV	1.79	Shear	0
As-machined – SCMV (WL)	1.49 (± 0.01)	Shear	9.6 (± 3.3)

* The as-received SCMV had previously been machined so does not represent the as-forged condition.



6.1.4 Hardness and elastic modulus of the white layers

Hardness and elastic modulus profiles were obtained for each alloy using nanoindentation. It can be seen from the hardness profile of cross-sectional Ti-6Al-4V samples, as shown in Figure 6.14, that the white layer was measured to be up to 15% harder than the bulk material, whereas there was no observable

trend in hardness, with increasing depth beneath the machined surface, in samples without a white layer. The samples without a white layer show greater levels of uncertainty in the measured hardness value (6.8%) compared with those with a white layer (3.4%). It can be seen from the profiles shown in Figure 6.15 that elastic modulus was measured to increase with depth beneath the machined surface in three of the five Ti-6Al-4V samples with a white layer and, similar to hardness, the uncertainty in elastic modulus was greater in surfaces without a white layer.

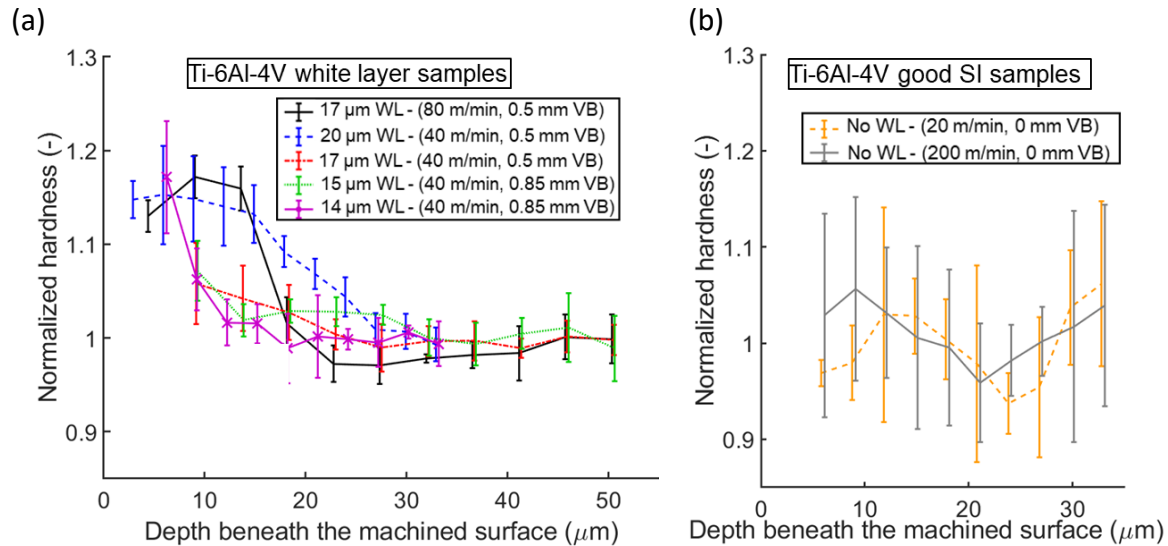


Figure 6.14 – Hardness profiles in the cross-section of the machined surface for (a) surfaces with a white layer and (b) surfaces without a white layer. Error bars represent one standard deviation of the mean.

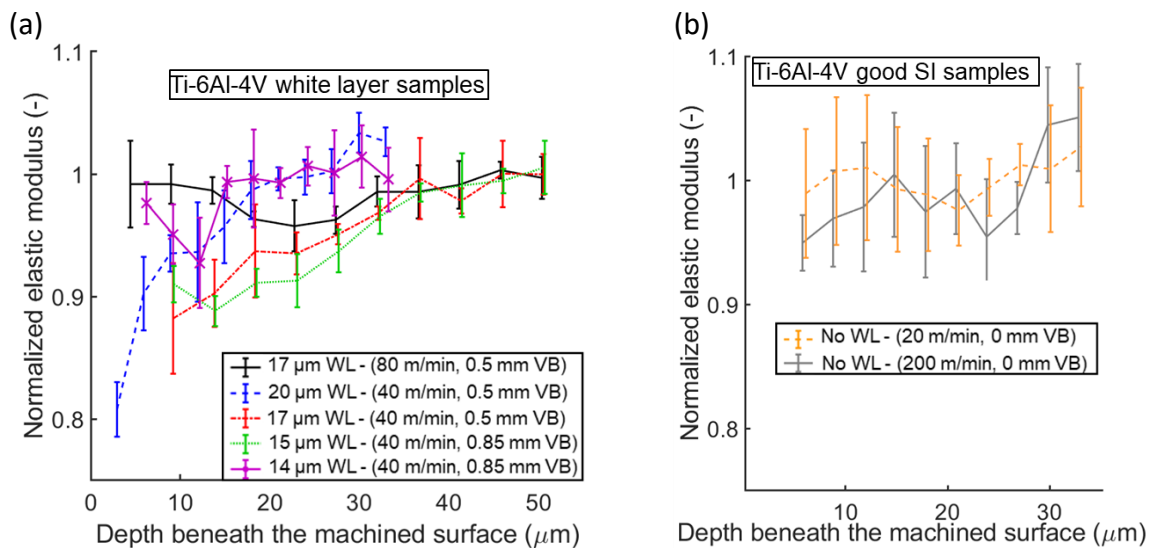


Figure 6.15 – Elastic modulus profiles in the cross-section for samples with (a) a white layer and (b) no white layer.

Due to the anisotropy of the HCP crystal structure which can lead to variation in hardness and elastic modulus with orientation, nanoindentation measurements were also obtained on top of the machined

surface in Ti-6Al-4V, in addition to the cross-section. It can be seen from Figure 6.16 that there is a 20-30% higher hardness value measured when indenting on top of the white layer, compared to in the cross-section, with a similar relationship for elastic modulus but the change for a surface with no white layer is lower. Therefore, it is apparent that the development of a strong crystallographic texture during white layer formation, as identified in section 6.1.3, is resulting in a significant anisotropy of the mechanical properties of the white layer.

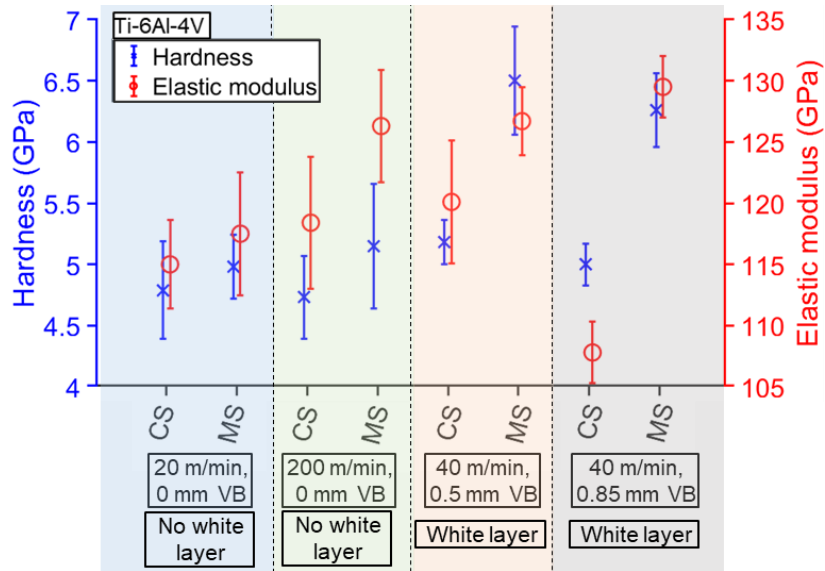


Figure 6.16 – Hardness and elastic modulus results from nanoindentation measurements of Ti-6Al-4V samples on the machined surface (MS) and in the cross-section (CS) samples. Error bars represent one standard deviation of the mean.

The hardness and elastic modulus profiles for SCMV are shown in Figure 6.17 (a) and (b), respectively. The white layer in SCMV is up to 45% harder than the bulk material and there is a slight increase in elastic modulus near the surface for the samples containing a white layer and one machined surface without a white layer. Due to the limited number of indentations that can be made within the white layer in a cross-sectional sample whilst still adhering to ASTM E384 [255] and the comparatively thin white layers in SCMV compared to Ti-6Al-4V, a tapered cross-sectional sample was produced which facilitated an increased number of measurements within the white layer, as shown in Figure 6.17 (c). The hardness and elastic modulus of this tapered sample are shown in Figure 6.17 (d). It can be seen that the hardness decreases linearly through the white layer, reaching bulk values beyond the depth of the white layer. The elastic modulus, by contrast, appears to decrease by approximately 2%, near the machined surface, the opposite relationship than was observed for the non-tapered samples. There is a clear trend that the material properties are more uniform in the white layer according to the smaller error bars.

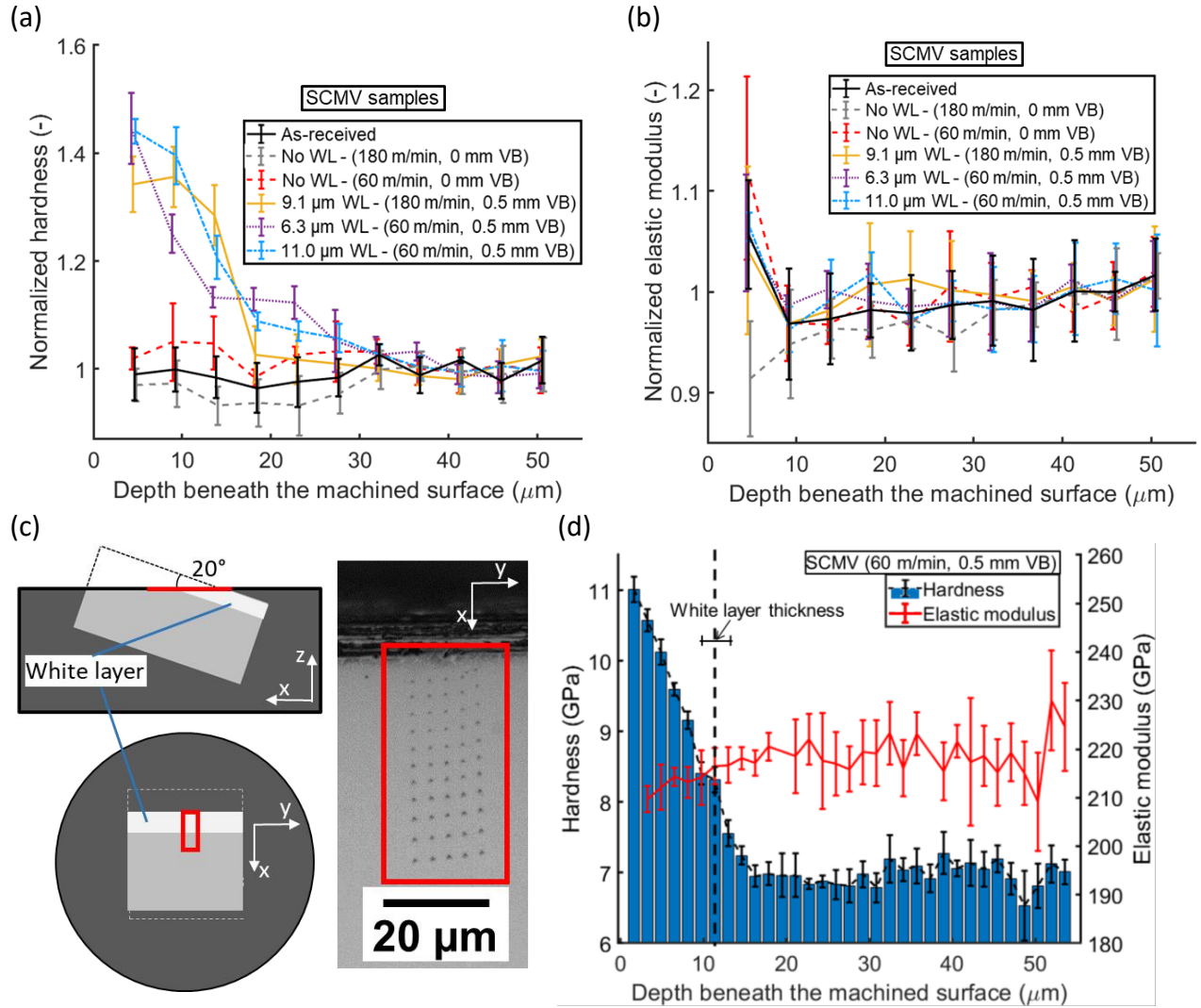


Figure 6.17 – Profiles of (a) hardness and (b) elastic modulus with depth for SCMV machined surfaces. (c) A schematic of the tapered sample used to increase the extent of the white layer in the plane of the nanoindentation and (d) the resulting hardness and elastic modulus profile (60 m/min 0.5 mm VB). Error bars represent one standard deviation of the mean.

Hardness and elastic modulus profiles for IN-718 are shown in Figure 6.18 (a) and (b), respectively. Hardness in the near-surface region was measured to be as much as 20% greater for surfaces with a white layer compared to the bulk, however, there is a greater difference in hardness between different IN-718 surface with a white layer compared to the other alloys studied. For one surface of the surfaces with a white layer, the hardness increased with depth and was greatest in the swept grain region. Elastic modulus, by contrast, was found to exhibit no clear trend with depth, as observed for SCMV surfaces.

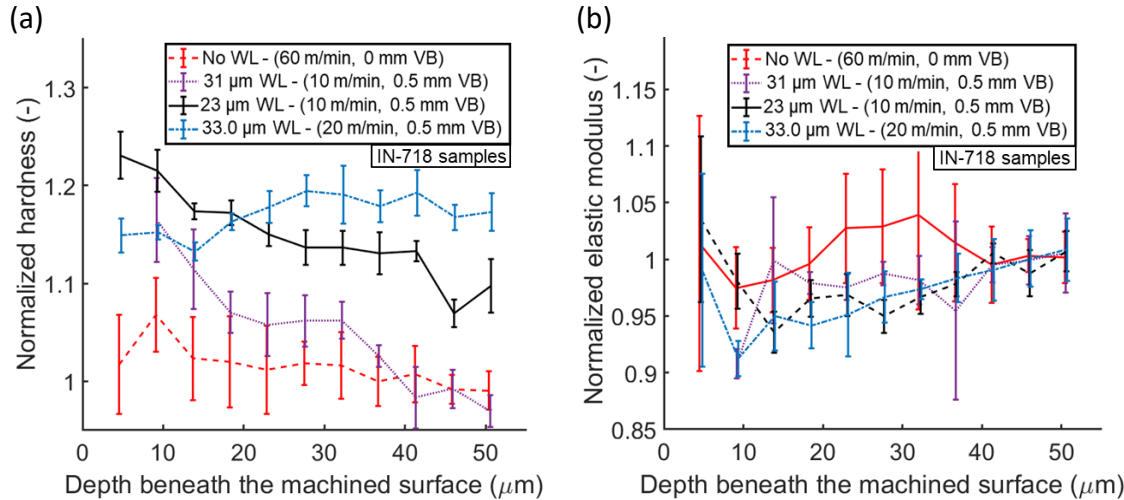


Figure 6.18 – (a) Hardness and (b) elastic modulus profiles with depth for IN-718 machined surfaces. Error bars represent one standard deviation of the mean.

6.1.5 Machined surface residual stress profiles

Residual stress profiles were measured for Ti-6Al-4V and SCM4V, due to the lack of literature on the residual stress accompanying white layer formation in titanium alloys and the sensitivity of the ferromagnetic BN NDT method to residual stress, respectively. As previously outlined, due to the examples already highlighted in the literature and the prohibitively high cost of these measurements, residual stress measurements were not obtained for IN-718.

XRD Residual stress and corresponding peak broadening variations with increasing depth beneath the machined surface in Ti-6Al-4V are shown in Figure 6.19 (a), for the cutting direction and feed directions. A compressive surface residual stress state was measured for all samples tested, including the as-received material. However, a 600-1000 MPa near-surface compressive residual stress was measured for a 16 μm white layer in contrast to the lower 0-200 MPa compressive stress for an 8 μm white layer. Both surfaces possess compressive stresses of at least 550 MPa at depths up to 150 μm beneath the surface, far beyond the extent of the white layers.

Peak broadening results for the same set of Ti-6Al-4V surfaces are shown in Figure 6.19 (b). It can be seen that the peak breadth is greatest, at all depths, for those samples with a white layer. The peak breadth of a machined surface with no white layer is slightly lower than that with a white layer but both types of machined surface exhibit reduced peak breadth with depth tending rapidly towards values comparable to that of the unmachined material.

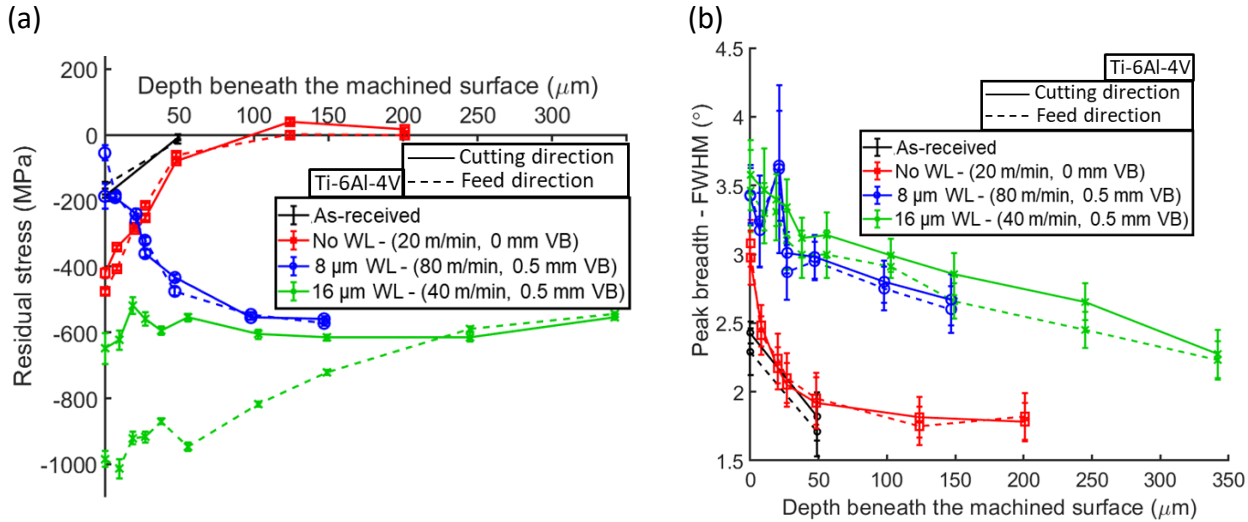


Figure 6.19 – (a) Ti-6Al-4V residual stress profiles and (b) corresponding peak broadening values beneath the machined surface in the cutting and feed directions. Error bars represent one standard deviation of the mean.

Residual stress and peak broadening measurements for SCMV are shown in Figure 6.20 (a) and (b) respectively. A compressive residual stress of up to 1800 MPa was quantified at the machined surface for samples containing a white layer, with the largest stresses in the feed direction and stresses present in a layer that extends to at least 0.8 mm beneath the surface. This 1800 MPa compressive stress is greater than the yield stress in the bulk material [30], however, the white layer is considerably harder than the bulk material, due to work hardening, so these high residual stresses are possible in the deformed near-surface regions. For surfaces with no white layer, a tensile residual stress was measured at the machined surface that changes to a compressive state within the first 100 μm from the surface and then returns to a zero residual stress state with further depth. Similar to the titanium samples, peak breadth was larger for a SCMV surface with a white layer, however, in contrast to titanium, the peak breadth decreases comparatively rapidly to bulk values beyond the extent of the white layer, suggesting that peak broadening in the swept grain region is lower.

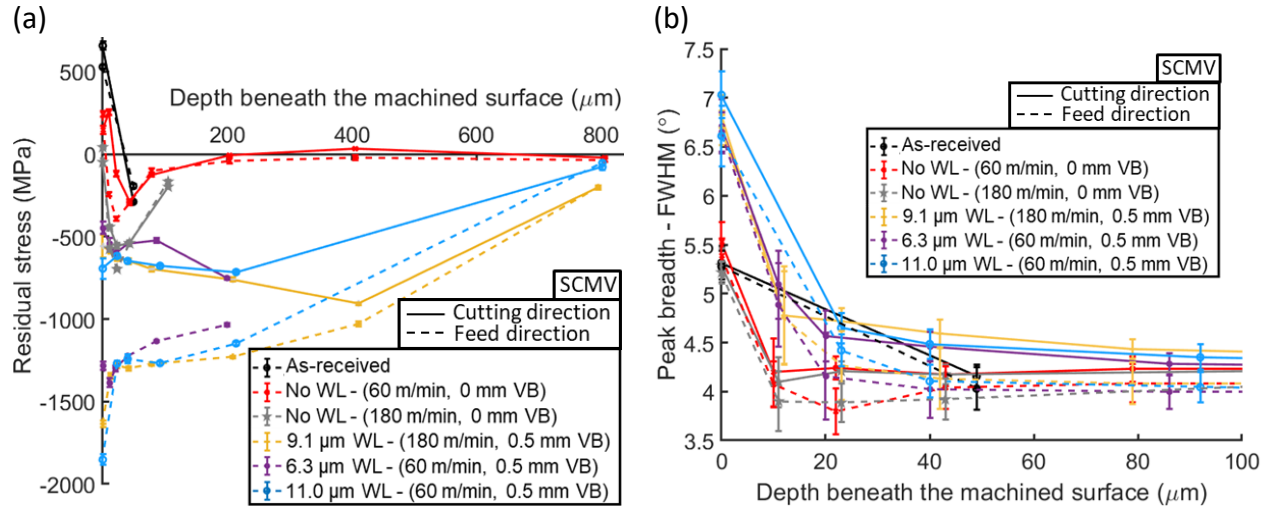


Figure 6.20 – (a) SCMV residual stress profiles and (b) corresponding peak broadening values beneath the machined surface in the cutting and feed directions.

6.1.6 Chemical composition of the white layer in Ti-6Al-4V

Profiles of chemical composition with depth beneath the machined surface for Ti-6Al-4V samples, measured using DSIMS, are shown in Figure 6.21 (a) for a surface with no white layer and Figure 6.21 (b) and (c) for samples containing a white layer. The concentration of alpha stabilizing elements (C, N, O) is higher in the first 5-10 μm beneath the machined surface when a white layer is present. This is highlighted in more detail in Figure 6.22 where the aluminium equivalent intensity is plotted for every surface. In addition to the presence of alpha stabilizing elements, a higher concentration of cutting tool elements, cobalt and tungsten was also recorded in surfaces possessing a white layer.

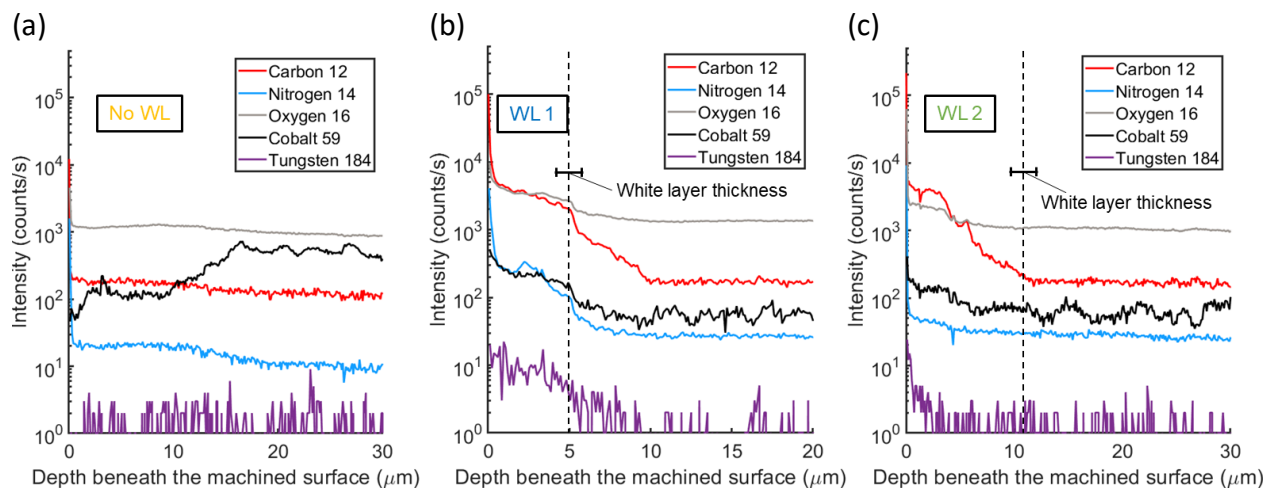


Figure 6.21 – Plots showing the relative presence of elements with depth beneath the machined surface for (a) a machined surface with no white layer (20 m/min, 0 mm VB) (b) a machined surface with a 5 μm white layer (80 m/min, 0.5 mm VB) and (c) a machined surface with an 11 μm white layer (40 m/min, 0.5 mm VB).

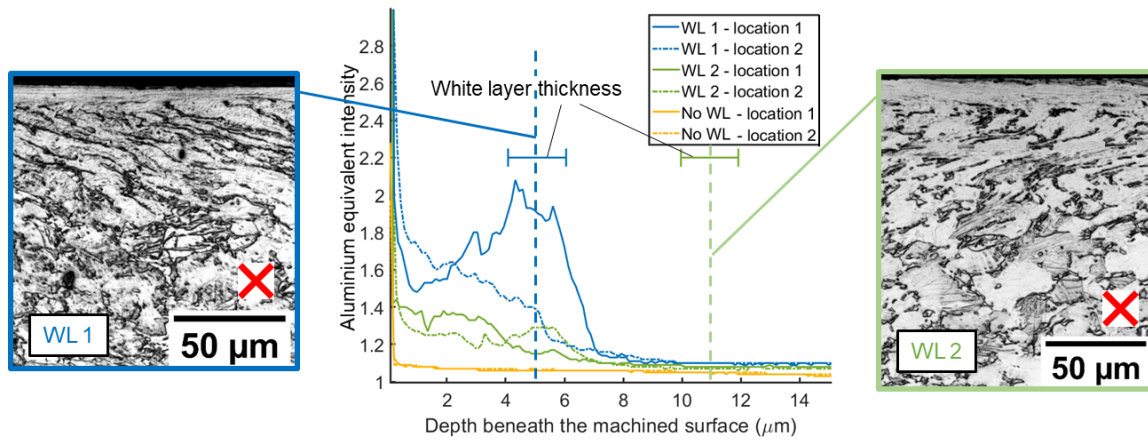


Figure 6.22 – Aluminium equivalent intensity for the three samples tested. Each sample was measured in two locations.

6.1.7 Investigation of the strain in the machined surface during white layer formation

Measurement of the strain within the near-surface region in orthogonally milled Ti-6Al-4V was used to investigate the amount of deformation required to cause white layer formation. The effects of tool wear, cutting edge radius, cutting speed and climb vs conventional milling on the strain imparted in the workpiece were investigated and attempts were made to measure the strain within the white layer itself. Cutting conditions from the trials explicitly discussed in this section are presented in Table 6.1, the full set of cutting conditions used in the trials are shown in the appendix section 13.3.

Table 6.4 – Selected cutting conditions from the Ti-6Al-4V orthogonal milling trials.

Experiment No. \ Run order	Cutting Speed V_c (m/min)	Insert wear VB (mm)	Cutting edge radius (mm)	a_p (mm)	a_e (mm)	Feed per tooth f_t (mm/tooth)	Climb/ conventional milling
1	20	0.265	137	8	0.5	0.5	Climb
2	20	0.265	137	8	0.5	0.5	Conventional
3	20	0	30	8	0.5	0.5	Climb
7	20	0.5	86	8	0.5	0.5	Climb
9	80	0.5	11	8	0.5	0.5	Climb
10	80	0.5	13	8	0.5	0.5	Conventional
11	80	0.5	86	8	0.5	0.5	Climb
12	80	0.5	93	8	0.5	0.5	Conventional

6.1.7.1 Differences between naturally worn and artificially worn inserts

In the orthogonal milling trials, artificially worn inserts with a 0.5 mm wear land were used to encourage the generation of surfaces containing white layers, similar to the square shoulder milling trials. In addition to this, an off-the-shelf cutting insert was used to orthogonally mill a Ti-6Al-4V workpiece until the tool became worn. The cross-sectional profiles of the cutting tool geometry, in the region of the cutting edge, for unworn, naturally worn and artificially worn inserts are shown in Figure 6.23 (a). The larger cutting edge radius (137 μm), visible in the profile for the naturally worn insert in Figure 6.23 (a), indicates that tool wear in this cutting configuration primarily resulted in edge radius rounding, rather than the formation of the flat wear land that is produced by artificially wearing the insert. The progression of tangential, radial and out-of-plane forces during repeated orthogonal milling cuts for the naturally worn insert is shown in Figure 6.23 (b) for its unworn and worn states, alongside a plot of the forces for an identical cut using an artificially worn insert. For the naturally worn tool, the radial cutting force, i.e. the equivalent of the compressive thrust force, increases by as much as three times compared to the unworn tool, whereas the tangential force is reduced by almost 10%. The artificially worn insert gives rise to comparable radial forces but larger tangential forces.

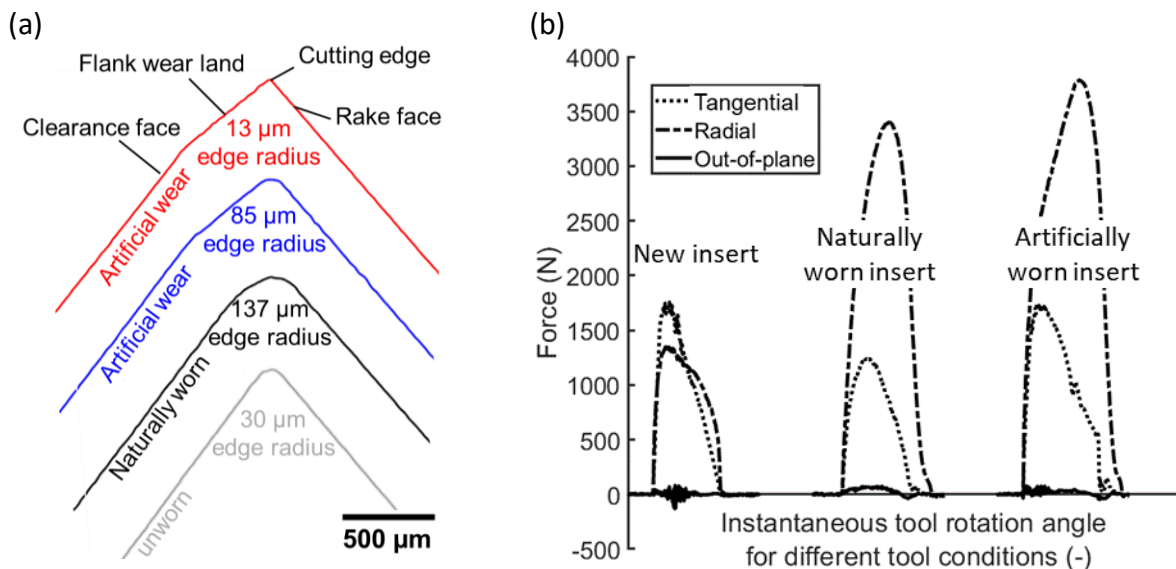


Figure 6.23 – (a) Cross-sectional profiles of different cutting tools near the cutting edge and (b) the forces when machining with a new insert, a naturally worn insert and an artificially worn insert (all 20 m/min, climb milling).

When inspecting cross-sectional micrographs of the full cutting arc following orthogonal milling, the material removed from the workpiece was measured to be less than the intended depth of cut in almost all of the machined samples. The difference in material removal when machining with a worn insert

compared with an unworn insert under the same cutting conditions is illustrated in Figure 6.24 for a desired 0.5 mm depth of cut. The largest deviation was measured for samples during climb milling with naturally worn inserts where no material was removed, despite the correction of the tool offsets to account for the smaller size of the insert when worn. As such, when machining with the naturally worn insert, the workpiece will most likely have been deformed through rubbing and ploughing without chip formation. When machining with unworn inserts, the desired 0.5 mm depth of cut was always achieved. Discussion around this error is provided in section 6.2.3.

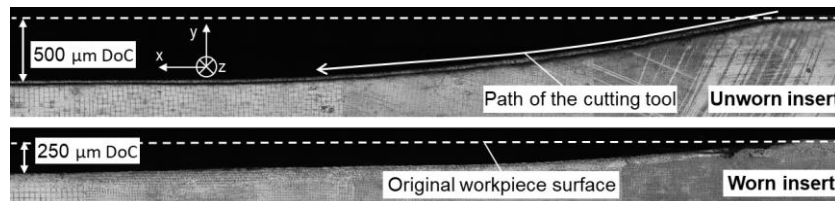


Figure 6.24 – Cross-sectional optical micrographs of the cutting arcs for two orthogonal milling cuts using identical cutting conditions but with an unworn and worn insert respectively. The original workpiece surface is highlighted and the achieved material removal is labelled.

6.1.7.2 Surface integrity of orthogonally milled samples

The geometry of the orthogonal cutting operation necessitated a high feed and so the same region of material was not machined on multiple tool passes, as occurred for square shoulder milling. Consequently, the resultant surface integrity during these orthogonal milling trials could only arise due to cutting and rubbing of the workpiece during one tool pass, rather than subsequent rubbing of the same region of material. A white layer was generated in surfaces machined with both naturally worn and artificially worn inserts in the orthogonal milling trials, as shown in Figure 6.25 (a-c) but, as would be expected following the results of the square shoulder milling trials presented in section 6.1.1, no white layer was generated when machining with unworn inserts (Figure 6.25 (d)). The white layers observed were more than four times thinner than those generated in the previous square shoulder milling trials, despite the comparable tool wear and cutting speeds. No white layer was observed when a conventional milling tool rotation was employed, or when an artificially worn insert with a sharp cutting edge was used, as shown in Figure 6.25 (e) and (f) respectively.

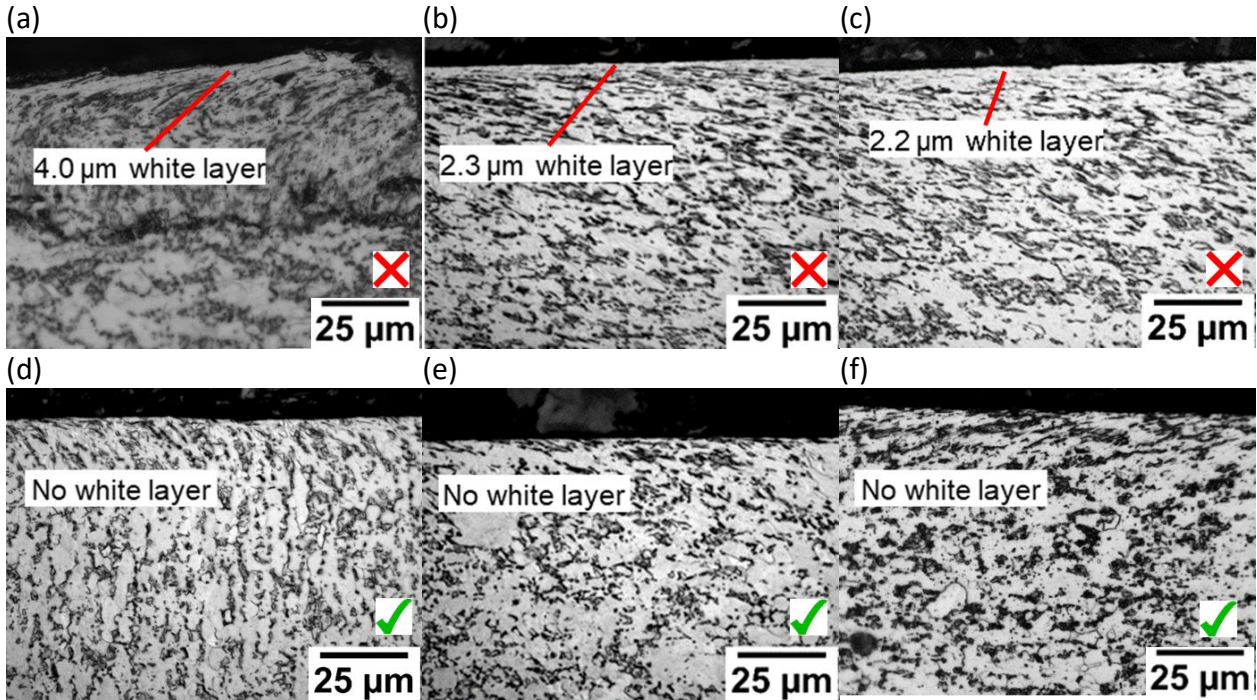


Figure 6.25 – Cross-sectional optical micrographs of surfaces machined in (a) trial 1 (b) trial 11 (c) trial 7 (d) trial 3 (e) trial 12 and (f) trial 9.

The thickest white layer was generated with the naturally worn inserts, however, the surface was not uniformly covered, with a large proportion of the surface possessing no white layer. The wear land of the naturally worn inserts and artificially worn inserts are compared in Figure 6.26 (a) and (b), respectively. In the former, a wear land of 0.265 mm was measured compared with the 0.5 mm wear land in the latter. However, as highlighted previously in Figure 6.23 (a), the cutting edge radius was larger for the naturally worn insert but there was some edge chipping in places.

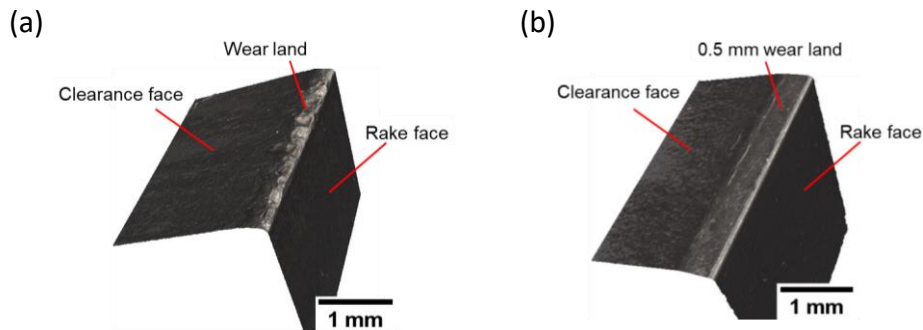


Figure 6.26 –Alicona point cloud images of (a) a naturally worn insert and (b) an artificially worn insert.

The regions across the cutting arc in which cutting with chip formation and rubbing without chip formation occur are shown in Figure 6.27. During climb milling, the depth of cut (0.5 mm) and feed (0.5 mm/rev) were selected to ensure the instantaneous chip thickness varied from approximately 130 μm,

which was larger than the cutting edge radius, through to zero chip thickness, with the reverse occurring for conventional milling. As such, for all trials there was a region at one end of the cutting tool path where the instantaneous chip thickness was greater than cutting edge radius and so a chip was formed, except for the cuts using the naturally worn insert in which the cutting edge radius was larger than the maximum instantaneous chip thickness. At the other end of the tool path, there was a region where the instantaneous chip thickness tended towards zero and ploughing and rubbing could occur without chip formation.

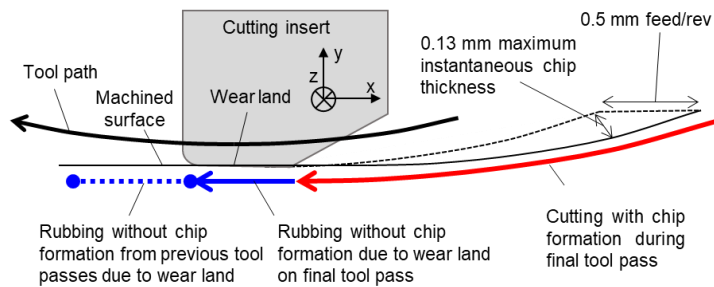


Figure 6.27 – A schematic outlining the key regions of the machined surface, the final cutting arc is made up of the final tool pass, plus the region with rubbing but no chip formation from approximately 6 previous tool passes.

A white layer was only observed to form when chip formation was not occurring i.e. when the instantaneous chip thickness was much smaller than the cutting edge radius and therefore ploughing or rubbing could occur. However, from the error on the depth of cut error shown in Figure 6.24, it is apparent that material in the rubbing region, where chip thickness should be zero in theory, may still be ploughed underneath the cutting edge due to a non-zero instantaneous chip thickness arising from the failure to achieve the full depth of cut. In sections 6.1.7.3 - 6.1.7.5, strain maps of different surfaces will be reported to better understand why white layers formed under certain conditions, but not others. Strain maps were obtained from the rubbing region and regions where the cutting edge was both smaller than and larger than the instantaneous chip thickness. However, due to varying grid quality post-machining, which resulted in regions with no resolvable grids, comparisons were only made between strain maps from identical locations along the cutting arc rather than for the full cutting arc.

6.1.7.3 The effect of tool wear and tool edge radius on deformation near the machined surface

The true shear strain imparted into the machined surface is shown in Figure 6.28 (a) and (b) for an artificially worn and an unworn cutting insert, respectively. It can be seen that strain is significantly higher for the surface machined with a worn insert, with values reaching as high as 0.6, 40 μm beneath the

machined surface. It is not possible to quantify the strain at the machined surface itself, due to the large resolution of the grids in this rubbing region of the cutting arc, however it can be seen from Figure 6.28 (c) and (d) that the shear, XX and YY strains are all higher for the artificially worn insert. The depth of deformation is over 100 μm for the worn insert, nearly twice as large as for the unworn insert.

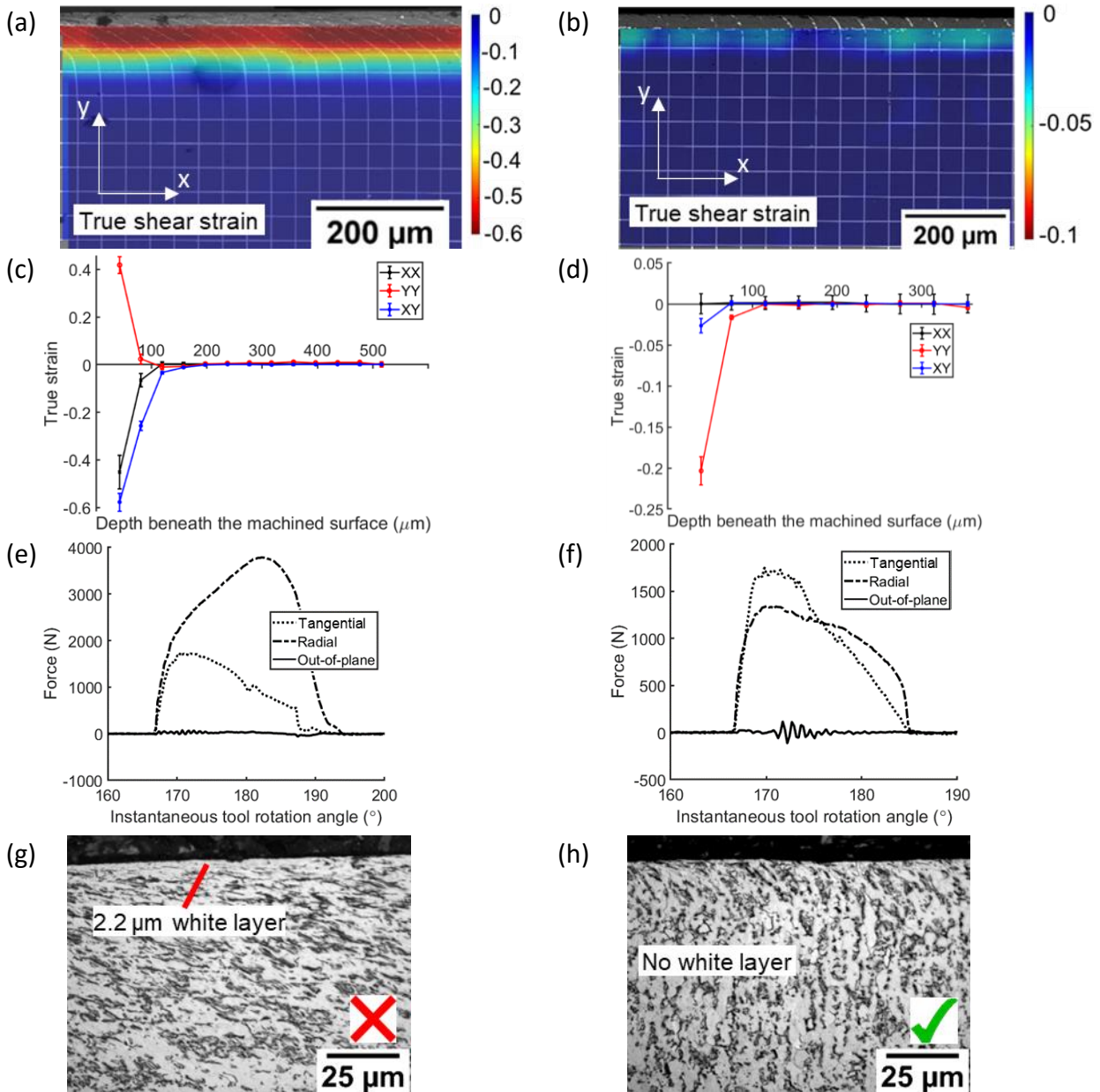


Figure 6.28 – True shear strain maps for a surface machined with (a) an artificially worn insert $VB = 0.5 \text{ mm}$ (trial 7) and (b) an unworn insert (trial 3) with corresponding true strain profiles in (c) and (d) respectively. The forces measured during machining are shown in (e) and (f) for each insert and micrographs of the same regions of material as the strain maps are shown in (g) and (h). The tick and cross on the micrographs represent the results of SI inspection.

These observations of high strain for the surface machined with a worn insert are supported by the large radial (negative y-direction) force of over 3000 N measured, as shown in Figure 6.28 (e) and (f), which is over 2000 N larger than for the unworn insert. It should be noted, however, that the tangential (negative x-direction) forces were a similar magnitude for both surfaces. As would be expected based on the large difference in the strain maps and profiles for the two types of insert, a white layer was only produced when machining with worn inserts, as shown in Figure 6.28 (g) and (h).

For the surfaces machined with worn inserts the YY strain was always positive, whereas when machining with the unworn inserts the YY strain found to change direction with depth beneath the surface depending on the location along the cutting arc. Outside of the rubbing region of the cutting arc, the YY strain was positive near the machined surface but became compressive with increased depth before returning to a value of zero in the bulk material, as shown in Figure 6.29. The reason for this difference in YY strain for worn and unworn inserts is due to the nature of the chip formation process and is explored further in discussion section 6.2.2.

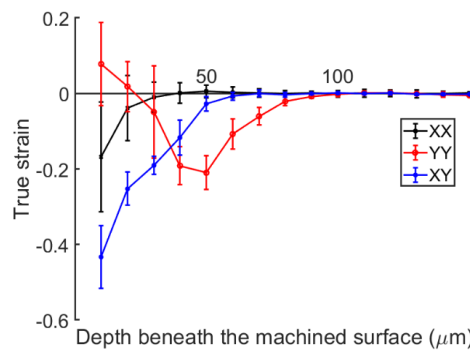


Figure 6.29 – True strain profile from trial 3 outside of the rubbing region where the edge radius was smaller than the instantaneous chip thickness. The YY strain (red) can be seen to change direction with depth beneath the machined surface.

Figure 6.30 (a) and (b) compares the effect of a small (13 μm) and large (85 μm) cutting-edge radius, respectively, on the strain imparted into the workpiece. It can be seen that an 85 μm radius leads to strain values 1.5-3 times larger than a 13 μm radius insert, for the same cutting parameters. Additionally, the depth of deformation is at least twice as large in the rubbing portion of the cutting arc as shown in the strain profiles in Figure 6.30 (c) and (d). The increased deformation with the large edge radius insert can be attributed to the larger radial force during machining which was 2-3 times greater than for a tool with a smaller cutting edge radius, as shown by the force plots in Figure 6.30 (e) and (f). The tangential force, by contrast, was similar for both types of insert.

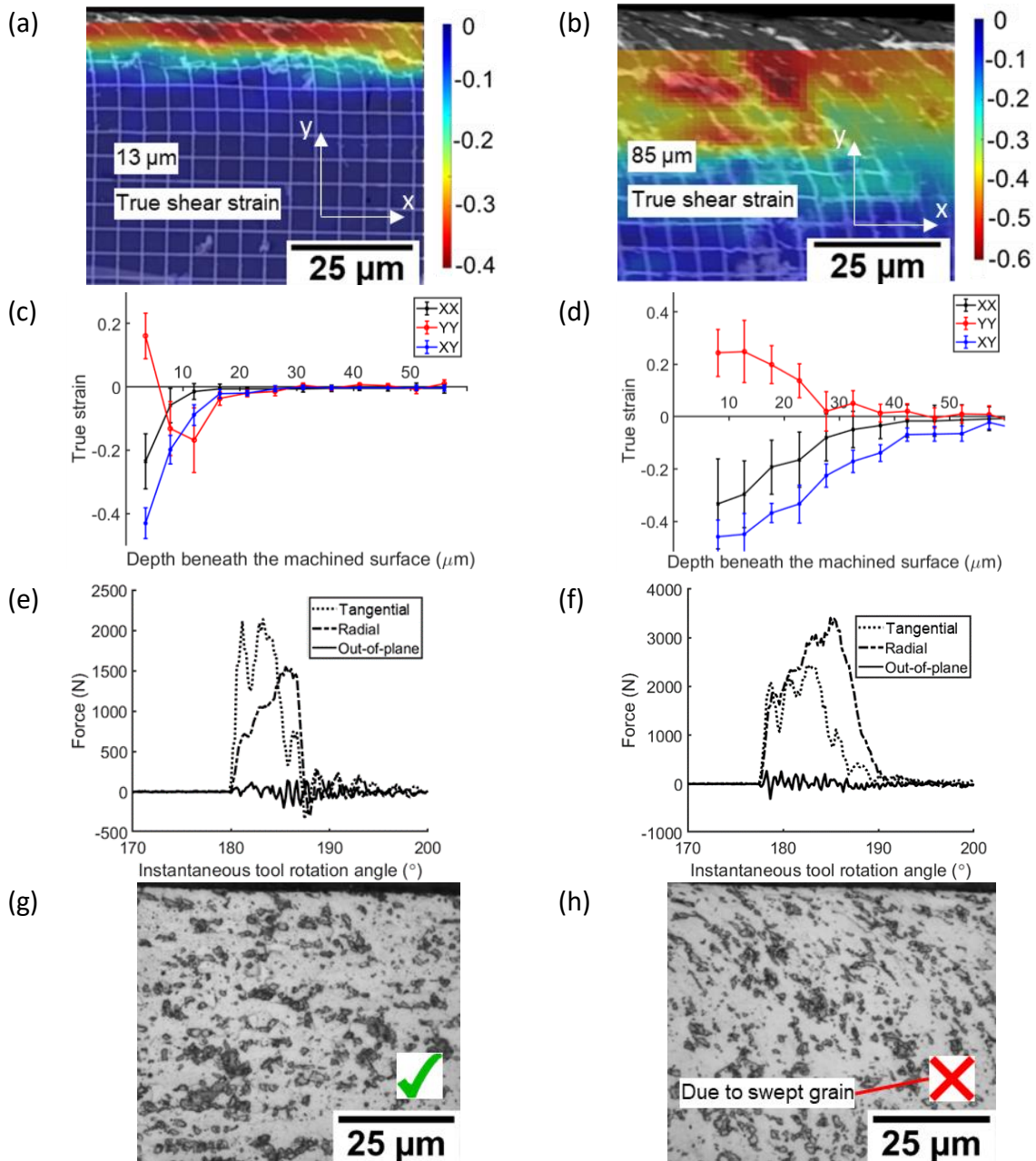


Figure 6.30 – True shear strain maps for a surface machined with an insert with (a) a 13 μm cutting edge radius (trial 10) and (b) an 85 μm cutting edge radius (trial 12) with corresponding true strain profiles in (c) and (d) respectively. The forces measured during machining are shown in (e) and (f) for each insert and micrographs of the same regions of material as the strain maps are shown in (g) and (h).

White layers were only produced for surfaces machined with the larger cutting edge radius inserts, as shown previously in Figure 6.25. This is unsurprising considering the greater deformation in the near-surface when machining with the larger cutting-edge radius as visible in the strain maps and the micrographs in Figure 6.30 (g) and (h). However, it can be seen from Figure 6.30 (g) and (h), that in the specific regions of these strain maps, no white layers were produced however a large swept grain region was identified for the surface machined with the large cutting edge radius insert and this led to a surface

integrity inspection fail. It should be noted that the YY strain in Figure 6.30 (c) for the 13 μm edge radius insert is tensile at the surface and compressive immediately beneath when machining, a behaviour also identified when machining with the unworn inserts with a 30 μm edge radius in Figure 6.29. The same trend was not observed for the insert with an 85 μm edge radius.

The largest subsurface deformation across all of the trials was measured after machining with a naturally worn insert, as shown in Figure 6.31 (a-c). It should be noted that, although the grid size in the strain maps in Figure 6.31 (a-c) is 5 μm and therefore offers the highest resolution of all the grids used in this study, the distortion of the grids in the first 10 μm beneath the surface was so severe that the grid intersections could not be identified. However, the strain evolution profiles in Figure 6.31 (e) show that despite the inability to resolve the grid lines in the white layer region, the plastic strains at the machined surface must exceed 0.7.

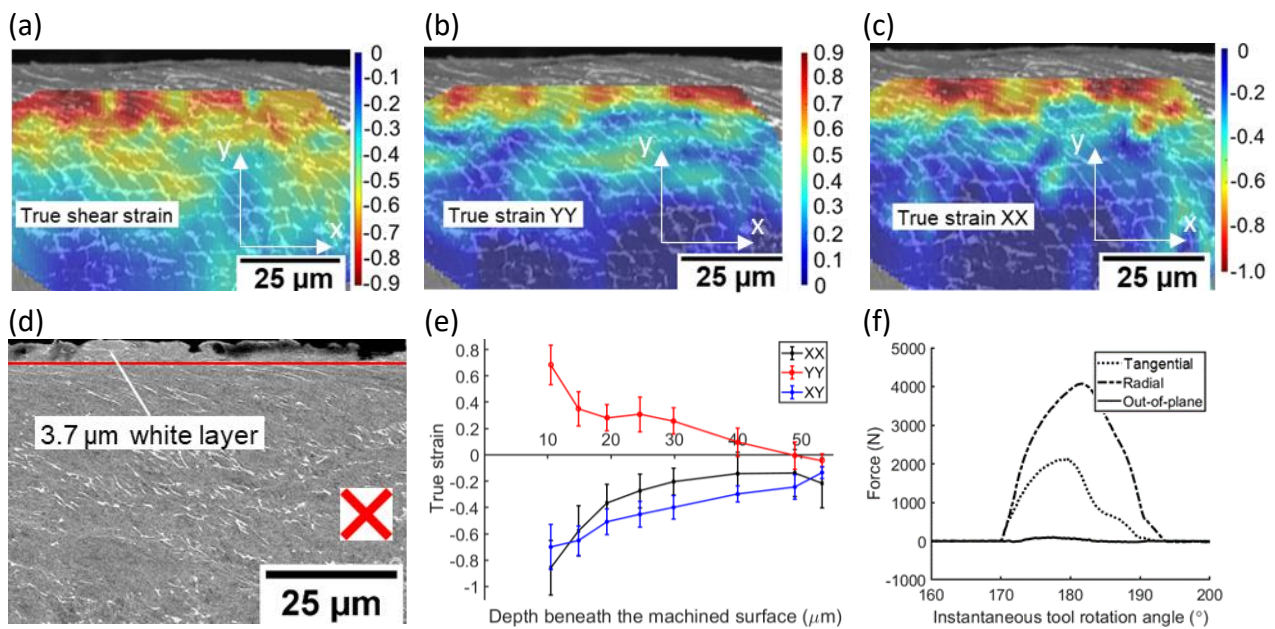


Figure 6.31 – (a-c) True strain maps, (d) an SEM image of the strain map region, (e) the true strain profiles with depth and (f) the corresponding forces during machining for a workpiece machined with a naturally worn insert (trial 2).

The larger strain when machining with naturally worn inserts is reflected in the increased white layer thickness, visible in Figure 6.31 (d), relative to surfaces machined with artificially worn inserts in Figure 6.25 (b) and (c). It is apparent from trends visible in the strain profile in Figure 6.31 (e) that despite the inability to resolve the grid lines in the white layer region, the strains at the machined surface are all beyond 0.7. As is consistent with previous results presented in this chapter for a surface with a white layer,

the radial cutting force was larger than for a surface with good surface integrity, reaching values as high as 4000 N, as shown in Figure 6.31 (f).

6.1.7.4 The effect of climb vs conventional milling on strain evolution in the machined surface

Figure 6.32 (a) and (b) compares the strain measured in the workpiece near-surface following climb and conventional milling, respectively. It can be seen that the maximum strain was larger during climb milling, however, the depth of deformation, as shown in the strain profiles in Figure 6.32 (c) and (d) is slightly larger for the conventional milling operation. For both operations, the radial force reached a maximum of approximately 3300 N, as shown in Figure 6.32 (e) and (f), but the tangential force during the conventional milling operation was greater than for climb milling. Figure 6.32 (g) and (h), show that no white layer was produced in the vicinity of the strain maps for climb or conventional milling, however, it has been shown previously in Figure 6.25 that a white layer was only produced during climb milling. The formation of white layers in the surface machined through climb milling can be attributed to the higher maximum deformation levels during this operation.

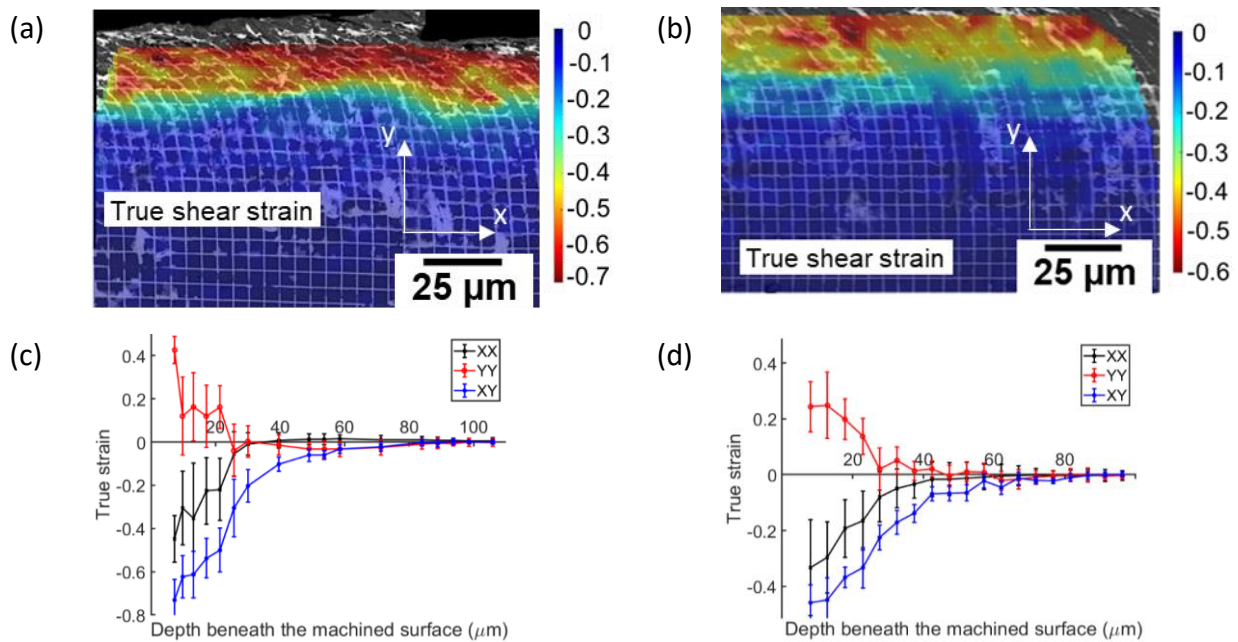


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Figure 6.32 continued

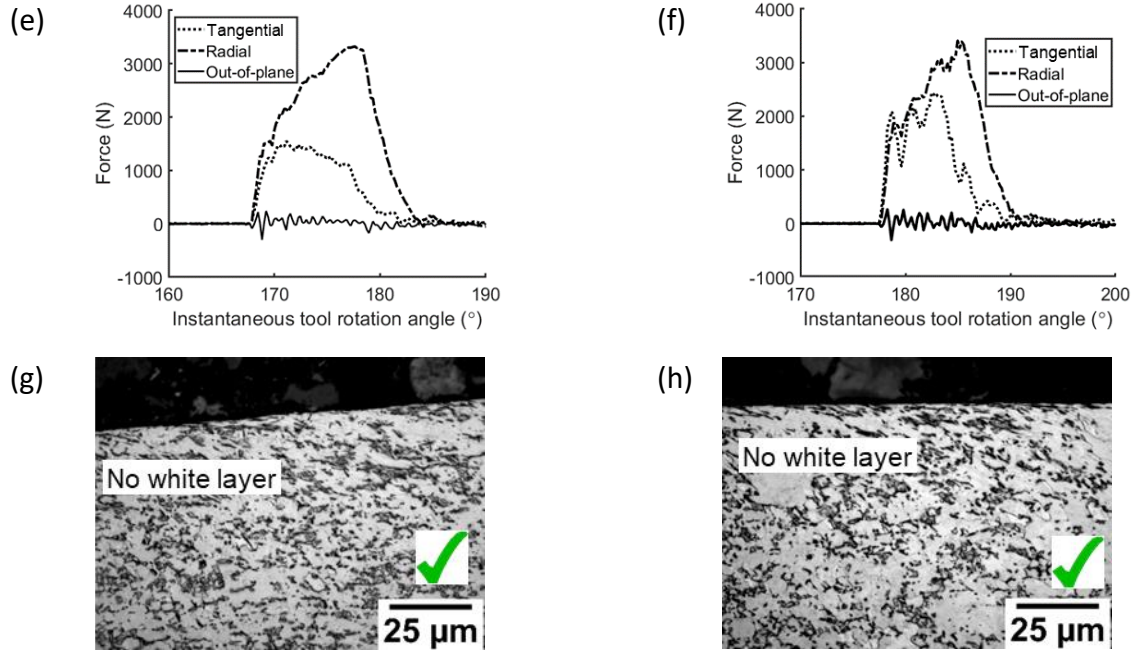


Figure 6.32 – True shear strain maps for a surface machined using (a) climb milling (trial 11) and (b) conventional milling (trial 12) with corresponding true strain profiles in (c) and (d), respectively. The forces measured during machining are shown in (e) and (f) for climb and conventional milling, respectively, and micrographs of the surfaces are shown in (g) and (h).

6.1.7.5 The effect of cutting speed on strain in the machined surface

The strain imparted in the workpiece near-surface at high (80 m/min) and low (20 m/min) cutting speeds is shown in Figure 6.33 (a) and (b), respectively. It can be seen that the use of lower cutting speeds results in higher strain levels in the immediate subsurface, up to an extra 0.3 true strain above the higher speed condition. It can be seen from the strain maps in Figure 6.33 (a) and (b) and the strain profiles in Figure 6.33 (c) and (d) that the depth to which deformation was measured was larger at 20 m/min. The cutting forces for the two different cutting speed conditions, as shown in Figure 6.33 (e) and (f), indicate that the radial force was larger for the lower cutting speed, therefore giving rise to enhanced deformation for this surface. Both high and low cutting speeds resulted in white layer formation in the rubbing region of the cutting arc, as shown previously in Figure 6.25 (b) and (c), however, only the low-speed condition gave rise to white layer formation outside of the rubbing region, as shown in Figure 6.33 (g) and (h).

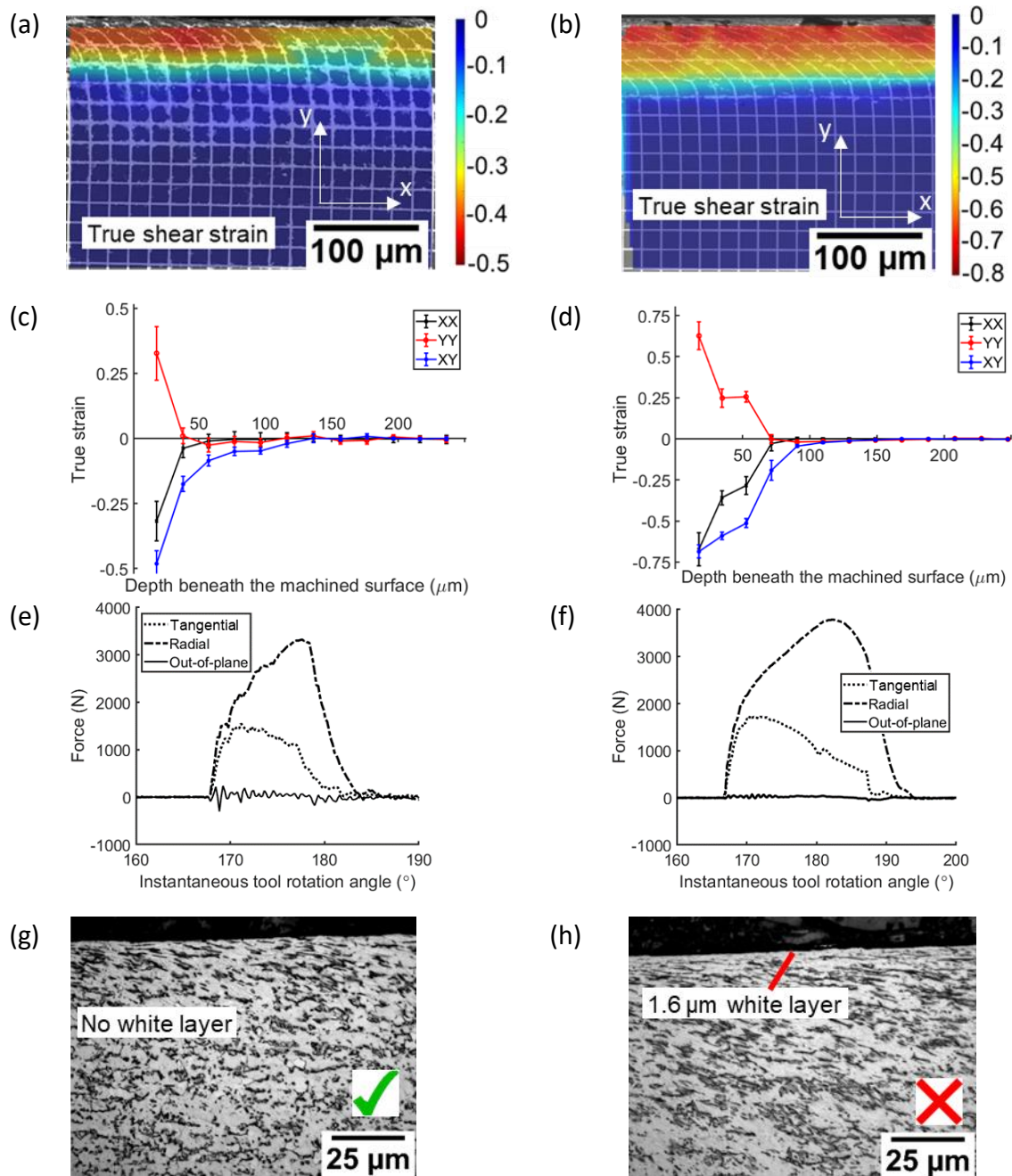


Figure 6.33 – True shear strain maps for a surface machined at (a) 80 m/min (trial 11) and (b) 20 m/min (trial 7) with the corresponding true strain profiles shown in (c) and (d), respectively. The forces measured during machining are shown in (e) and (f) for high and low speed, respectively, and micrographs of the surfaces are shown in (g) and (h).

6.2 Discussion

6.2.1 Key physical properties of white layers

The results from the destructive characterisation of Ti-6Al-4V, IN-718 and SCM423 machined surfaces indicate that white layers are usually formed when machining with worn cutting inserts, are composed of ultra-fine grains, are harder than the bulk material and can exhibit different surface residual stress states

and crystallographic texture. White layers in titanium, specifically, possess a strong basal texture and show an increased concentration of alpha stabilizing elements.

The white layers in all three materials contain ultra-fine grains, smaller than is resolvable with a FEG-SEM, as shown in the SEM micrographs in Figure 6.2 and Figure 6.3. From quantification of the grain diameter in neighbouring regions, where the grain structure is more resolvable, it is predicted that the grains in the white layers must be smaller than 100 nm. Ultra-fine grain sizes in the white layer have been reported previously in hard turned steels [73] and a nickel superalloy [77], as such the comparable observations in the materials studied here is not surprising. Although the white layers resemble the α -phase in optical micrographs of Ti-6Al-4V surfaces, heat-treatment of the white layer has shown that the β -phase is still present, in a refined form, at the α -phase grain boundaries, as presented in Figure 6.4.

A lower elastic modulus near the machined surface, as measured in a number of Ti-6Al-4V samples, has also been reported previously for a nickel superalloy [64]. This reduction was attributed to grain refinement, however, it has been reported that elastic modulus reduction due to grain refinement is only significant for grain sizes below 10 nm in nanostructured material [267]. As such, the significant reduction measured here for Ti-6Al-4V cannot be solely due to this mechanism. Instead, the lower elastic modulus in the white layer is thought to be primarily due to nanoindentation into the prismatic planes when performing measurements on a cross-sectional sample, because of the strong crystallographic texture of the white layer. It has previously been shown that indentation in the prismatic HCP plane produces lower measured values of elastic modulus [268], as such the texture anisotropy of the white layer is thought to significantly affect these elastic modulus measurements. The absence of an increase in elastic modulus in two of the samples could be a consequence of indentation in a region with different crystallographic orientation as the basal texture in the white layer is not entirely uniform.

For SCMV, there was a slight increase in elastic modulus in some cross-sectional samples both with and without white layers so the change cannot be attributed to the presence of a white layer and is most likely instrumentation variation due to indentations near the edge of the sample. In the tapered sample, where more indentations could be undertaken within the white layer, the elastic modulus was instead measured to decrease, as such there is no consistent trend in elastic modulus when a white layer was present in SCMV. There was similarly no consistent relationship for IN-718 samples. The comparable grain size of the

white layer in SCMV and IN-718 relative to Ti-6Al-4V and the lack of a consistent change in elastic modulus indicates that grain size effects are negligible factors in these nanoindentation measurements. It has already been shown that there is only a small change in texture between machined surfaces with and without white layers in SCMV and IN-718, therefore the lack of a significant change in elastic modulus is unsurprising.

The white layer in each alloy was measured to be harder than the bulk material and this can be attributed to a combination of work hardening, grain size and the effects of crystallographic orientation. The lower hardness in the cross-section of the white layer in Ti-6Al-4V, compared with the values measured on the machined surface, as shown in Figure 6.16, can be linked to the basal crystallographic orientation of the white layer, because indentation into the prismatic plane results in a lower hardness than the basal plane [39]. However, the hardness of the Ti-6Al-4V white layer in the cross-section is still greater than the bulk material hardness, despite its prismatic orientation, so this increase in the cross-sectional samples must be attributed to a combination of work hardening and grain refinement, which more than offsets the hardness reduction from the change in crystallographic orientation relative to the bulk.

Increased hardness in the white layer of a nickel superalloy [64] and a grade of steel [70] has already been reported in the literature. However, the values reported in each study describe that the hardness increases by as much as 60% and 25%, respectively, relative to the bulk and this is greater than the percentage changes reported earlier in this chapter. The increase in hardness in the white layer in SCMV can be attributed to strain hardening and the reduced grain size in the white layer, which provides additional barriers to dislocation motion, relative to a large-grained microstructure. The absence of a strong crystallographic texture shift between machined SCMV with and without a white layer, in comparison to titanium, means that hardness variations arising from orientation effects should not be significant.

The hardness change in the white layer in IN-718 is around a 10-30% increase relative to the bulk. Whilst the hardness increase relative to the bulk can be attributed to strain hardening and grain refinement, the increased hardness through the white layer to the swept grain region in one of the samples could be related to the effects of dynamic recrystallisation, because a similar behaviour has been reported

previously for this material [133]. Kruk et al. [133] attributed the lower hardness of the white layer, compared to the swept grain region, to the dissolution of the γ'' precipitates under the heat introduced to the surface during machining.

Hardness does not reach the bulk value immediately beneath the white layer in IN-718, SCM4V or Ti-6Al-4V machined surfaces due to additional strain hardening in the swept grain region. Strain introduced by the machining process exceeds the depth to which white layers are observed, as indicated by the distorted material visible in optical micrographs throughout this chapter, as well as the considerable weight of evidence provided by the micro-grid strain measurements.

The basal texture of the titanium white layers generated with worn tools can be attributed to the large compressive forces exerted on the workpiece during cutting. This compression results from the high pressure applied by the machining centre to keep the cutting insert at the desired depth of cut, in combination with the large contact area of the ground inserts. It has already been stated [269] that the c-axis of the HCP crystal re-orientates under compression towards the axis of loading, therefore indicating that the texture in the white layer is dominated by compression. It is clear from the investigation into different initial workpiece textures in Ti-6Al-4V that white layer formation cannot be avoided by altering the forming route for the workpiece material prior to machining.

The texture in the swept grain region of the Ti-6Al-4V samples, as well as surfaces with good surface integrity, is characteristic of the shear deformation that is visible in optical micrographs of these samples and has been observed elsewhere in the literature. A comparable shear texture has also been reported previously in magnesium, another HCP metal, after large strain extrusion machining [270]. This basal pole rotation under macroscopic shear loading during machining can be attributed to the alignment of the basal planes of the HCP crystal with the shear plane, facilitating deformation via basal slip [271].

The opposite direction of the shear texture for in a Ti-6Al-4V sample with good SI and a swept grain sample can be attributed to the nature of the cutting operation when machining with a worn insert. Using a worn insert results in additional rubbing without chip formation during the second half of the tool rotation, as shown in Figure 6.34. By contrast, for a new insert, the forces shown in Figure 6.8 (a) indicate that there is only cutting with chip formation in the first half of the tool rotation and very little rubbing during the second half. Thus, the final tool pass direction that deforms the workpiece surface for a worn

insert is in the opposite direction than that for a new insert when considering the central region of the machined shoulder width, resulting in a different texture direction.

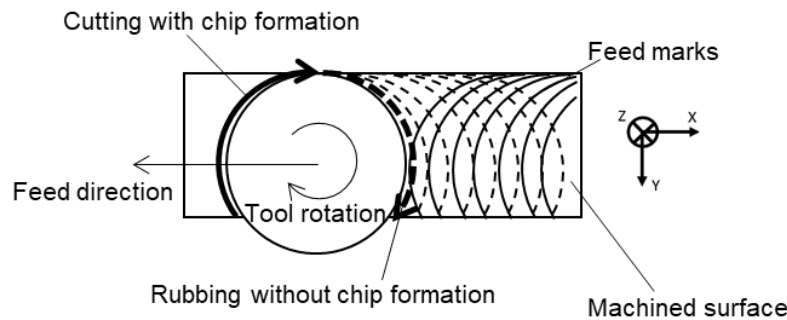


Figure 6.34 – Schematic indicating the key regions of the tool rotation responsible for the difference in shear textures between worn and unworn inserts.

The shear texture recorded for a white layer in turned IN-718 has previously been reported [272], so the comparable texture measured here for a milled surface is to be expected as phase transformation was not judged to have occurred. Whilst the texture in SCMV white layers has not been observed elsewhere, white layers in other steel alloys have been shown to exhibit a shear texture when the phase transformation mechanism was not activated [143]. This deformation-dominated shear texture, alongside results from XRD phase analysis, is therefore evidence that the white layer formed in SCMV is not the result of a phase transformation. It should be noted that the shear texture observed here could also be considered as a compressive texture rotated about the feed direction due to the shearing of the workpiece in the cutting direction.

Although a shear texture was identified for both the as-received SCMV and the surfaces machined with and without worn inserts, the direction of the shear texture was reversed in the as-received material. The reason for this difference can be attributed to the prior turning of the as-received material using a right-handed tool holder, whereas a left-handed tool was used in this study, resulting in a cutting velocity and shear texture in the opposite direction.

The large compressive forces at high levels of wear that were measured during white layer formation in all three alloys when cutting with artificially worn, ground inserts have also been previously observed when milling IN-718 [249] and when hard turning steel [75], with naturally worn inserts. One study [75] specifically identified that a higher compressive force accompanies white layer formation.

Due to the $\alpha + \beta$ microstructure of the as-received titanium material, a higher concentration of retained- β and or the emergence of α'' would be expected in the case of phase transformation under high temperatures during white layer formation, as observed in the shear band of titanium chips [97]. These $\alpha \rightarrow \beta$ and $\beta \rightarrow \alpha''$ transformations would be observable through XRD measurements, however, it can be seen from the XRD patterns shown in Figure 6.5 (a) that there was no significant β or α'' transformation. Therefore, white layer formation at low cutting speeds and using a tool with high levels of wear in titanium alloys does not appear to involve a temperature-induced phase transformation.

Similarly, for SCMV, a higher concentration of retained austenite would be expected if the white layer had formed via the phase transformation mechanism, as has been reported in the literature [143]. The absence of any peaks corresponding to the austenite phase in Figure 6.5 (b) suggests that the white layer has not formed via the phase transformation. This hypothesis is also supported by the results from optical microscopy in which no over-tempered dark layer was observed beneath any of the white layers.

In nickel superalloys, the matrix and precipitate phases all possess a coherent FCC crystal structure. As such, the dissolution of these precipitate phases would not be observable on an XRD pattern as diffraction angles associated with the crystallographic planes of these phases overlap. Additionally, the orthorhombic δ -phase is not visible in the XRD patterns for any surface due to its small volume fraction. It is therefore apparent that XRD phase analysis provides no evidence for or against the white layers observed in IN-718 being formed by a phase transformation mechanism. However, the deformation visible on the swept grain region and the deformation-dominated shear texture suggests that the white layers were formed via the SPD mechanism.

The contrasting near-surface residual stress states measured in the two Ti-6Al-4V samples with white layers, as shown in Figure 6.19 (a), indicates that white layers in titanium cannot be associated with a particular residual stress state. This difference in residual stress between the two surfaces must be a consequence of the different thermomechanical loads imparted on the surface by machining at the particular combination of cutting parameters and tool conditions used to generate the surfaces. The surface with an 8 μm white layer, which contains a smaller compressive residual stress near the surface, was machined at 80 m/min, compared to 40 m/min for the other surface with a white layer. Higher cutting speeds result in a greater rate of heat generation due to the higher strain rates imposed and this can

encourage a more tensile residual stress, as shown by Velásquez et al. [65]. However, despite the different thermomechanical loads experienced, the same metallurgical change, white layer formation, was observed. At depths greater than 50 μm , both surfaces with a white layer have significantly greater compressive residual stresses than the surfaces without a white layer, due to the enhanced plastic deformation when cutting with worn inserts. The depth to which residual stress was measured far exceeds the depth of deformation indicated by the thickness of the swept grain region due to the gradual accommodation of the plastic deformation at the surface, with increasing depth.

Large compressive residual stresses were measured near the machined surface and at greater depths for all surfaces containing a white layer in SCMV. Compressive stress was greatest in the feed direction for these samples, as has previously been recorded for steel surfaces containing a white layer [108]. The large compressive stresses in SCMV provide additional evidence that the phase transformation mechanism was not responsible for white layer formation, as the heat required for phase transformation typically results in tensile residual stresses [150].

It should be noted that all samples with white layers in Ti-6Al-4V and SCMV showed increased XRD peak broadening relative to all other samples. Broadening of XRD peaks, as outlined previously, is primarily caused by a reduction in grain size, typically significant for grains below 200 nm in diameter [146], and lattice distortions, such as those caused by an increase in dislocation density [146]. The small grain size of the white layer region has been shown through FEG-SEM imaging and an increase in dislocation density is expected in the white layer and swept grain regions due to SPD. Therefore, an increase in peak breadth coinciding with the presence of a white layer is to be expected, however, the peaks used for peak breadth measurements were high diffraction angle (140° and 152°) peaks and at these high angles, lattice strain effects can dominate [146]. The dominance of lattice strain effects is evidenced in Ti-6Al-4V by the increased peak breadth at depths beyond the white layer as well as for machined surfaces without a white layer. Due to their comparatively larger grain size, peak broadening cannot be due to grain size effects in these surfaces containing no white layer. Therefore, peak broadening of this high angle peak used in the XRD measurements must be largely due to lattice distortions introduced in the near-surface region during machining. The broadening of XRD peaks due to lattice strain in machined surfaces without a white layer is to be expected, as shear textures were observed for these surfaces, indicating near-surface deformation. It should be noted that these lattice strains in all machined surfaces cannot be solely due to the

macrostrains that give rise to the measured residual stress profile, as the two Ti-6Al-4V surfaces with a white layer exhibited different surface residual stress states, yet exhibited comparable near-surface peak breadths.

It is apparent for the surfaces with a white layer that the effect of machining on the workpiece surface integrity extends far beyond the swept grain region. This is because peak broadening and residual stress are still different from their baseline values, even at a depth of 350 μm and 800 μm in Ti-6Al-4V and SCMV respectively.

The increase in the concentration of O, N, C, Co and W in the near-surface for Ti-6Al-4V samples containing white layers, relative to a machined sample with no white layer, is not thought to cause white layer formation. This is due to the discrepancy between the white layer thickness and the depth to which the elemental concentration was measured to be above bulk levels. The relatively high concentration of carbon to depths of nearly 10 μm in the surfaces with a white layer, as well as the presence of cobalt and tungsten, is thought to arise due to tool-workpiece contact. The cutting inserts used in the trial were tungsten carbide with a cobalt binder, therefore, C, W and Co can be accounted for by the transfer of atoms between the tool and workpiece. In an attempt to explain the element transfer, a maximum tool contact time of 0.05 seconds (s) was calculated. This contact time was found using the size of the tool wear land (0.5 mm x 2 mm), tool diameter (20 mm), the cutting speed (40 m/min), the feed (0.06 mm/rev) and the knowledge that the tool contacts each workpiece region multiple times, due to the length of the contact patch and the two halves of the tool rotation.

With a contact time (t) of 0.05 s , for tool elements such as carbon to diffuse a distance (x) of 5 μm , the required diffusion coefficient (D) can be calculated using Fick's second law, as described in [5], by using the relative DSIMS intensities in the bulk (C_{bulk}), at a depth of 5 μm ($C_{5\mu\text{m}}$) and the surface ($C_{surface}$) as shown in Eq. 6.1.

$$\frac{C_{5\mu\text{m}} - C_{bulk}}{C_{surface} - C_{bulk}} = 1 - \text{erf}\left(\frac{x}{2\sqrt{Dt}}\right) \quad \text{Eq. 6.1}$$

$$\frac{2 \times 10^3 - 2 \times 10^2}{6 \times 10^3 - 2 \times 10^2} = 0.310, \quad \text{erf}\left(\frac{x}{2\sqrt{Dt}}\right) = 0.69, \quad \frac{x}{2\sqrt{Dt}} \approx 0.73,$$

$$D = 2.35 \times 10^{-10} \text{ m}^2/\text{s}$$

This is greater than the diffusion coefficient for interstitial diffusion of carbon in titanium at temperatures near the β -transus [273]. Considering the temperature during machining did not cause a phase transformation and so cannot have reached the β -transus, the diffusion of carbon into the surface cannot be explained by the diffusion that would result from a cutting tool simply being held against the workpiece at high temperature. The enhanced diffusion is thought to be facilitated by the fine grain structure of the white layer and the SPD caused by the rubbing of the ground wear region of the inserts over the machined surface on numerous tool passes. Grain boundary diffusion rates are enhanced in nanocrystalline material, as identified previously, due to a larger grain boundary area [136], particularly when the nanostructure is formed by SPD [137]. Thus, it is predicted that the nanocrystalline structure of the white layer in addition to the SPD of the surface facilitates enhanced diffusion rates of tool constituent elements, rather than the alternate situation where the white layer is formed because of alpha stabiliser diffusion.

The diffusion of oxygen and nitrogen into the machined surface is not reliant on tool contact, instead, time at high temperatures can lead to surface enrichment of these alpha stabilizing elements. However, the time required at high temperatures to form alpha-case is of the order of hours rather than seconds [274], as such, despite the diffusion of alpha stabilizing elements, alpha-case is not thought to have formed. This is also evidenced by the β -grains that were seen to be present within the white layer in the sample heat-treated beneath the beta transus, as shown in Figure 6.4.

6.2.2 The formation mechanism for white layers in titanium

As highlighted in the previous section, the basal texture measured in samples with a white layer is indicative of compression acting normal to the machined surface. It has already been stated in the literature review that reorientation of the HCP crystal under compression is due to activation of the $\{10\bar{1}2\}$ tensile twinning mode, which involves 85° pole rotation [35]. Also, twinning, rather than slip, is the dominant mechanism during compression at high strain rates and low temperatures [34]. The extremely high strain rates experienced during machining, when compared to other production processes, can therefore activate twinning as the dominant deformation mechanism. This is despite the high temperatures that could be experienced during the machining of titanium. Due to the 85° rotation in $\{10\bar{1}2\}$ tensile twinning in addition to the comparatively random pole orientation of the as-received material, for this

mechanism to occur there must be activation of additional slip or twinning systems before final $\{10\bar{1}2\}$ twinning in a proportion of the grains.

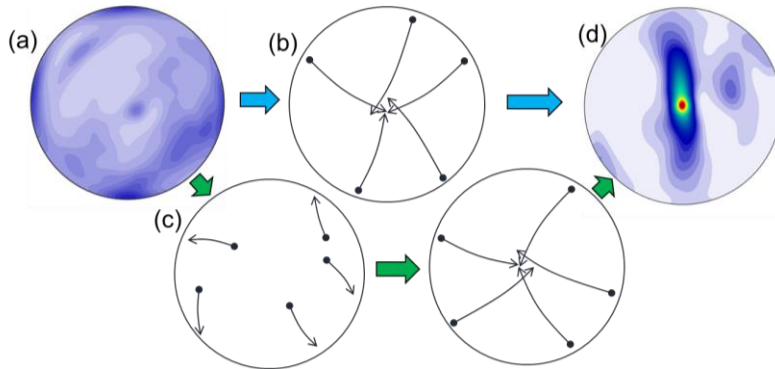


Figure 6.35 – Schematic outlining the possible routes to a basal texture from the starting workpiece texture: (a) (0002) Pole figure for the as-received material. (b) Basal pole rotation during $\{10\bar{1}2\}$ tensile twinning. (c) Basal pole rotation in which the specific slip or twinning system involved depends on prior orientation followed by $\{10\bar{1}2\}$ tensile twinning. (d) The basal texture in the white layer.

The prior crystallographic orientation of the structure, as shown in Figure 6.35 (a), dictates which specific systems drive the re-orientation of a particular grain [37]. Therefore, in this case of white layer formation, the large-scale re-orientation of the HCP crystals occurs, depending on the prior orientation, either by $\{10\bar{1}2\}$ twinning as shown in Figure 6.35 (b) or via other deformation mechanisms initially, followed by final activation of the $\{10\bar{1}2\}$ twinning mechanism, as shown in Figure 6.35 (c). The HCP re-orientation route that occurs will be the one with the lowest energy; however, this energy will be dictated by the initial crystallographic orientation of a grain. The $\{10\bar{1}2\}$ twinning deformation mechanism results in the basal texture presented in Figure 6.35 (d), as it accommodates tensile strain perpendicular to the compression direction [36] and so is the most stable HCP orientation under the applied machining conditions. Additional deformation modes must be involved prior to final $\{10\bar{1}2\}$ twinning, because the same basal texture was observed in the white layers of FAST workpieces that originally possessed a random texture and were, therefore, orientated unfavourably for activation of this twinning mode.

The lack of a temperature-dependent $\alpha \rightarrow \beta$ phase transformation in the white layer measured from XRD phase analysis, in addition to the distorted microstructure visible immediately beneath the white layer, indicates that SPD could be responsible for the observed grain refinement. The material in the white layer region is experiencing higher temperatures, due to heat generation during strain, and large forces over a very short time during machining, therefore, a form of recrystallisation may have occurred to

generate the fine microstructure shown in Figure 6.2 (b). Considering the duration of the events during machining, this is anticipated to be DR.

At high strain rates, such as those that can be imposed during machining operations, CDR prevails over DDR due to the short time at high temperature [101]. Additionally, the texture resulting from CDR is typically representative of the deformation i.e. the recrystallisation has little effect on the texture, whereas in DDR the deformation texture is weakened [99] and it can be said that the texture is indicative of recrystallisation. The resulting crystallographic texture of the white layers formed by machining with worn tools is a strong compressive deformation texture, as shown in Figure 6.7 (a), rather than a recrystallisation texture. Therefore, it is believed that CDR has occurred in the generation of the microstructure of the white layer, driven by SPD. The limited time at high temperature restricts grain growth such that the grains within the newly formed white layer are as much as 100 times smaller than the bulk material grain size.

It is apparent that the mechanism of white layer formation in Ti-6Al-4V when using worn inserts at speeds below 100 m/min, as presented in this study, is different in a number of ways from those which have been proposed for the refined grain structure in Ti-6Al-4V machined at speeds above 250 m/min speeds [96]. When using worn tools and low speeds, there was no precipitation of the β -phase, indicating that the temperatures reached were not as large compared to high-speed milling. It was also identified that the $\{10\bar{1}2\}$ twinning mode is more dominant when machining with worn inserts, compared to the $\{10\bar{1}1\}$ mode that has been identified in high-speed milled titanium [96]. However, it should be noted that CDR is the mechanism attributed to grain refinement in both cases.

The results from the investigation into the strain imparted into the machined surface during orthogonal milling support the idea that SPD is driving the white layer formation. It was not possible to measure the strain in the white layer itself, due to the comparatively large grid size relative to the white layer thickness, however, profiles of true strain with depth suggest that the magnitude of the true shear, XX and YY strain within the white layer region exceeds 0.7. This is similar to the findings which have been published for white layers in steels [153]. This value can be predicted because, despite the inability to resolve the grid structure within the white layer region, comparable strain values were measured in these samples at a larger depth beneath the machined surface compared to surfaces without a white layer. The increasing distortion of grains in the swept grain region nearer to the white layer, visible in optical micrographs, as

well as the strain profiles suggests that the strain increases at least linearly near the machined surface, if not with a higher order. The results from the strain trials therefore indicate that a true strain of the order of at least 0.7-0.75 is required for white layer formation through dynamic recrystallisation. Below these values, only SPD appears to occur, resulting in the formation of swept grain, a detrimental surface integrity feature nonetheless.

The positive value of the YY strain component near the machined surface for the sample machined with a large cutting edge radius, as shown in Figure 6.28 (e) and Figure 6.30 (f), means the material has extended, despite the compressive radial force on the surface in this direction. This extension can be attributed to excessive shearing of material in the primary shear zone in front of the cutting tool, which causes positive YY strain for material passing through the zone. However, not all material which passes through the deformation zone in front of the tool goes on to form part of the chip, instead, some material subsequently passes underneath the cutting edge [85] forming a region of material at the surface of the machined workpiece that has positive YY strain, as illustrated in Figure 6.36 (a). This deformation in front of the cutting tool is enhanced when the ratio of the uncut chip thickness to the cutting edge radius is smaller, as previously reported in the literature [114]. After passing underneath the cutting edge, the material machined with worn tools undergoes further deformation in the tertiary shear zone due to frictional contact between the tool flank face and the newly formed machined surface, as evidenced by the cutting force plots for worn inserts. Under this rubbing condition, the near-surface workpiece material is dragged in the direction of machining, resulting in increased shearing visible in the strain maps.

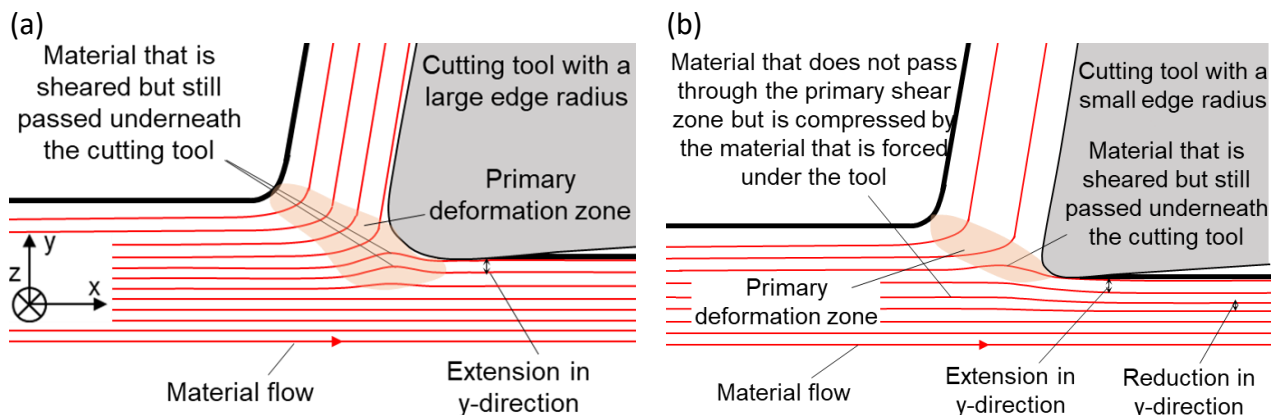


Figure 6.36 – Schematics showing (a) the extension of material in the y-direction near the machined surface for a cutting tool with a large edge radius relative to the uncut chip thickness and (b) the extension and reduction of material in the y-direction near the machined surface for the case where the cutting edge radius is smaller relative to the uncut chip thickness.

In workpieces machined with new inserts and ground inserts with a small cutting edge radius, the positive YY strain at the surface followed by negative strain at greater depths, as shown in Figure 6.29 and Figure 6.30 (c), can also be attributed to chip formation. The material nearest the surface is sheared in the primary zone by the material that goes on to form part of the chip, so it is elongated. By contrast, the material at greater depths does not experience the same strain as it does not pass through the primary shear zone, instead, it is only compressed when the material which passed through the primary shear zone, but did not form part of the chip, passes underneath the cutting edge, as shown in Figure 6.36 (b). The tertiary shear zone will also contribute significantly to the deformation in the subsurface due to the effect of frictional contact between the workpiece and both the tool nose and flank face. The same compressed region is not observed for the trials with larger cutting edges due to the greater extent of the deformation zone in front of the tool for a larger edge radius.

6.2.3 The effect of tool and cutting parameters on white layer formation

The formation of white layers in the orthogonal milling trials, which only occurred when machining with worn inserts, in addition to the large strains measured relative to the surfaces machined with unworn inserts, suggests that tool wear is fundamental to white layer formation through SPD and dynamic recrystallisation. It can be inferred from the results presented in Figure 6.30, that the cutting edge radius had a significant impact on the strain imparted on the machined surface. For tools with identical 0.5 mm wear lands, an increase in the depth of deformation as well as the true strain values near the surface was recorded when changing from a 13 μm to an 85 μm cutting edge radius. A larger cutting edge radius increases the contact area of the tool and additionally raises the volume of workpiece material which experiences machining with a negative rake angle, encouraging ploughing of the surface [116]. As such, the increased depth and level of deformation with a larger edge radius is to be expected as a greater amount of the workpiece material is ploughed under the cutting tool, rather than forming part of the chip.

The dragging of the tool flank face across the workpiece surface is immediately apparent from the cutting force plots from the orthogonal milling trials. It can be seen from figure Figure 6.28 (e) and (f) that the maximum forces for the new tool occur in the early stages of the tool rotation which shows that material deforms in the initial stages of contact. By contrast, for a worn tool, high forces are observed for a larger range of tool rotations and the maximum force occurs in the radial direction during the latter stage

of contact. This highlights the considerable contact between the flank face and the workpiece surface which results in the dragging of the surface.

In the orthogonal milling trials, the naturally worn tool produced a surface with a thicker white layer and larger deformation in the near-surface region, as shown in Figure 6.25 and Figure 6.31, despite a smaller wear land compared to the artificially ground inserts. It can be inferred, therefore, that the effect of cutting edge radius on the resultant strain distribution in the machined surface was greater than that of the wear land. The 137 μm cutting edge radius of the naturally worn insert increased the amount of deformation arising from ploughing, relative to the unworn inserts with a 30 μm cutting edge, as well as both types of ground inserts with edge radii of approximately 85 μm and 13 μm respectively. This was because the cutting edge radius was comparatively large compared to the uncut chip thickness, i.e. the t_1/r ratio was smaller, and therefore more material was pushed into the tertiary deformation zone adding higher compressive stress and subsequent plastic deformation. It should also be noted that the progression of wear during machining with the naturally worn insert may have led to enhanced contact between the flank face and the workpiece, relative to the ground inserts, such that the region on the flank face of the insert that rubs the machined surface was larger for the naturally worn inserts. The flat wear land of the artificially worn inserts, as shown in Figure 6.26 (b), cannot contact the workpiece entirely across the full wear land width as the tool follows a curved path, yet the wear land is straight.

In the square shoulder milling trials, by contrast, the cutting edge-radius was held constant for new and artificially worn inserts yet white layers only formed when using inserts with artificial flank wear. It is clear that both cutting edge radius and flank wear are critical variables which require controlling to minimise white layer formation, the relative strength of their effects is dependent on the geometry of the cutting operation.

On all orthogonally milled surfaces where a white layer was formed, the desired 0.5 mm depth of cut was not achieved, as shown in Figure 6.24. The depth of cut error was most severe on the surfaces machined with naturally worn inserts, where the tool appeared to plough and rub the surface rather than remove the workpiece material as a chip. A failure to achieve the desired depth of cut was identified for both large (85 μm or greater) and small (11 μm) cutting edge radius inserts but not for new inserts with no wear land. The reason for the deflection must, therefore, be a consequence of tool and workpiece

deflection under the high forces with worn inserts, particularly in the radial direction, which prevented the tool from removing the desired amount of material. The reduced tangential force for the naturally worn insert, relative to the unworn inserts, as shown in Figure 6.23 (a), can also be attributed to a reduction in the depth of cut due to the deflection of the cutting tool under the large radial forces.

Climb milling, rather than conventional milling, is typically the preferred milling operation as it is more stable and reduces the amount of ploughing that occurs before the uncut chip thickness is large enough for chip formation. The enhanced rubbing measured for climb milling, evidenced by the strain map and profiles in Figure 6.32, is therefore unexpected. This result is thought to be primarily a result of the failure to achieve the full depth of cut, as the error on material removal was observed to be greater for climb milling. The radial forces measured during climb and conventional milling are of similar magnitude, as shown in Figure 6.32 (e) and (f). However, due to the unknown instantaneous chip thickness, which is affected by the depth of cut error, it is not possible to separate the radial forces due to chip formation from the radial forces due to ploughing and rubbing. As such the difference in the material removal error for these two milling modes cannot be explained by examining the machining forces.

The difference in the material removal for the two modes of milling can instead be attributed to the way in which the chip is formed, as this is the key difference between climb and conventional milling. It is proposed that, in these trials, when forming a thick-to-thin chip during climb milling, the high initial chip thickness led to early deflection of the cutting tool, under high cutting and radial forces, which was never subsequently recovered through the remainder of the tool rotation before tool exit. By contrast, for conventional milling, the initial chip thickness was small so the initial deflection of the tool was lower and when the tool was at the end of the cut, where the instantaneous chip thickness was larger, a chip was already being formed so the reduction in the depth of cut due to deflection was smaller.

White layers were only formed during orthogonal milling in the regions of the cutting arc where the cutting edge radius was larger than the theoretical instantaneous chip thickness, this is due to the effect of ploughing under these conditions. The failure to achieve the desired depth of cut due to deflection in trials with worn inserts means that the true instantaneous chip thickness at any point along the cutting arc will have been lower than it would have been for the full depth of cut. This means the ploughing effect

will have been exaggerated in these trials relative to the case of no tool deflection, as the ratio of the chip thickness to cutting edge radius will have decreased, resulting in enhanced deformation.

6.2.4 Micro-grid quality post-machining

The ability to resolve the grid intersections following deformation is fundamental to micro-grid strain measurements. Creating a small micro-grid does not guarantee the strain can be measured, as highlighted by the inability to measure strain in the white layer using 5 μm grids in Figure 6.31 (a-c), which indicated a limit to the applicability of this grid size for the measurement of extreme strains. The quality of the grids post-machining varied significantly and lessons can be learned from the examples of poor grids to improve the overall grid quality for future work. An image of a grid showing two of the main examples of grid imperfections, smearing and grid transfer is shown in Figure 6.37 (a), alongside higher magnification images of each feature in Figure 6.37 (b) and (c).

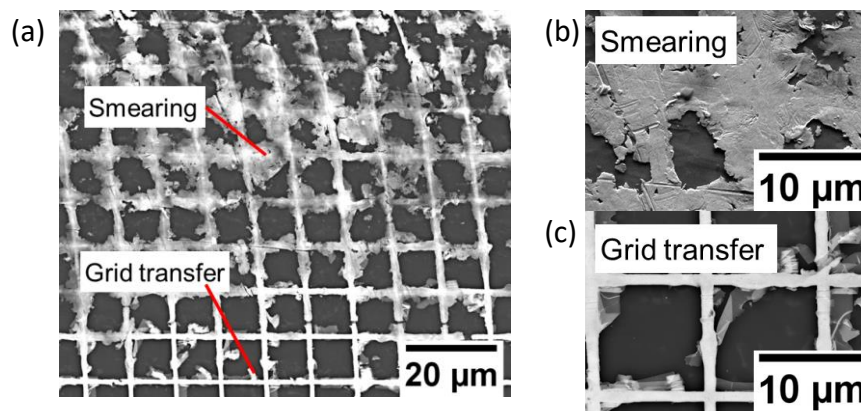


Figure 6.37 – SEM images of micro-grids post-machining: (a) smearing and grid transfer on a 10 μm grid, (b) a higher magnification image showing smearing and (c) a close up of transferred grid material.

The smeared appearance of grids such as those in Figure 6.37 (b) could arise via one of three mechanisms, deformation of the grids when the two plates are clamped together in the orthogonal workpiece holder, melting of the grids under the heat generated during deformation, or surface contamination from residual dried coolant. The melting point of pure gold, as used for the grids, is 1064 $^{\circ}\text{C}$, consequently, melting of the grids during machining would be evidence that this temperature was exceeded. It can be seen from Figure 6.38 (a) that the grids cannot have melted as the smeared grid region lies over 100 μm away from the machined surface, i.e. beneath the region of greatest strain and therefore heat generation. The composition of the smeared material, measured using SEM EDXS, is highlighted in

the composition map for gold in Figure 6.38 (b). It can be seen that the smeared material is gold, so it must arise from the deformation of the grids under the clamping force rather than residual dried coolant.

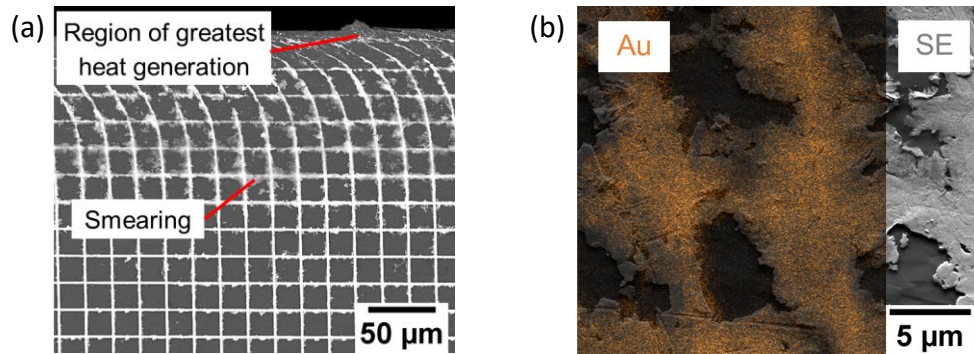


Figure 6.38 – Images of (a) a smeared region of micro-grid beneath an unaffected region and (b) an SEM-EDXS composition map for gold superimposed on a secondary electron image of the same region of the workpiece.

Due to the nature of printing physical grids onto the orthogonal workpieces rather than creating channels in the surface, the grids possess a finite size above the workpiece, in this case 100 nm, so the absence of any smearing of the grids under the clamping force is unavoidable. However, it may be possible to reduce the amount of smearing by decreasing the thickness of the gold layer that is deposited. Smearing occurs in a particular region on the surfaces due to the edge rounding of the samples, which arises from the mechanical polishing process. This rounding means that when two plates are clamped together and a shim is used to ensure plain strain in the grid region, a small region of material supports a large proportion of the clamping force. Consequently to reduce smearing, the polishing method used to prepare the orthogonal workpieces needs to be altered. Typically, over-polishing results in edge rounding, so the time spent polishing could be reduced.

Due to uncertainty about the most appropriate method for creating the gold grids with EBL, thermal evaporation deposition or sputtering, each orthogonal workpiece pair consisted of both types of grid, on opposite surfaces. Grid imperfections such as those in Figure 6.37 (c) were a consequence of the sputtered grids adhering to the thermal evaporation deposition grids and then peeling off from the workpiece when the workpiece pair was separated. The thermal evaporation deposition grids had greater adhesion, so this method of preparation is recommended for future work. Transfer of grids can be avoided by printing grids on only one of the workpieces in a pair.

The final issue encountered with strain measurement from the micro-grids was that of out-of-plane deformation, as highlighted in Figure 6.39, caused by burr formation at the machined edge when the

workpieces were not in contact near the machined surface. These grids could not be used for strain measurements, as deformation was not under the plane strain conditions required for the micro-grid method. Out-of-plane deformation can be avoided by minimizing edge rounding effects and ensuring workpiece contact prior to machining.

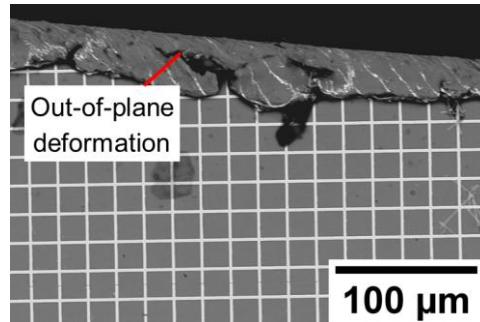


Figure 6.39 – SEM Micrograph showing out-of-plane deformation that has arisen because of poor workpiece contact.

6.2.5 The potential for non-destructive detection of white layers in aeroengine alloys

It has been identified in this chapter that the white layers in all three aeroengine alloys have a nanoscale grain structure, consequently, a non-destructive technique that can detect white layers based on this property offers the optimum solution. This is because the technique would be sensitive to an intrinsic property of the white layer and could be applied to any of the aeroengine alloys, thereby increasing both the applicability and versatility of the solution. Smaller grain sizes and higher lattice strains give rise to XRD peak broadening, as highlighted previously, and results presented in this chapter have shown that XRD peak breadth is greater when a white layer is present. Therefore, it may be possible to detect white layers in aeroengine alloys non-destructively using XRD peak breadth analysis of machined surfaces. By focusing on peaks with smaller diffraction angles, which are more sensitive to grain size effects relative to strain effects, it may be possible to detect white layers through the effect of their small grain size on XRD peak breadth. Ultrafine grains are a fundamental property of the white layer so it is desirable to be able to detect white layers based on this property in the first instance, rather than a property such as residual stress, which is not intrinsic to the white layers themselves.

In addition to nanoscale grains, it has also been measured that white layers formed in Ti-6Al-4V have a particular crystallographic texture and this affects the relative intensity of different peaks in XRD measurements. As such, it may be possible to use XRD intensity measurements as an indicator of the presence of a white layer. The strong basal texture and the anisotropy of the HCP crystal in titanium

directly affect the elastic modulus of the white layer, as evidenced by the nanoindentation results. Thus, SAW testing may also facilitate the non-destructive detection of white layers via the effects of crystallographic texture on the propagation velocity of SAWs. This is because the velocity of a SAW wave propagating on a test piece is related to the material elastic constants, according to Farnell [185].

Finally, the large compressive residual stresses in machined surfaces with white layers in SCMV, in addition to their small grain size, may facilitate the detection of anomalous surfaces that contain white layers using Barkhausen noise, due to the effect of these properties on domain wall motion. However, such a test would not be detecting a fundamental property of the white layers, so is a less desirable solution than the XRD or SAW methods.

It is therefore proposed that the following methods be investigated for the non-destructive detection of white layers.

- A grain size-sensitive method (XRD peak broadening – all alloys)
- A texture-sensitive method (XRD intensity – Ti + Ni, SAW velocity - Ti)
- A residual stress-sensitive method (Barkhausen noise – SCMV)

In the next chapter, the key principles of these techniques are used to propose a set of methodologies for detecting white layers, which will then be tested.

6.3 Concluding remarks

- White layers in titanium, formed under the applied machining conditions, are composed of ultra-fine equiaxed grains with an average diameter of less than 100 nm. Both the α and β phases are present.
- The white layer is up to 15%, 25% and 45% harder than the bulk material in Ti-6Al-4V, IN-718 and SCMV respectively.
- Machining induced white layers in Ti-6Al-4V have a basal texture because of the compressive stress state imposed during machining with worn inserts. This results in a change in the elastic modulus of the white layer, due to the elastically anisotropic HCP structure.

- White layers in SCMV and IN-718 possess a shear texture, however, this is the same texture which is also observed for machined surfaces with good surface integrity.
- XRD phase analysis suggests that the white layers observed in this study did not form via an $\alpha \rightarrow \beta$ phase transformation mechanism in titanium or austenitisation in SCMV, but there was no evidence for or against transformation in IN-718. However, the shear deformation observed for IN-718 suggests a phase transformation did not occur for this alloy.
- Residual stress profiles beneath the machined surface indicate there is no single residual stress state that can be associated with the presence of a white layer in titanium. In steel, a large compressive residual stress was measured for all samples with a white layer.
- White layers in titanium can possess enhanced concentrations of alpha-stabilizing elements as well as elements transferred from the cutting insert. The presence of these elements was attributed to enhanced diffusion rates aided by the nanoscale grain structure and the SPD in the white layer. The discrepancy between the elemental composition at different depths and white layer thickness indicates that the white layers did not form because of the alpha-stabiliser diffusion.
- It has been proposed that white layers in Ti-6Al-4V are formed by continuous dynamic recrystallisation driven by SPD under the high strain and strain rates experienced during metal cutting.
- White layer formation in Ti-6Al-4V using heavily worn cutting tools involves the activation of the $\{10\bar{1}2\}$ tensile twinning mode.
- Micro-grid strain measurements indicate that a true strain greater than 0.7 is required to initiate white layer formation through SPD and dynamic recrystallisation in Ti-6Al-4V.
- Results from orthogonal milling experiments show that white layer formation is promoted when machining with cutting tools with a larger cutting edge radius. For these particular trials, climb milling led to white layer formation but conventional milling did not.

- A white layer was found to form only in regions, during orthogonal milling, where the cutting edge radius was larger than the instantaneous chip thickness, particularly for the case of rubbing with zero chip thickness.
- White layers in aeroengine alloys may be detectable using XRD peak breadth measurements, due to the high lattice strain and small grain size in the white layer.
- The strong texture of the white layers in Ti-6Al-4V and the anisotropy of mechanical properties caused by the HCP crystal structure may allow non-destructive detection using XRD intensity measurements or SAW testing.
- The large compressive residual stresses present with the white layers in SCMV may facilitate surface integrity assessment using Barkhausen noise inspection in this material.

7 Non-destructive methods for detecting machining induced white layers - methodology

This chapter describes non-destructive evaluation methods that have been designed to detect white layers in a machined surface based on the knowledge gained from the characterisation work discussed in the previous chapter and the key fundamental concepts of each method identified in the literature review. A grain size-sensitive method employing the XRD technique will be described, before introducing crystallographic texture-sensitive techniques, based on both surface acoustic wave methods and XRD. The final technique, a residual stress-sensitive method that utilises Barkhausen noise inspection for detecting white layers in ferromagnetic materials is also described.

7.1 White layer detection through XRD peak breadth measurement – XRD- Δ PB

The results presented in the previous chapter identified a nanoscale microstructure in the white layers of all three aeroengine alloys. The sensitivity of x-ray diffraction to small grain sizes through a peak broadening effect has already been highlighted in the literature review. As such, the methodology for an XRD technique for white layer detection based on the measurement of peak breadth (XRD- Δ PB), is introduced here.

To develop the XRD- Δ PB test, an Xpert³ powder diffractometer equipped with a Copper K α x-ray tube and a Ni filter was used to inspect machined surface samples in Bragg-Brentano geometry, i.e. the diffraction vector was normal to the surface [275]. The variation of x-ray intensity with diffraction angle was measured for samples of each of the three alloys in this study. The range of diffraction angles used was aligned with those previously outlined for the XRD phase analysis. Prior to the analysis of the results, each measurement was corrected for background intensity and smoothed with PDF-4+ [223] software using an 11-point moving average to filter out the high-frequency noise in the diffraction pattern. The FWHM of each x-ray diffraction peak was then calculated from the corrected data using a custom script in MATLAB. This was achieved by finding the peak intensity, then using linear interpolation between the points either side of the half-maximum value to quantify the diffraction angle at half-maximum on the rising and falling sides of the XRD peak. The FWHM was then taken the difference between these two half-maximum values. The determination of FWHM is illustrated in Figure 7.1 (a).

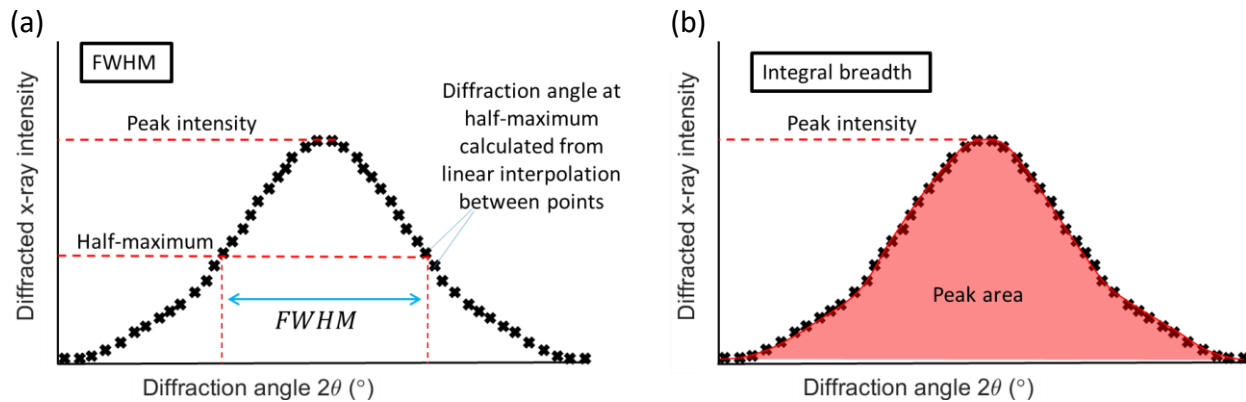


Figure 7.1 – A schematic outlining the determination of (a) FWHM and (b) integral breadth from an XRD pattern.

For comparison, the integral breadth (IB) of the x-ray peaks, another common measure of peak breadth, was also determined. IB is calculated from the area under the peak divided by the peak height, as shown in Figure 7.1 (b). To calculate IB, peaks with a known distribution, in this case a pseudo-Voigt, a linear combination of Gaussian and Lorentzian distributions [276], are fitted to the diffraction pattern and the area under the curves is calculated. IB can be useful for textured materials due to the consideration of peak intensity during breadth calculation, however, it is known to be less suited to situations where peaks tails are overlapping [277] as this adds uncertainty to the peak fitting process.

Due to anisotropic strain effects, arising from the machining-induced texture of each sample, the most appropriate crystallographic plane for detecting white layers was determined by comparing the breadth for each peak in diffraction patterns of at least five samples in each alloy. Following this, additional measurements were taken with a larger number of samples focussing on one specific peak in each material to assess the capability of the technique.

To establish the resolution of the technique for white layer detection, machined Ti-6Al-4V surfaces were electropolished (EP), as shown in Figure 7.2 to remove a portion of the white layer without the deformation introduced by mechanical polishing. This allowed the creation of samples with white layer thinner than the XRD inspection volume, because all as-machined samples had white layer thicknesses greater than 5 μm . EP samples were inspected using XRD before undergoing cross-sectional microscopy to measure the remaining white layer thickness.

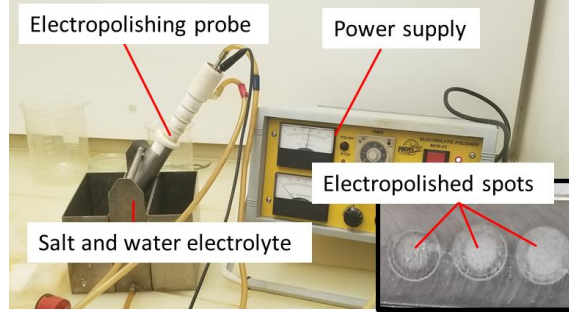


Figure 7.2 - The electropolishing equipment and associated electropolished spots on a machined surface used to investigate the effect of white layer thickness on the XRD method for white layer detection.

Following experimental investigations, a mathematical model was created to estimate the minimum detectable white layer thickness based on data from the experimental results, which was adjusted for the limited sample size by considering t -distribution tables [278]. This thickness ($t_{90/95}$) is the size for which there is a 90% probability of detection (POD) with 95% confidence [279]. To enable the determination of $t_{90/95}$, theoretical POD curves, showing the probability of detecting a white layer at different thicknesses were produced based on the mean and variance of the peak breadth distributions for samples with and without white layers.

The Williamson-Hall (W-H) method is a method commonly used to determine the relative contribution of inhomogeneous lattice strain and crystallite size broadening effects on the overall peak broadening during XRD measurements. Both the basic W-H method and modified W-H methods were used in this study to assess the relative contributions to the overall peak broadening in a surface containing a white layer. In the basic W-H method the total peak broadening can be considered the sum of the broadening from strain and crystallite size effects [280], as highlighted in Eq. 7.1 which is the combination of Eq. 4.7 and Eq. 4.8.

$$PB_{total} = \frac{k\lambda}{D\cos(\theta_{hkl})} + 4\epsilon \tan(\theta_{hkl}) \quad \text{Eq. 7.1}$$

This can be arranged into the form outlined in Eq. 7.2 and by plotting $PB_{total}\cos(\theta_{hkl})$ against $\sin(\theta_{hkl})$, the strain (ϵ) and crystallite size (D) can be determined, from the gradient and y-intercept respectively. However, this method is suitable only for an estimation of crystallite size and strain and additionally is ill-suited to highly textured solid samples containing a large number of dislocations [277]. Indicators of poor suitability to the W-H method include low R-squared values during linear regression analysis as well as strain anisotropy, as described next.

$$PB_{total}\cos(\theta_{hkl}) = \frac{k\lambda}{D} + 4\varepsilon \sin(\theta_{hkl}) \quad \text{Eq. 7.2}$$

In some cases, the peak breadth measured for different crystallographic planes does not vary exactly according to the relationships described above, instead, the peak broadening can also be dependent upon the particular crystallographic plane, this is known as strain anisotropy. In this situation, two planes with similar Bragg angles can exhibit vastly different peak broadening [281]. This deviation from expectation can arise due to the different elastic modulus values for crystallographic planes, which can result in anisotropic strain depending on the particular plane being studied [281]. Additionally, dislocations and their resultant strain field can effect peak broadening in a non-uniform manner such that the peak breadth is dependent on the activation of different slip systems and their relative contribution to the lattice strain [282, 283].

One study in which strain anisotropy was observed in nanocrystalline titanium is particularly relevant in the context of this work [284]. It was reported that the peak broadening obtained from XRD patterns was influenced by the relative content of different types of dislocations in different crystallographic planes. This led to a non-linearity of peak broadening with changing diffraction angle.

Modified W-H methods can be used to determine crystallite size and lattice strain for surfaces exhibiting strain anisotropy [277]. The anisotropic strain model [285] corrects for the different elastic modulus (E_{hkl}) of each crystallographic plane which results in different strains under the same load. The modified equation is given in Eq. 7.3, where σ is the lattice stress.

$$PB_{total}\cos(\theta_{hkl}) = \frac{k\lambda}{D} + \frac{4\sigma \sin(\theta_{hkl})}{E_{hkl}} \quad \text{Eq. 7.3}$$

The uniform energy density model [285] as described by Eq. 7.4, expands on the previous model by assuming the stress is also anisotropic. In Eq. 7.4, $u = \frac{1}{2}E_{hkl}\varepsilon_{hkl}^2$ and is the deformation energy per unit volume as a function of strain, from Hooke's law.

$$PB_{total}\cos(\theta_{hkl}) = \frac{k\lambda}{D} + \left(\frac{5}{2^{\frac{5}{2}}} E_{hkl}^{-\frac{1}{2}} \sin(\theta_{hkl}) \right) u^{1/2} \quad \text{Eq. 7.4}$$

However, even these modified methods do not take into account the direction of the x-ray diffraction vector compared to the dislocation line vector, which can lead to anisotropic peak broadening when

different types of dislocations are responsible for the lattice strain in grains of different orientation [283]. Ungár and Borbély [282] proposed a modification to the W-H method which uses dislocation contrast factors for different slip systems to account for this anisotropy. The analysis is complex and requires knowledge of the specific slip systems activated, the relative amount of edge and screw dislocations and the elastic constants of the material. Therefore, this type of analysis is beyond the scope of this study. However, due to the activation of different slip and twinning systems including $\{10\bar{1}2\}$ twinning during white layer formation, it is apparent that the XRD results will be affected by this dislocation contrast factor.

During W-H analysis on white layer samples, the peak breadths of different Ti-6Al-4V α -phase peaks were corrected for instrumental peak broadening using an annealed sample of the same material and then the dependence of peak breadth upon the diffraction angle was measured. It should be noted that IB was used as the peak broadening measure for the W-H and modified W-H methods.

Due to the limitations of the W-H methods when the dislocation contrast factor is significant, a number of samples were also heat-treated between 250-700 °C, as shown in Table 7.1, to facilitate the qualitative evaluation of the relative effect of size and strain broadening by inducing grain growth and lattice strain relief. The heat treatments were carried out in a vacuum furnace to prevent diffusion of elements such as oxygen into the near-surface, as this interstitial element could change the lattice spacing, resulting in additional peak broadening.

Table 7.1 – Heat treatments used in an attempt to separate crystallite size and inhomogeneous lattice strain broadening.

Temperature (°C)	Hold-time (mins)
250	12
350	12
450	12
550	12
700	120*

*note this is heat treatment used previously to grow the grains in the white layer for SEM analysis of the phase content.

The lower temperature heat treatments were used to anneal the white layer without significantly increasing the grain size and the higher temperatures were used to encourage grain growth. Subsequent XRD- Δ PB measurements and SEM imaging could be used to indicate whether strain or size broadening was playing a significant role in any peak broadening associated with the presence of a white layer. The lowest heat treatment temperature of 250 °C was selected as titanium processed through high pressure torsion,

which has a highly refined microstructure due to SPD, has been shown to anneal at temperatures as low as this, without visible grain growth [225, 286]. Zhilyaev et al. [225] also identified that grain growth became significant when heat treating above 300 °C. Below this temperature, changes in grain size were attributed to the disappearance of distortions near the grain boundaries.

A final small-scale study to investigate the potential for this XRD- Δ PB method to be used for swept grain detection was undertaken on Udimet-720 samples obtained from industry. These nickel superalloy samples were generated externally to this project and industry certified inspection had already taken place on them. The results of the destructive surface integrity inspection were only revealed after non-destructive testing so the inspection was blind.

7.2 White layer detection through XRD intensity ratio measurement – XRD- Δ IR

XRD may also be capable of facilitating the detection of white layers in titanium due to the effect of crystal orientation on the measured diffraction peak intensity. It has already been established that x-rays can be used to determine texture through pole figure measurements, however, it can take many hours to capture the required data. Simple XRD patterns plotting intensity against diffraction angle, as used in phase analysis, can also provide basic indicators of the texture. The measured intensity during XRD inspection is fundamentally related to the number of diffracted x-rays which reach the detector at a particular diffraction angle. In turn, this number is related to the area of material illuminated by the x-ray beam that has the interatomic spacing, and thus crystal orientation, required for diffraction of the incident x-rays. Therefore, the measured intensity at a particular diffraction angle is related to the texture of a material.

In addition to texture, experimental factors such as wavelength and specimen detector distance, alongside atomic factors such as multiplicity, composition and systematic absences [146] affect the measured intensity. These factors are not discussed in detail here as, although they can significantly affect peak intensities, they do not cause noticeable variation between XRD patterns of the same material examined under the same experimental conditions, with no phase changes. Variation, in this case, must arise due to changes in crystallographic texture between samples and thus, the relative intensity in an XRD pattern can be used as an indicator of texture.

The strong textures measured in machining induced white layers in titanium should alter the relative intensities of the peaks in an XRD pattern and allow detection of the highly textured white layer. To investigate the potential for such an XRD method, XRD measurements as described in section 7.1 were carried out. However for the XRD- Δ PB method, instead of calculating the peak broadening, the intensity of the peak associated with the basal texture of the white layer, (0002) in titanium, was measured along with the intensity of the neighbouring peak ($10\bar{1}1$), as highlighted in Figure 7.3. The ratio between the two peaks was then used as an indicator of the presence of basally orientated white layers.

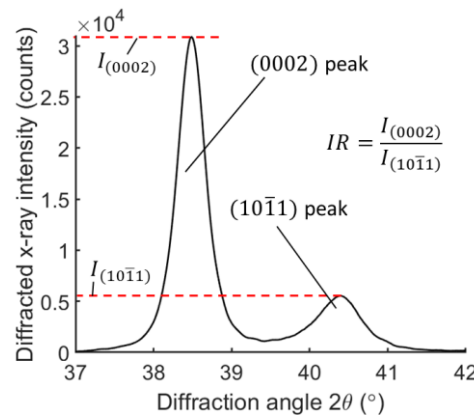


Figure 7.3 - A schematic outlining the determination of IR from an XRD pattern.

The neighbouring peak was selected to allow for faster scan times with limited goniometer movement during the XRD measurements, although, any second peak could be chosen as it is the relative height of the (0002) peak that is of interest. To quantify the relative strength of the as-received workpiece textures relative to samples with a white layer, intensity ratio (IR) measurements were undertaken on as-received material with different crystallographic textures, as it has already been shown that texture can vary considerably depending on the prior production route.

Although no distinct texture that separates surfaces with a white layer from other surfaces was identified from pole figures in machined IN-718 and SCMV, changes in the intensity of different peaks was recorded during pole figure measurements for SCMV and phase analysis measurements for IN-718 surfaces, when a white layer was present. This height change indicates that although there is no change in the type of texture in the near-surface when a white layer is present, there is a change in its relative strength. Therefore, IR measurements were carried out in IN-718 using the (111)/(200) peaks as these were measured to change in height during phase analysis measurements. Similarly, in SCMV IR

measurements were undertaken on the (110)/(200) planes respectively as the MRD in pole figures for these planes changed when a white layer was present.

7.3 A texture-sensitive SAW method for white layer detection

In addition to XRD, SAW methods have also been identified as NDT methods that are sensitive to crystallographic texture changes such as those that occur within the white layer in Ti-6Al-4V. Many forms of SAW inspection exist, however, in this study only the laser-induced SAW testing method SRAS was investigated, due to the non-contact nature of the inspection, which affords a more automated process. In SRAS, as outlined schematically in Figure 7.4, a pulsed ND:YAG laser passes through a grating pattern and forms a generation patch on the surface of the test piece [188]. This generates a surface wave due to thermoelastic expansion, wherein energy absorption causes a rise in local surface temperature, which causes the material under the laser to expand [287]. The wavelength, λ_g , of the generated wave is defined by the period of the grating pattern. The SAW velocity, v_s , is dictated by the material properties of the area of the test piece directly beneath the generation patch (200 μm in diameter). The frequency of the generated wave packet is then related to the wavelength and velocity through Eq. 7.5.

$$v_{SAW} = f_s \lambda_g$$

Eq. 7.5

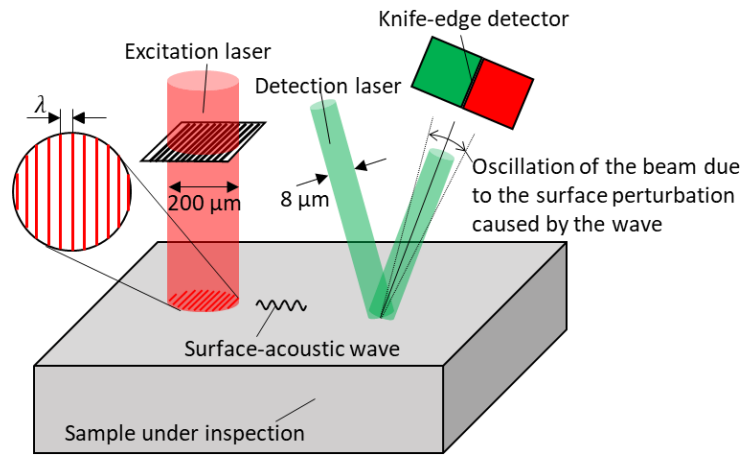


Figure 7.4 – Schematic of the SRAS system

The generated SAW propagates across the surface of the test piece and the perturbation caused by the passing wave is detected using a second laser adjacent to the excitation laser. The propagation distance between the generation patch and the detection laser is typically less than 50 μm . The detection laser is reflected off the surface and into a split photodiode detector, which measures the frequency of the laser's spatial oscillations caused by the passing SAW, as shown in Figure 7.4. The SAW velocity can then be

calculated from Eq. 7.5. This measurement process is then repeated over the entire surface of the sample using a raster scan, to build up a velocity map. The key advantage of SRAS frequency-based velocity measurement over time-based measurement in some other SAW methods is that although the velocity of the wave changes as it travels through different regions of material, due to elastic modulus differences, the frequency is unaltered. Consequently, the velocity found from the frequency measured by the detection laser is specific to the region of material underneath the generation patch. In time-based velocity measurement, the material between the transducer and detector would influence the velocity measurement.

To measure crystallographic orientation, SRAS velocity maps in multiple directions must be obtained and used to approximate velocity circles for each region of the sample. A velocity circle describes the angular dependence of velocity propagating on a particular plane. A larger number of scans therefore gives a closer approximation to the true velocity circle for the region of the sample under inspection. By comparing the measured velocity circles against known velocity circles calculated from elastic constants, a crystallographic orientation can be assigned to each pixel in the map based on cross-correlation fitting methods [186]. The resulting output is an orientation map of the surface, comparable to an inverse pole figure map usually obtained from EBSD. Due to the strong basal texture of white layers in machined titanium and the sensitivity of the SAW velocity to crystal orientation, it is predicted that SRAS could be used to detect anomalous machined surfaces based on velocity and crystal orientation measurements.

To investigate this hypothesis, titanium samples were scanned using the SRAS equipment at the University of Nottingham. A 24 μm SAW wavelength was selected for the scans to maximise the signal from the white layer whilst minimizing noise during the frequency measurement. Smaller wavelengths facilitate inspection of thinner layers, however, there is a trade-off with reduced signal quality as the wavelength approaches the size of the detection beam. The increased noise at low wavelengths significantly affected preliminary measurements at a 16 μm wavelength. A 20 μm lateral step size was chosen to give a faster scan rate whilst providing enough lateral resolution to distinguish macrozones, which are regions of a material with a similar crystallographic orientation. These macrozones arise during the prior forging of $\alpha+\beta$ titanium alloys [288].

To prepare samples for SRAS, 10-15 mm square blocks were sectioned from the square shoulder milled Ti-6Al-4V workpieces and then mounted in Bakelite, before being ground and polished to a mirror finish by removing the smallest amount of material possible. Typically, around 5 μm of material removal was required to obtain the necessary surface finish. Polished samples are required for SRAS the instrumentation used due to the limitations of the photodiode detector [194] which requires a flat surface. Deviations in surface height limited to approximately one micron in order to accurately measure the frequency of the passing SAW. The material removal required to produce a smooth surface means that the white layer thickness on all of the SRAS samples will be lower than the equivalent unpolished machined surface, however, this was an unavoidable step due to current roughness limitations of SRAS [193]. The polishing process may also introduce additional deformation, which could alter the crystallographic texture, however, only ultra-fine grinding (P2500) was used before mirror polishing with colloidal silica suspension. Consequently, the region of affected material is expected to be smaller than the SAW interaction volume. It should be noted that on-going improvements, as highlighted in the literature review, might soon facilitate the inspection of surfaces without this prior preparation step.

Due to the uniformity of the velocity circle for basally orientated HCP crystals, only two scans are required to indicate a basal orientation. Therefore, surfaces were scanned using different SAW propagation directions (0° , 40° , 90° , 130° as highlighted in Figure 7.5 (a)) and pairs orthogonal scans were combined to infer basally orientation material. It should be noted that for a full orientation study, 18 scans at 10° intervals are required to build up high-quality velocity circles and determine other HCP orientations accurately, however, this was not possible for this study due to the finite time available on the SRAS equipment. Due to the symmetry of the velocity circle, a 0° propagation direction scan is equivalent to 180° and 90° is equivalent to 270° , as such scans between 180° and 360° offer no additional information. The SAW frequency was measured at each sampling point during the raster scan of the test piece surface and used to calculate the SAW propagation velocity. The velocity was then compared against the expected velocities for a SAW propagating in titanium ($2700\text{--}3200\text{ ms}^{-1}$). If the velocity was found to lie outside this range, it was removed from the data set as it could only arise from an erroneous measurement. Additionally, an optical image of each surface was obtained concurrently to the SRAS measurement. If the greyscale value of the pixel was beneath a set threshold value, then the equivalent velocity measurement

was removed, as a low optical value typically indicates a scratch or surface imperfection that can influence the measurement, as highlighted in Figure 7.5 (b).

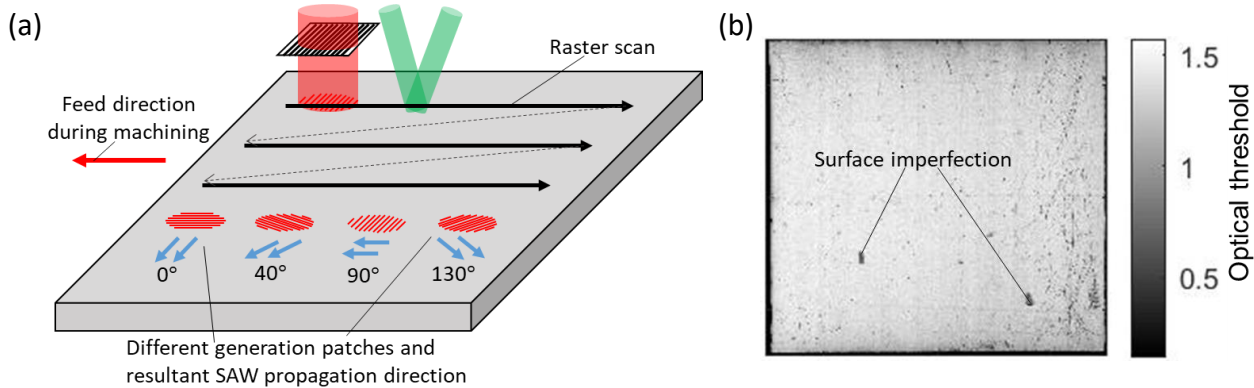


Figure 7.5 – A schematic (a) highlighting the SRAS raster scan over a machined surface and the different propagation directions relative to the machining feed direction and (b) an optical image of the surface under inspection used to remove regions with surface imperfections from the SRAS velocity maps.

An estimate of crystal orientation was obtained by combining the velocity data from scans of the same surface with different SAW propagation directions. This was carried out using the SRAS orientation search algorithm, as described in detail in [186]. In this method, the approximated velocity circle associated with each sampling point across the machined surface is compared to a database of theoretical velocity circles, which describe the directional dependence of the velocity of the dominant SAW mode on a particular plane. Using cross-correlation methods, the theoretical velocity circles for different orientations and the experimentally measured velocity circle are compared and the orientation which gives the best fit is attributed to that particular region of the surface. The greater the number of directions scanned, the greater the resolution of the experimental velocity map. However, due to the uniformity of the velocity circle for a SAW on the HCP basal plane, as highlighted in Figure 4.5 (a), only two scans are required to identify the basal plane. The orientation maps in this study are therefore indicative of only the basal plane, however, this should be sufficient for the identification of white layers in Ti-6Al-4V through orientation measurements.

To investigate the change in SAW propagation behaviour with depth beneath the machined surface, a sample containing a white layer was successively re-ground and polished using P2500 grit paper and a Struers MD Chem polishing pad with colloidal silica suspension and then inspected with SRAS. This process allowed SRAS measurements to be carried at different depths beneath the machined surface, through the white layer region and the distorted material beneath until only the bulk material remained. Material

removal was quantified using a digital micrometre, accurate to 2 μm , to facilitate comparison with the optical micrographs of the associated machined surface.

7.4 A residual stress-sensitive method for white layer detection in ferromagnetic alloys

White layers have been shown to possess extremes of residual stress, with large compressive stresses measured in the majority of the samples created in this study. Barkhausen noise (BN) is known to be sensitive to changes in residual stress in ferromagnetic materials, so the development of a BN methodology for white layer detection is discussed in this section.

The large compressive residual stresses that we measured for the white layers formed in SCMV, in addition to their nano-scale grain structure, should provide additional barriers to magnetic domain wall motion, relative to a surface without a white layer. To investigate the potential of a BN NDT method for white layer detection, a Stresstech Rollscan 350 Barkhausen analyzer, as shown in Figure 7.6 (a), was used to inspect as-machined SCMV surfaces, with no surface modification or sectioning required. This commercially available BN inspection kit can be used with various probe types at alternating magnetic field frequencies between 0-1000 Hz and peak-to-peak voltages of 0-16 V_{pp}. A Stresstech S1-15-32-02 sensor, designed for outside diameter measurements on curved surfaces was selected for the magnetizing yoke and the pick-up coil, due to its suitability for inspection of turned surfaces. The Rollscan system utilises an alternating magnetic field in the magnetizing yoke to generate BN in the test piece, which is then measured by a pick-up coil between the arms of the yoke, as shown in Figure 7.6 (b). The Barkhausen noise is measured by the Rollscan 350 in units of magnetoelastic parameter (mp), an arbitrary unit equivalent to the RMS of the induced voltage in the pick-up coil.

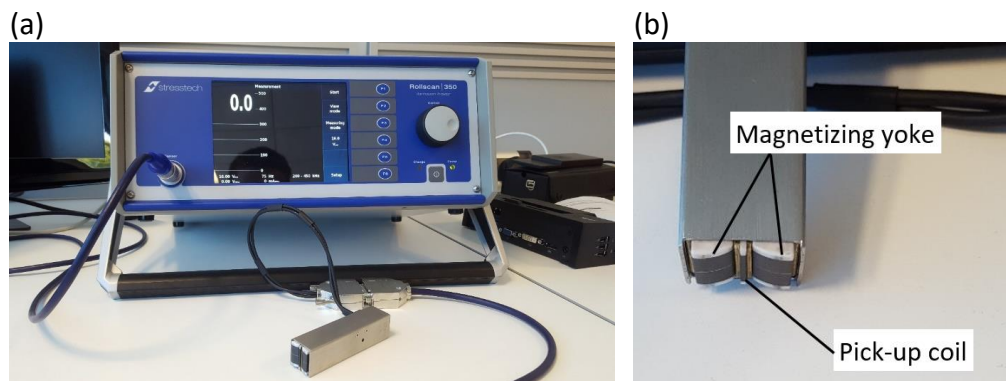


Figure 7.6 – (a) The Rollscan 350 unit and S1-15-32-02 sensor and (b) a close-up of the sensor highlighting the yoke and pick-up coil.

The Barkhausen noise penetration depths for different filter frequencies, calculated using confidential Roll-Royce material property data and Eq. 4.1, are given in Table 7.2. It should be noted that the penetration depth of the BN inspection method is the depth from which the BN signal is detected at a particular filter frequency. The depth to which BN is generated but ultimately filtered out is greater than the penetration depth for BN reaching the detector. Due to the small thickness of a white layer compared to the penetration depth (50-75 μm), even at the maximum input frequency, the highest frequency filter on the Rollscan 350 was chosen for preliminary measurements, to restrict the penetration depth to the smallest possible near-surface region.

Table 7.2 – Penetration depths for the three filter frequencies available on the BN system as well as the estimated high pass filter required to restrict the penetration depth to within the white layer.

Bandpass filter frequency (kHz)	Penetration depth (μm)
10 – 70	340 – 130
70 – 200	130 – 75
200 – 450	75 – 50
12,000	< 10

Initial BN measurements were undertaken at a single magnetizing frequency of 180 Hz and a peak-to-peak voltage of 16 V_{pp} , as this corresponds to the fast but basic measurement capability of the equipment and so is a useful baseline test. Following preliminary basic BN measurements, the Rollscan was used for magnetizing voltage sweep (MVS) and magnetizing frequency sweep (MFS) measurements, which cover the full spectrum of input conditions, rather than an individual point, and allow the extraction of additional BN signal features.

During a MVS the voltage is varied from 0 V_{pp} up to 16 V_{pp} at a constant frequency and the BN is sampled throughout the sweep. The alternating magnetic field used to magnetise the part is proportional to the magnetizing current, however, the voltage measured in the MVS does not increase linearly with the current. Consequently, the magnetizing current was also recorded by the Rollscan during MVS and used to plot a magnetizing current sweep (MCS), since the current-based analysis is more appropriate for relating the results to the underlying magnetic physics. As well as the MCS, the first and second derivatives of the BN during a MCS were also plotted, as shown in Figure 7.7. The key features of the plots are described below.

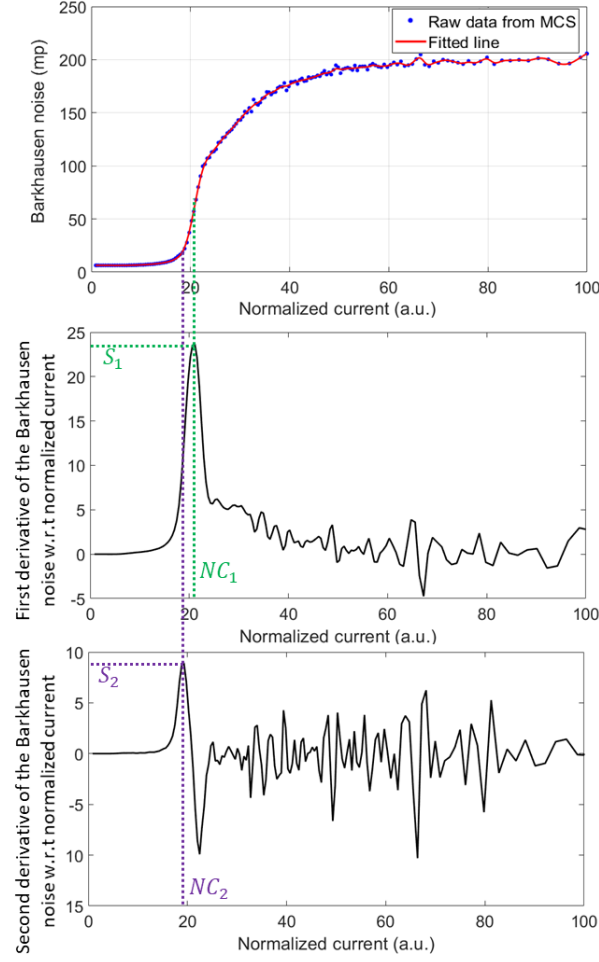


Figure 7.7 – The plots and key features obtained from MCS analysis.

By exporting the raw data to MATLAB, additional features could be determined from post-processing of the data to allow a more in-depth comparison of the BN characteristics of white layers. Key features, as previously outlined by Santa-aho et al. [214], were extracted from the MCS plots, which was smoothed using a moving average filter. The features extracted were the maximum slope (S_1) and the current (NC_1) at which this slope was observed, obtained from the first derivative of the BN with respect to the current (BN'). The maximum second derivative (S_2) and the corresponding current (NC_2) were also calculated.

In the MFS measurements, the magnetizing frequency was varied from 10 Hz up to 1000 Hz for a constant V_{pp} and the BN was measured at each frequency. The key features extracted from the MFS plots were the maximum BN noise (BN_{max}) and the frequency at which this maximum was recorded (f_{max}), as shown in Figure 7.8. The key features from both MFS and MCS measurements are summarised in Table 7.3.

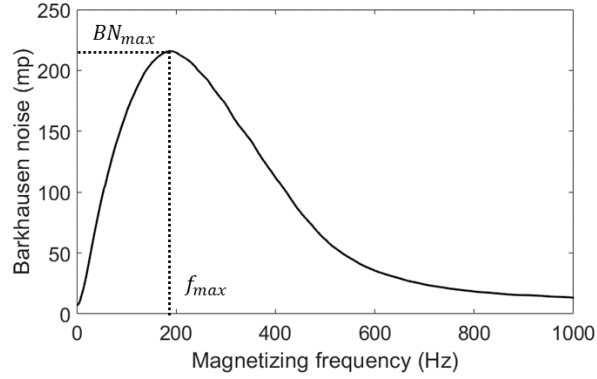


Figure 7.8 – The plot and key features obtained from MFS analysis.

Table 7.3 – The key features from BN inspection of SCMV machined surfaces

Parameter	Definition
BN_{max}	Max BN during a MFS
f_{max}	Frequency at which BN_{max} is observed
S_1	Maximum slope of the MCS
NC_1	Current at which S_1 is observed
S_2	Maximum second derivative of the MCS plot
NC_2	Current at which S_2 is observed

To relate the results from BN inspection to the underlying mechanical and topological properties of the machined surfaces, the hardness and residual stress results for SCMV, presented in Figure 6.17 and Figure 6.20, were weighted according to their depth beneath the surface, within the BN penetration depth. This weighting was necessary due to the exponential decay of the strength of eddy currents with depth in BN testing [167] and the comparatively large penetration depth of the BN method relative to the other NDT methods investigated. The weighting was taken as 1 at the surface and $1/e$ at the skin depth of $50\ \mu\text{m}$ and it facilitated an approximation of the enhanced sensitivity of BN to the near-surface region.

8 Non-destructive methods for detecting machining induced white layers – results and discussion

This chapter presents the results from the non-destructive evaluation of white layers in machined surfaces, with reference to the physical properties that have been determined through destructive characterisation. It is shown that the grain-size sensitive XRD- Δ PB method can be used to detect machining-induced white layers in all aeroengine alloys. Texture-sensitive XRD- Δ IR and SRAS methods are also shown to facilitate the detection of Ti-6Al-4V surfaces containing a white layer and for the case of SRAS, surfaces that contain a swept grain region. The XRD- Δ IR is also applicable to nickel alloys but not steel surfaces. For ferromagnetic materials, Barkhausen noise is shown to be sensitive to the residual stress state, allowing ferromagnetic surfaces with thick white layers to be detected. At the end of the chapter, the proposed NDT methods are evaluated against the current destructive process.

8.1 A grain size sensitive method for white layer detection (XRD- Δ PB)

In this section, the results of XRD- Δ PB measurements are presented before a discussion of the results in the context of the physical properties of white layers and an assessment of the method.

8.1.1 Initial peak selection

The amount by which XRD peaks are broadened for a particular crystallographic plane is not linearly related to the diffraction angle in textured materials due to anisotropic peak broadening, as explained previously in section 7.1. Thus, to establish the peak most sensitive to broadening effects in each alloy under investigation, the peak breadths for different crystallographic planes were measured in a small set of machined samples with and without white layers.

8.1.1.1 Ti-6Al-4V

Results from measurements on Ti-6Al-4V samples using both FWHM and IB analysis are shown in Figure 8.1 (a) and (b) respectively. It can be seen that, in general, peak breadth increases for higher diffraction angle planes in all samples using both peak breadth measures, as would be expected considering the relationships for strain and crystallite size broadening outlined previously. Peak breadth was also increased in samples with a white layer, relative to a sample with no white layer, for all peaks except the {0002} and {0004} peaks. The different behaviour of these peaks is discussed in section 8.1.6. Fewer peaks are shown

for the IB analysis due to overlap in the tails of some diffraction peaks which prevented measurement of breadth using this method. A number of peaks allow samples with a white layer to be identified through an increase in peak breadth, however, it can be seen from the diffraction pattern in Figure 8.1 (c) that the relative intensity of each peak is different, as such the time taken to capture the data required for peak breadth measurement is dependent on peak selection.

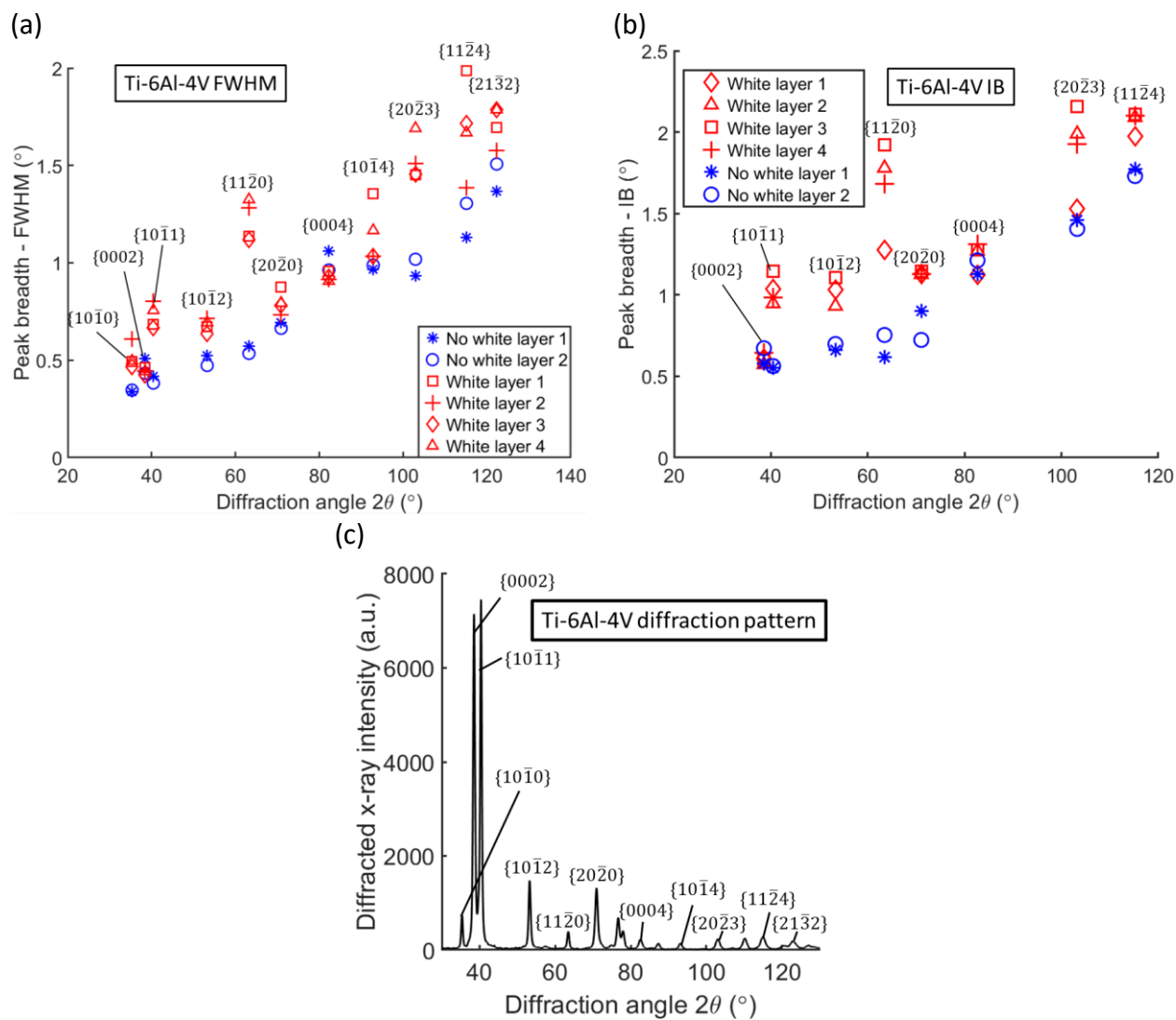


Figure 8.1 – Plots highlighting the dependence of (a) FWHM and (b) integral peak breadth on diffraction angle and thus crystallographic plane for Ti-6Al-4V and (c) a diffraction pattern for Ti-6Al-4V.

To highlight the peak most sensitive to the presence of a white layer, the percentage difference in peak breadth between surfaces with a white layer and those without in both peak breadth measurement methods is reported in Table 8.1 for the most suitable peaks.

Table 8.1 – The percentage difference between average peak breadths (PB) for surfaces with (PB) and without (PB) white layers for several crystallographic planes in Ti-6Al-4V. The error the PB difference measurement is the standard error of the mean.

Peak	2 θ	FWHM PB/PB difference (%)	IB PB/PB difference (%)
{10 $\bar{1}$ 1}	40.5	82 ($\pm$ 9)	84 ($\pm$ 7)
{11 $\bar{2}$ 0}	63.2	120 ($\pm$ 18)	143 ($\pm$ 25)
{20 $\bar{2}$ 3}	103.0	56 ($\pm$ 17)	33 ($\pm$ 8)

For FWHM analysis, the greatest distinction between the sample types is provided by the {11 $\bar{2}$ 0} plane, but this is a higher diffraction angle peak with a much lower intensity than the {10 $\bar{1}$ 1} peak, as shown in Figure 8.1 (c), which provided the next largest percentage separation between samples types. A peak that produces fewer diffracted x-rays requires a longer measurement time to acquire the data needed for XRD- Δ PB measurements. Similarly the {20 $\bar{2}$ 3}, which is the third most sensitive peak, is a high-angle peak, meaning intensity is lower compared to the low-angle {10 $\bar{1}$ 1} peak. Calculation of the integral breadth increases the sensitivity of the {11 $\bar{2}$ 0} peak for the detection of white layers, however, it has minimal effect on {10 $\bar{1}$ 1} peak. The calculation of the integral peak breadth increases the time required for inspection using the XRD peak broadening method from under 5 minutes per sample to approximately 15 minutes, due to the additional post-processing steps involved. Due to the low change to {10 $\bar{1}$ 1} peak breadth between both methods and the increase in post-processing time for IB measurement, a decision was made to take the {10 $\bar{1}$ 1} FWHM peak breadth forward for further investigation with the full set of Ti-6Al-4V samples. However, it should be emphasized that all planes apart from {0002} and {0004} could be used for white layer detection. Peak choice may be dependent on the diffraction angle range of the equipment used as the Proto iXRD, for example, used in this study for residual stress measurements, is limited to diffraction angles between 123°-171°. Based on the increase in post-processing time for the IB method during measurements in Ti-6Al-4V, only the FWHM method was taken forward for peak selection in the other aeroengine alloys.

8.1.1.2 IN-718

The FWHM peak comparison methodology was also applied in the selection of the most appropriate peak for IN-718. The peak breadth for different crystallographic planes is shown in Figure 8.2 (a). It can be seen that the {200} and {311} peaks facilitate a large separation between samples white layers and those without, with reduced separation for the other three peaks. Evidence of anisotropic peak broadening can be seen from Figure 8.2 (a) due to the non-linear variation in peak breadth with diffraction angle. The diffraction pattern in Figure 8.2 (b) shows that the {311} peak is a much lower intensity peak and therefore to obtain the same level of diffracted intensity as the {200} peak, the measurement time would have to be several times longer. The percentage peak breadth difference between surfaces with white layers and those without is shown in Table 8.2 for the two peaks that facilitate white layer detection with the most confidence. The larger separation when using the {200} peak in addition to the lower intensity of the {311} peak means that the {200} plane is most suitable and it was selected as the diffraction peak of interest in this alloy to take forward for measurement with the full sample set.

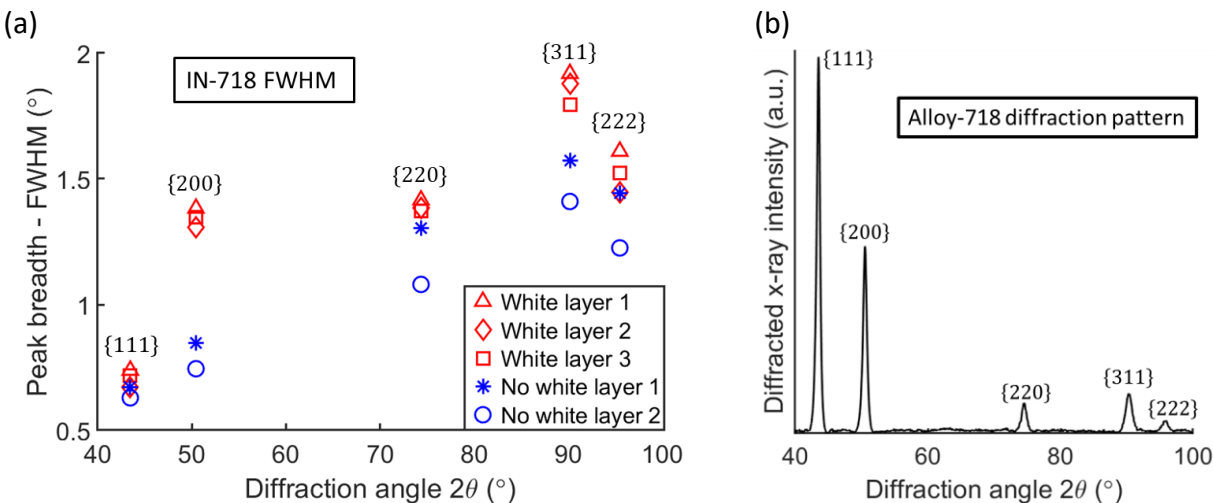


Figure 8.2 - A plot (a) highlighting the dependence of FWHM peak breadth on the crystallographic plane for IN-718 and (b) a typical XRD pattern for this superalloy.

Table 8.2 – The percentage difference between average FWHM PB in surfaces with (PB) and without (PB) white layers for different crystallographic planes in IN-718.

Peak	2θ	PB/PB difference (%)
{200}	50.4	69 (±8)
{311}	90.2	25 (±5)

8.1.1.3 SCMV

The most appropriate peak for detecting a white layer in SCMV is the $\{211\}$ or $\{220\}$ peak, as shown in the plot in Figure 8.3 (a). It can be seen that the $\{200\}$ peak allows separation between machined surfaces with and without white layers, but not the as-received material. This is primarily thought to be a consequence of a low peak intensity, as shown in Figure 8.3 (b), which results in a noisy diffraction peak and consequently a low-quality FWHM measurement. It can be seen from Figure 8.3 (b), that all peaks except the $\{110\}$ peak are low intensity, as such a longer measurement time is unavoidable. The percentage differences between sample types for the $\{211\}$ or $\{220\}$ peaks are given in Table 8.2. In this study, the $\{211\}$ peak was selected for measurement of the full sample set, despite the reduced sensitivity compared to the $\{200\}$ peak, due to the reduced measurement time facilitated by its greater intensity. However, these results show that either peak could have been selected. Similar to the other aeroengine alloys, peak broadening is not linear across different diffraction peaks, due to anisotropic peak broadening.

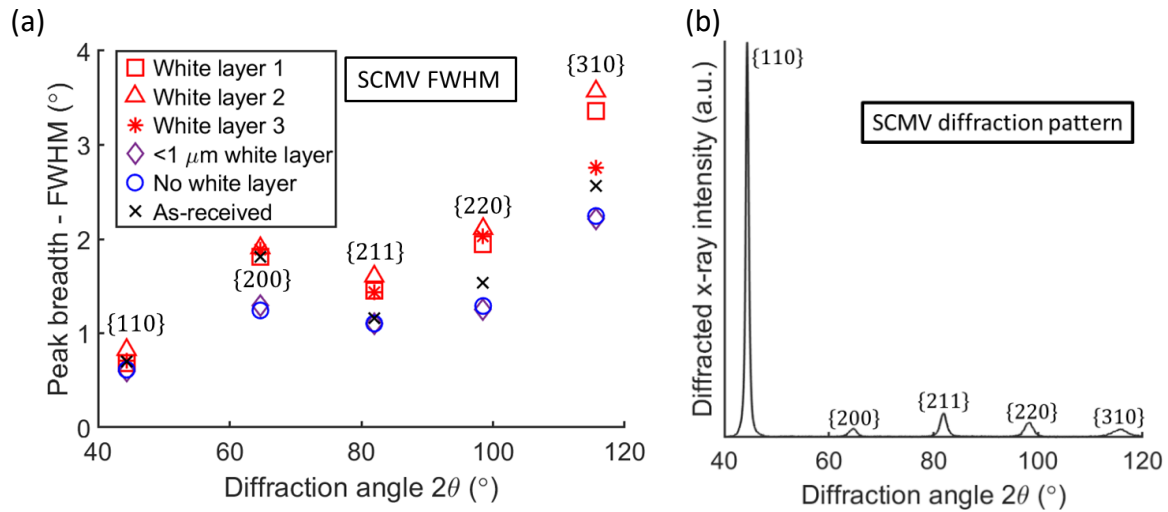


Figure 8.3 - A plot (a) highlighting the dependence of peak breadth on the crystallographic plane for SCMV and a diffraction pattern (b) for SCMV showing the relative intensities of each peak.

Table 8.3 – The percentage difference between average FWHM PB for different crystallographic planes in SCMV surfaces with and without white layers.

Peak	2θ	PB/PB difference (%)
$\{211\}$	82.0	32 (± 5)
$\{220\}$	98.5	44 (± 9)

8.1.2 XRD- Δ PB measurements to detect white layers

8.1.2.1 Ti-6Al-4V {10 $\bar{1}$ 1}

Reduced Ti-6Al-4V XRD patterns, covering only the peaks of interest for peak breadth and texture analysis, are shown in Figure 8.4 (a) for as-machined and as-received samples. It can be seen that the {10 $\bar{1}$ 1} peak is broader in surfaces with a white layer and has a lower intensity relative to the {0002} peak. This intensity change is explored in section 8.2.1.1. The {10 $\bar{1}$ 1} peak breadth for these samples is plotted in Figure 8.4 (b), where it can be seen that the XRD peak broadening method facilitates the detection of white layers in all Ti-6Al-4V surfaces examined. It is apparent that a simple upper threshold value, e.g. 0.68° for this experimental set-up, would allow surfaces with a white layer to be identified and a lower threshold value, e.g. 0.53° would be typical of as-received material or machined surfaces with no white layer. Any measurement above the lower threshold value could be considered indicative of an anomalous surface.

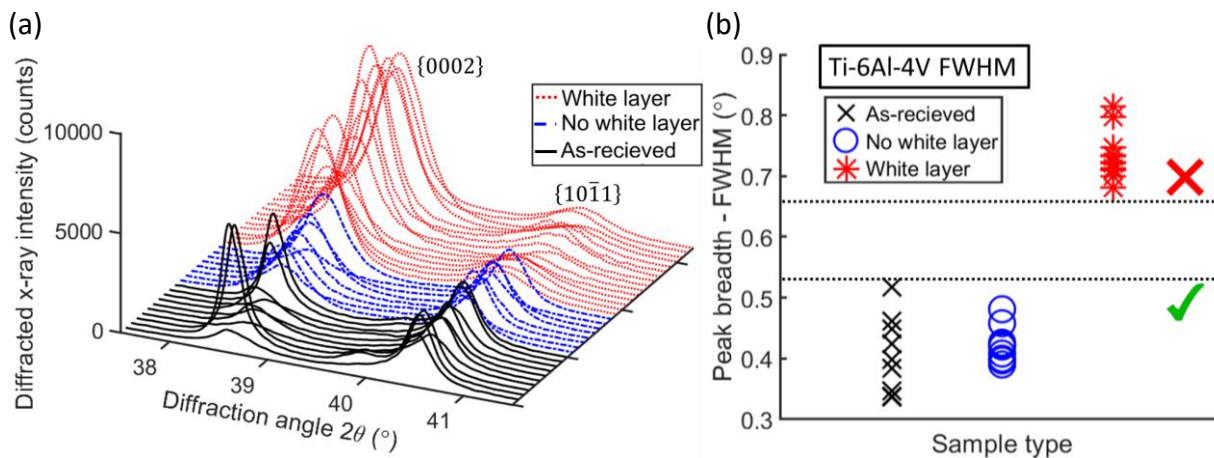


Figure 8.4 – XRD patterns (a) for 33 Ti-6Al-4V surfaces focusing on the peaks of interest for XRD- Δ PB and XRD- Δ IR measurements and a plot (b) of the peak breadth against sample type for all Ti-6Al-4V surfaces inspected. The symbols on the right indicate samples that would fail CME inspection (red cross) and samples that would pass (green tick). In the region of uncertainty between the two thresholds, a surface would fail inspection as it cannot be shown to be a good surface.

The XRD measurement itself is highly accurate and repeatable. When samples were measured twice without remounting, peak breadth was measured to vary by less than 1% between repeats, as shown in Figure 8.5 (a). However, XRD patterns are sensitive to errors in the sample mounting e.g. sample height and the sample tilt. To check for these effects the FWHM for each sample was measured twice, with the sample remounted before the second measurement, as shown in Figure 8.5 (b). This repeat test also helps capture errors on sample labelling as the XPert³ diffractometer allows up to 60 samples to be measured in series, meaning there is a potential for sample mix-up. If a discrepancy greater than 0.047° was recorded between repeat measurements, sample-mounting errors were deemed to have occurred and the surfaces

were measured a further two times. The results were then accepted if the 0.047° criterion was met. This criterion was chosen as it represents the 3σ confidence level for repeat measurements on the same sample with remounting.

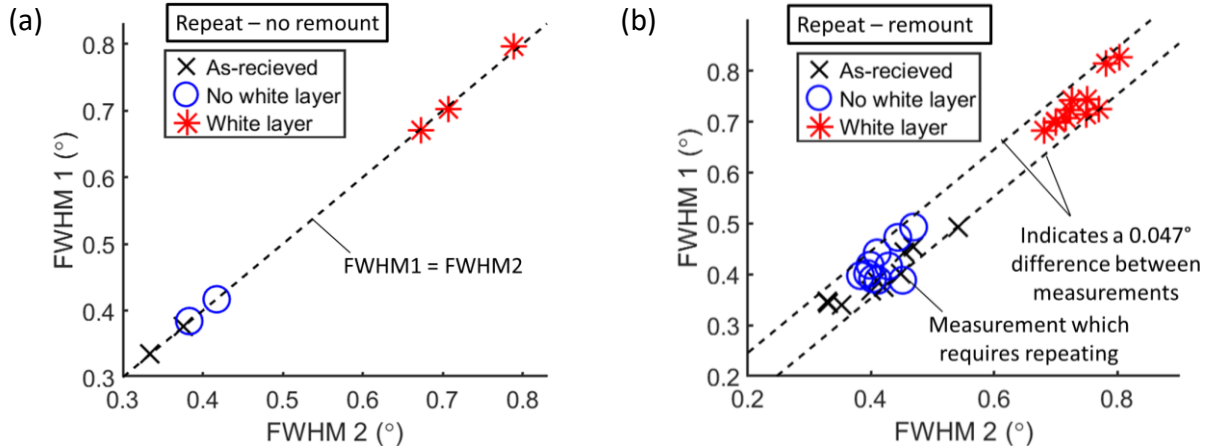


Figure 8.5 – Plots of (a) the results from XRD- Δ PB repeat measurements in which the sample was not remounted between measurements and (b) repeated XRD- Δ PB measurements in which the sample was remounted.

The dependence of peak breadth on the white layer thickness is shown in Figure 8.6. No relationship appears to exist between these two variables, however, the white layer thickness in all samples is greater than the depth of penetration of x-rays ($5\text{ }\mu\text{m}$ for 90% attenuation). Therefore, the x-rays do not interact significantly with the non-white layer material in any of these samples and so the absence of a relationship between peak breadth and the white layer thickness above $5\text{ }\mu\text{m}$ is unsurprising.

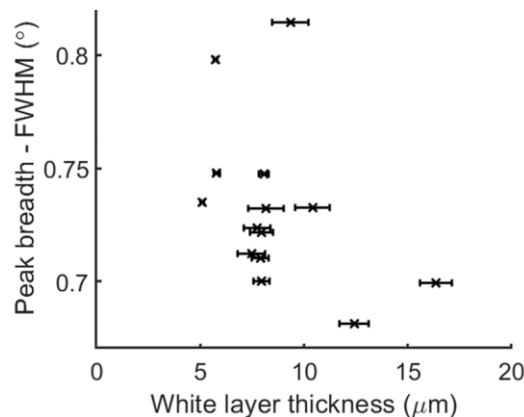


Figure 8.6 - Peak breadth plotted against white layer thickness for Ti-6Al-4V machined surfaces. Error bars represent the standard error of the mean.

8.1.2.2 IN-718 {200} and SCMV {211}

Results from XRD- Δ PB measurements in IN-718 and SCMV samples are shown in Figure 8.7 (a) and (b), respectively. In IN-718, the XRD peak broadening method again allows surfaces containing a white layer

to be distinguished from those without a white layer with a good level of confidence. There is a clear increase in peak breadth of nearly 0.3° in samples with a white layer. In the IN-718 samples inspected, a measurement exceeding a peak breadth of 0.8° is indicative of an anomalous surface. For SCMV surfaces, there is a 0.1° increase in peak breadth between white layer samples ($>1\text{ }\mu\text{m}$) and all other samples. For the samples with a suspected white layer thinner than $1\text{ }\mu\text{m}$, the thickness cannot be assessed with confidence due to the edge effect encountered during SEM imaging.

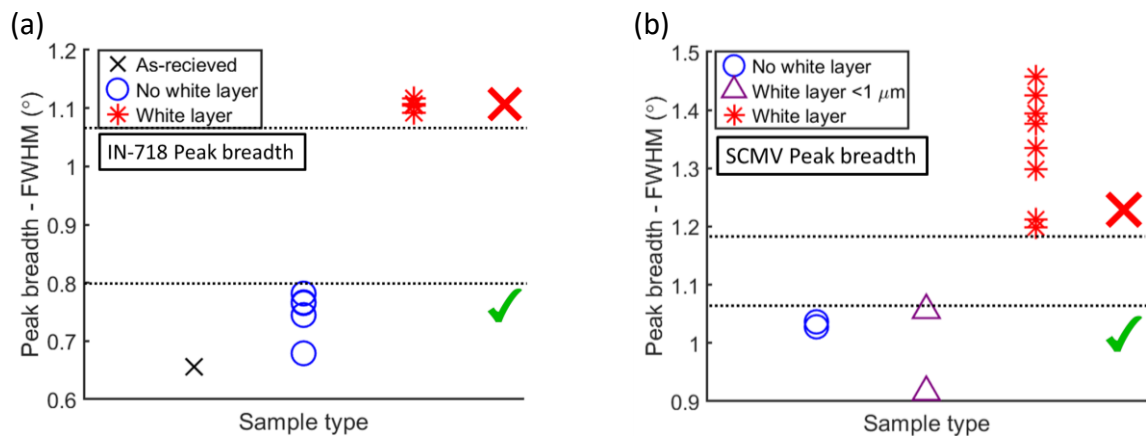


Figure 8.7 – Peak breadth plotted against sample type for (a) IN-718 and (b) SCMV surfaces inspected.

8.1.2.3 Validation tests

To validate the proposed XRD-ΔPB method for white layer detection, two small scale studies were carried out. In the first, measurements were carried out using the Xpert³ diffractometer on SCMV machined surfaces generated externally to this project. The second validation study aimed to test the transferability of the technique to other XRD equipment that negated the need for sectioning to fit the sample within the confines of the diffractometer.

The SCMV validation samples had been machined with new inserts rather than artificially worn inserts and destructive microscopy had been carried out according to the Roll-Royce CME 5043 process. The white layers in these samples were $2.5\text{ }\mu\text{m} - 4\text{ }\mu\text{m}$ in thickness and represent white layer generation without artificially worn inserts. The XRD-ΔPB method is again capable of identifying those surfaces that contain a white layer, with a clear distinction from those machined surfaces with no white layer, as shown in Figure 8.8.

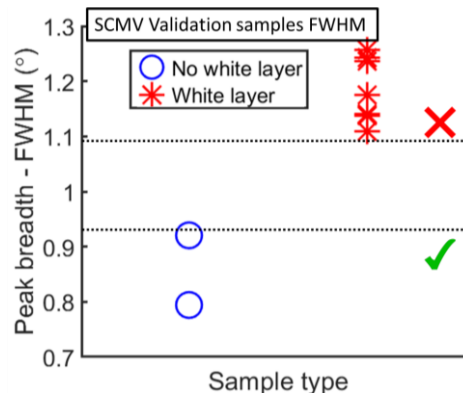


Figure 8.8 – Peak breadth for SCMV validation samples with and without a white layer.

In the second validation study, XRD- Δ PB measurements were carried out on a PANalytical Emyrean diffractometer, using a molybdenum x-ray tube. This machine facilitated the inspection of an un-sectioned workpiece as shown in Figure 8.9 (a). Similar XRD- Δ PB measurements were also undertaken with the Proto iXRD, as used previously when measuring residual stress profiles. The iXRD equipment is portable, as shown in Figure 8.9 (b), and can be used to test a wide range of shapes and sizes but is limited to high-angle diffraction peaks. The percentage difference between peak breadth for surfaces with and without white layers for the Emyrean and iXRD equipment are shown in Table 8.4. It can be seen that both diffractometers facilitate the distinction of surfaces with a white layer. The Emyrean offers greater resolution and this is unsurprising as the desirable $\{10\bar{1}1\}$ peak could be used. The reduction in sensitivity of the Emyrean compared to the Xpert³ can be attributed to the increased penetration depth when using molybdenum x-rays, which means the x-rays interact with non-white layer material.

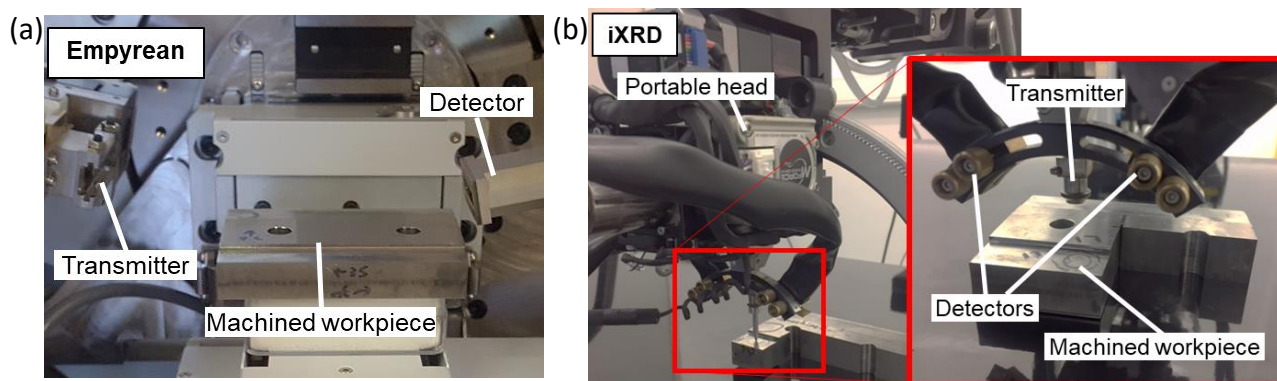


Figure 8.9 – Diagrams of (a) the Emyrean diffractometer and (b) the Proto iXRD portable diffractometer

Table 8.4 – The percentage difference between average FWHM PB for equipment that facilitates truly non-destructive detection of white layers and the value for the Xpert³ equipment for comparison.

Surface	PB/PB difference (%)
Empyrean	64
iXRD	17
Xpert ³	82

Although it has been shown that the XRD- Δ PB method can be used to non-destructively detect machining induced white layers in aeroengine alloys, its reliability when measuring a white layer thinner than the XRD penetration depth is unclear. To determine the white layer thickness limit at which this technique could be reliably implemented, the electropolishing process was applied to as-machined white layer samples to alter the layer thickness. Additionally, to evaluate how the main factors that affect peak broadening, crystallite size and inhomogeneous lattice strain, are influencing the peak breadth of white layers, further experiments using heat treatments and W-H analysis were undertaken. In both of these additional studies, only Ti-6Al-4V alloys were tested.

8.1.3 The effect of white layer thickness on peak broadening

Electropolishing was used to remove material from surfaces containing a white layer to produce layers with various thicknesses, which could then be inspected using the XRD- Δ PB method. From these measurements, the influence of white the layer thickness on the effectiveness of the method and the resolution limitations could be established. The dependence of peak breadth on white layer thickness for these EP samples is shown in Figure 8.10. When the white layer thickness is reduced through electropolishing, the x-ray interaction volume during subsequent inspection contains more of the swept grain region beneath the white layers, than for non-electropolished surfaces with thicker white layers. Consequently, the peak breadth was measured to be lower for thinner white layers due to the influence of non-white layer material. It is therefore more difficult to detect a thinner white layer as there is a smaller difference between the measured peak breadth for a thin white layer and that associated with a surface with no white layer. It should be noted that these results suggest that the white layer thickness cannot be accurately predicted, across all machined surfaces, using the XRD- Δ PB method. This is evident from Figure

8.10 where a particular peak breadth can be associated with a range of white layer thicknesses, depending on the sample under inspection.

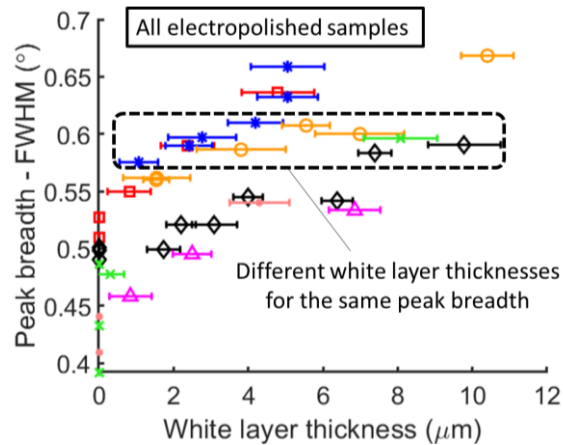


Figure 8.10 – A plot showing the dependence of peak breadth on the white layer thickness for surfaces in which the white layer thickness was reduced through electropolishing. Each colour corresponds to a particular machined surface. Error bars represent the standard error of the mean.

8.1.4 The effect of crystallite size and inhomogeneous lattice strain on overall peak broadening

XRD peak breadth was measured on machined surfaces with white layers before and after heat treatments at a range of temperatures, 250-700°C, and the microstructure was imaged post-heat treatment. The change in peak breadth, following heat treatment, is shown in Figure 8.11 and it can be seen that as the heat treatment temperature increases, the peak breadth decreases and the grain structure becomes more resolvable, with larger grains and more clearly defined grain boundaries visible in the SEM micrographs. It is not possible to quantify the grain size from the low-temperature heat treatment images as the grain boundaries cannot be discerned, but it is apparent that grain growth begins below 350°C as grain structure appears more resolvable in the associated image for this heat-treatment than for a lower temperature. Note that the spherical particles visible in the SEM images in Figure 8.11 are surface contaminants left over from the sample preparation process rather than any microstructural feature.

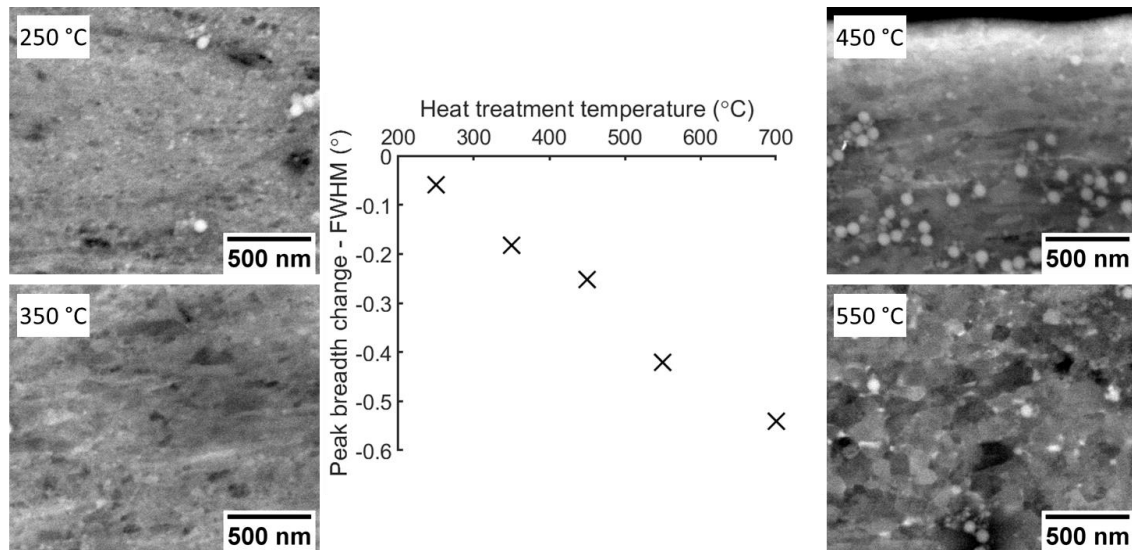


Figure 8.11 – The magnitude of the peak breadth decrease following the heat treatment of white layer samples and corresponding cross-sectional FEG-SEM images of these surfaces.

In addition to heat treatment of a surface with a white layer, an as-machined and an as-received sample without white layers were also heat treated at 700°C for comparison. Table 8.5 outlines the grain size before and after heat treatment and the theoretical and measured peak broadening. A reduction in the peak breadth for the as-received material and a machined surface with no white layer following heat treatment at 700°C, as shown in Table 8.5, reveals that peaks in these non-white layer surfaces are also subject to strain broadening effects. Analysis of the grain size before and after heat treatment revealed a small amount of grain growth in these surfaces, as shown in Figure 8.12. Using this information, theoretical peak breadth changes due to grain size effects could be calculated using the Scherrer equation (Eq. 4.7). It can be seen from Table 8.5 that the theoretical peak breadth changes due to grain size changes cannot account for the peak breadth changes in non-white layer samples following heat treatment and therefore, strain broadening must be occurring.

Table 8.5 – Peak breadth and grain size changes following heat treatment and theoretical PB due to grain size effects for as-received material, a machined surface with no white layer and a machined surface with a white layer.

Surface	Prior to heat treatment		Post heat treatment		Difference	
	Grain size (μm)	Theoretical PB (°)	Grain size (μm)	Theoretical PB (°)	PB measured (°)	PB theoretical (°)
As-received	11.1	7.6×10^{-3}	11.4	7.4×10^{-3}	-0.16	2×10^{-4}
No white layer	10.6	8.0×10^{-3}	13.1	6.5×10^{-3}	-0.13	1.5×10^{-3}
White layer	0.015 < WL < 0.1	0.54 > PB > 0.085	1.2	7.0×10^{-3}	-0.54	Up to 0.54

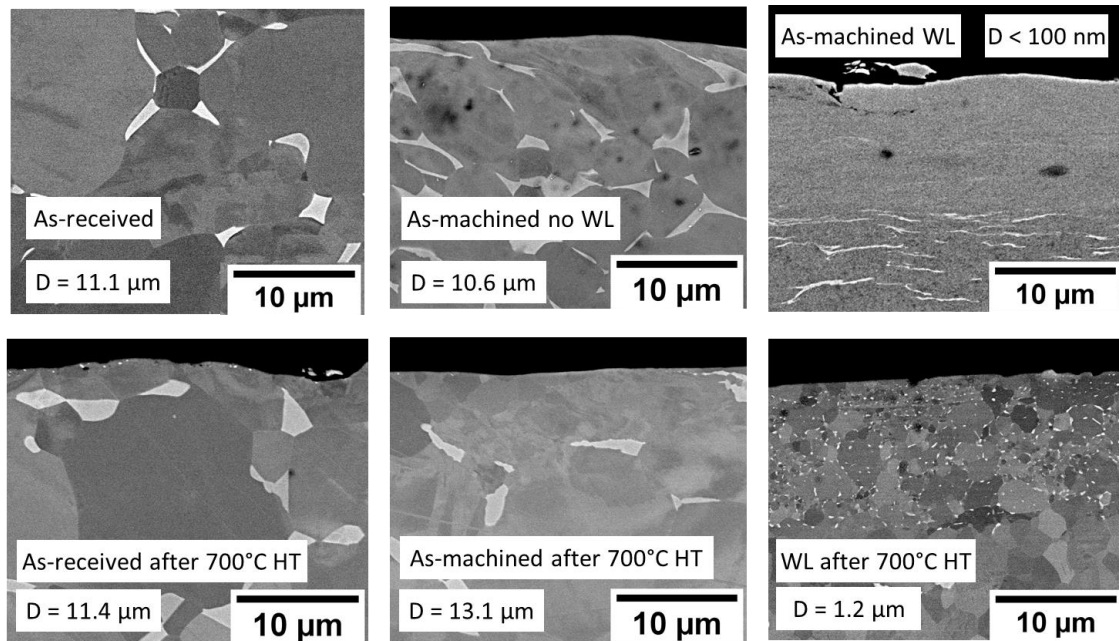


Figure 8.12 – Cross-sectional SEM images of as-machined and as-received surfaces before and after heat treatment.

To quantitatively investigate the relative effects of crystallite size and inhomogeneous lattice strain broadening on overall peak broadening, the W-H and modified W-H methods were used to examine a Ti-6Al-4V sample containing a white layer. The results from analysis using the basic W-H method, the anisotropic strain broadening method and the uniform energy density method are shown in Figure 8.13 (a), (b) and (c), respectively. Each point on the plots corresponds to a particular crystallographic plane. As highlighted previously, the y-intercept and gradient of these plots can be related to the crystallite size and lattice strain broadening, respectively. However, the data does not properly fit the linear relationship due to the considerable scatter of the points and as such it would not be appropriate to use these plots to quantify strain lattice and crystallite size broadening. Despite this, it can be seen that qualitatively, the plots for all methods have a positive gradient and this is indicative that the presence of inhomogeneous lattice strain is contributing to peak broadening alongside the crystallite size effects arising from the ultrafine grains known to be present in the white layer.

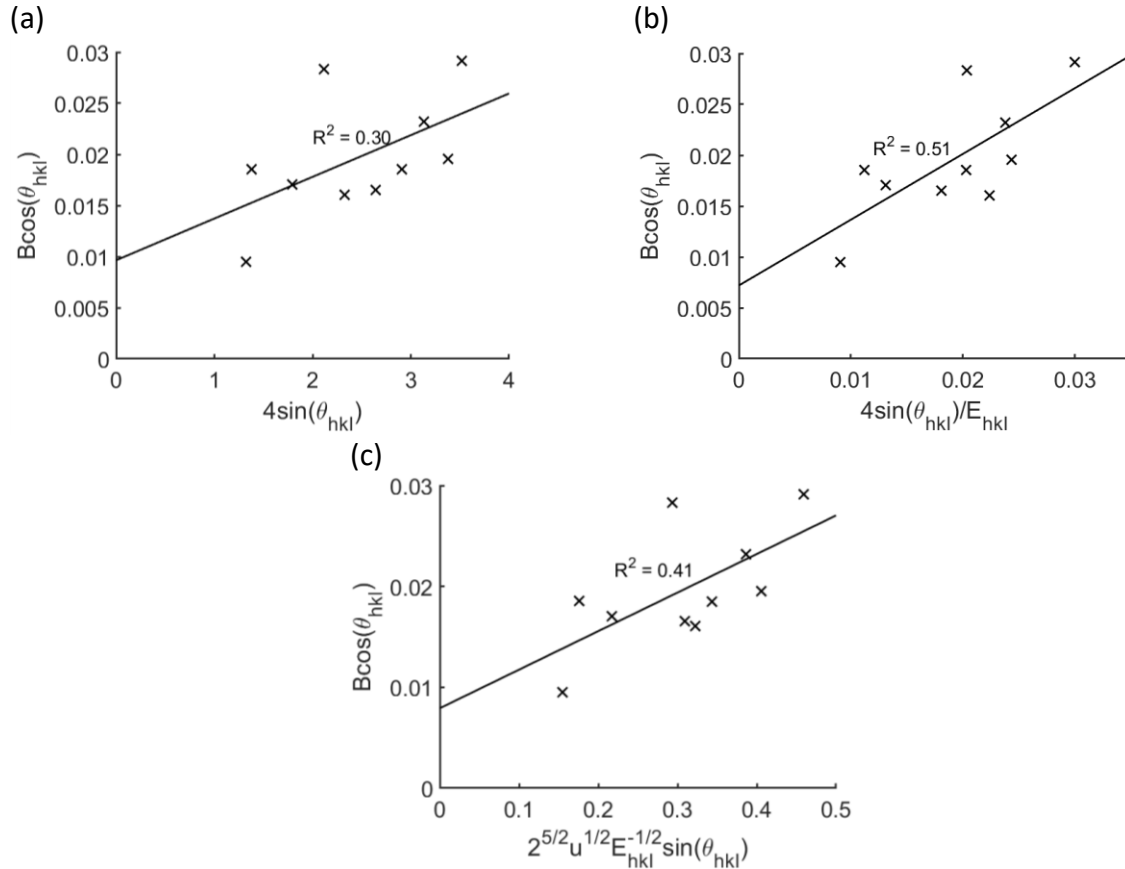


Figure 8.13 – Plots from (a) basic W-H, (b) the anisotropic strain model and (c) the uniform energy density model analysis of a surface with a white layer. Each point corresponds to a different crystallographic plane and in an ideal material without anisotropic strain broadening there would be a linear fit in the plots.

The effect of strain broadening on peak breadth measurements can also be seen by re-examining the peak breadth results from the proto iXRD residual stress profiling tests, reported in section 6.1.5, and shown again in Figure 8.14. It can be seen that the peak broadening is greater in samples that contained a white layer, even beyond the depth of the white layer itself. Outside of the white layer, the grain size increases with depth through the swept grain region up to the bulk grain size, so a decrease in peak breadth outside of the white layer is to be expected. However, the high peak breadth at depths beyond 100 μm for a surface that contains a white layer cannot be attributed to grain size effects. As such these results indicate that, for the high-angle diffraction peak used for the iXRD measurements, strain broadening is the most significant contributor to overall peak broadening. Additionally, it is apparent that the effects of machining on the crystal lattice, extend beyond the region depth to which deformation would be apparent in a cross-sectional micrograph.

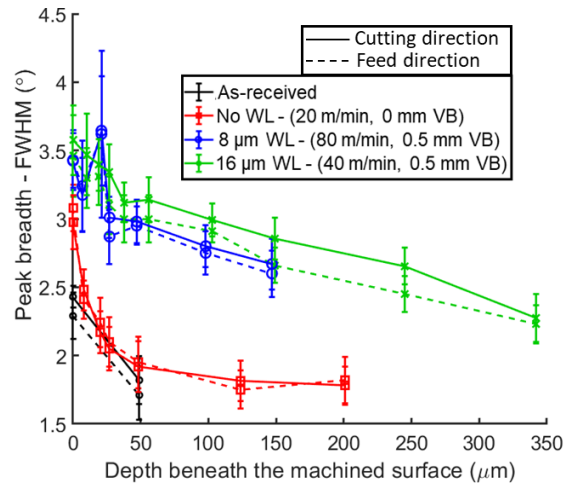


Figure 8.14 – Ti-6Al-4V peak broadening depth profiles beneath the machined surface showing the high peak breadth measured even outside of the white layer in samples machined with worn inserts.

8.1.5 Detection of swept grain using XRD peak broadening

The results from the small-scale study into the applicability of the XRD- Δ PB method for swept grain detection in Udimet 720 samples from industry are shown in Figure 8.15. It can be seen that the peak breadth increases with the maximum thickness of the swept grain layer in these superalloy samples, indicating that the XRD- Δ PB may be sensitive to this anomalous SI feature.

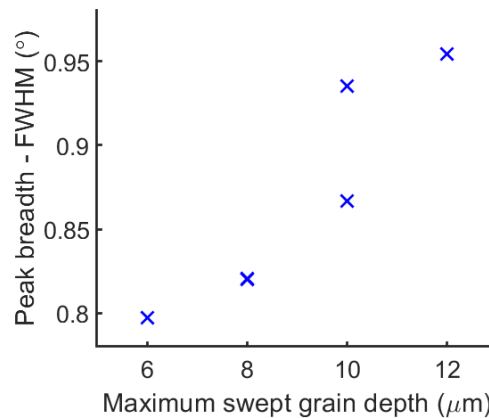


Figure 8.15 – Peak breadth plotted against maximum swept grain depth measured during a SI assessment.

8.1.6 Discussion and assessment of the grain-size sensitive XRD- Δ PB method for white layer detection

This work has significantly expanded on previous characterisation work [228], which used XRD peak broadening to estimate grain size in a white layer in rail steel. In previous work [228], XRD peak breadth measurement was only used as an analysis tool for characterizing the grain size in the white layer in a rail track, alongside other destructive techniques such as cross-sectional microscopy. Whereas, for the XRD- Δ PB research, it has been shown that XRD peak broadening can be used to facilitate the detection of machining induced white layers regardless of the metallic alloy under investigation when the measured

peak breadth exceeds a threshold value. The results from validation tests indicate that the XRD- Δ PB method can be used to detect thin white layers formed without the use of artificially worn inserts. These surfaces are more representative of the type of white layers that may be produced unintentionally during aeroengine manufacturing. In addition, measurements using alternative XRD equipment, which enabled machined surface measurement without destructive sectioning, show that the XRD- Δ PB method for white layer detection is transferrable between different equipment and sources of x-ray radiation and can be considered truly non-destructive.

Repeat XRD- Δ PB measurements, as shown in Figure 8.5, highlighted the sensitivity of the white layer detection method to sample mounting discrepancies whilst also indicating that in the absence of these effects, the technique is highly repeatable. The technique could also be affected by sample contamination effects such as oxide formation on the surface of metallic components. It is predicted that oxide formation would prevent the inspection of white layers using XRD if the oxide was thicker than the inspection depth of the x-rays. However, the location of the peaks corresponding to the oxide layer would occur at a slightly different diffraction angle to the base alloy peaks in the base alloy, so if the oxide was thin, a white layer could still be detectable as the peak of interest would still be measurable.

The lack of a clear relationship between the white layer thickness and the resulting XRD peak breadth when the white layer is thicker than the x-ray inspection depth is to be expected. In these situations, the extra white layer material beneath the x-ray inspection depth does not interact with the x-rays, so it cannot influence the XRD- Δ PB measurement. This result can be used to infer that the grain size and lattice strain in a white layer are independent of the thickness of the white layer, otherwise, a relationship between thickness and peak breadth would be expected. When the white layer is thinner than the inspection depth, a relationship was identified between peak breadth and layer thickness. However, there was noticeable variability in the white layer thickness that could be associated with a particular peak breadth in the EP samples and this can be attributed due to differences in crystallite size and lattice strain between samples machined under different cutting conditions, which are retained as the white layer is removed. It is evident that, while the presence of a white layer can be associated with an increase in peak breadth, the relative strength of the broadening effects in each particular white layer is different.

The decrease in peak breadth on EP surfaces, compared to non-electropolished surfaces, for white layers thicker than the x-ray interaction depth, can be attributed to the redistribution of micro-strains in the lattice following material removal when electropolishing. The redistribution occurs because the distance to the free surface from each point in the lattice is decreased, as material is removed, and there is therefore a degree of relaxation in the local strains. This observation can be used to infer that EP white layers are not fully representative of a machined surface with the same thickness of white layer material, however they serve to illustrate a general decrease in peak breadth for thinner white layers.

Results from inspection of as-machined surfaces showed that a large number of peaks are suitable for use in the XRD- Δ PB method due to the increase in peak breadth associated with a white layer, but the peaks vary in effectiveness due to anisotropic strain broadening and texture-effects. In Ti-6Al-4V the {0002} and {0004} peaks were found to show the reverse relationship for peak breadth when a white layer was present. This could be attributed to the strong crystallographic texture of the white layer in Ti-6Al-4V wherein, despite the small grain size, there are a large number of grains with a basal orientation. Consequently, the microstructure may appear, from the perspective of x-ray interaction, to be composed of comparatively large basal grains thus reducing the peak broadening. Additionally, anisotropic peak broadening and directional dependence of dislocation lattice strain, as outlined previously, could also contribute here and cause the non-linearity of the peak broadening with diffraction angle. It should be noted that the {0004} peak is simply the second-order diffraction peak for the {0002} plane and, as such, contains no additional information.

Results from heat treatments and W-H analysis into the effect of crystallite size and lattice strain effects on peak broadening indicate that both mechanisms contribute to the overall peak breadth measured during XRD- Δ PB inspection. During the heat treatment of as-machined samples, it could be seen that grain growth in the white layer in Ti-6Al-4V begins at temperatures lower than 350 °C. If the white is considered analogous to a nanocrystalline layer produced from SPD during high pressure torsion [225], in which grain growth has been reported as beginning above 300 °C, then it can be assumed that the initial decrease in peak broadening due to heat treatment at 250 °C is due solely to lattice strain relief. At heat treatment temperatures above 350 °C, both crystallite size and lattice strain effects can be expected to contribute to overall peak broadening. This is evidence by the 0.12° decrease in peak broadening for when moving from a 450 °C to a 700 °C heat treatment, as shown in Table 8.5. Due to the small change in grain size, this

decrease in peak breadth cannot be solely due to crystallite size effects as the Scherrer equation suggests only a 0.05-0.08° decrease in peak breadth that is attributable to size effects.

Similarly, for samples without a white layer that were heat-treated, the effect of lattice strain on overall peak breadth is significant as the change in grain size before and after heat treatment in surfaces without a white layer is insufficient to result in the peak broadening that was measured. Lattice strain is present in these non-white layer surfaces due to the forging and cross-rolling process in the as-received material and the effects of near-surface deformation during cutting in the as-machined sample. It can be inferred that the same lattice strain effects must be present in the white layer samples because the surface plastic strains are greater in these surfaces, as quantified during the strain grid measurements in section 6.1.7.1.

It was not possible to quantify the weightings of crystallite size and lattice strain at low diffraction angles, due to the compounding of their effects during heat treatments, however, it is apparent from the W-H results and the scans using different diffractometers that these weightings are dependent on the diffraction angle used and the diffraction peak under investigation. The more dominant effect of lattice strain on overall peak breadth at high diffraction angles during iXRD measurements is to be expected as at high diffraction angles (above $\theta > 70^\circ$), lattice strain effects are three times larger, relative to grain size effects, than at low diffraction angles ($\theta < 25^\circ$), as shown previously in Figure 4.18. The enhanced sensitivity to grain size effects at low diffraction angle XRD- Δ PB measurements, such as in the Xpert³ measurements, is preferable because the grain size is an intrinsic property of the white layer, whereas lattice strain effects are also present in non-white layer surfaces.

8.1.7 The theoretical resolution capability of the XRD- Δ PB method

Due to the limited number of samples with white layers thinner than the x-ray penetration depth, it was not possible to determine the resolution capability of the peak broadening method using as-machined samples. The EP surfaces could not be used either, as they do not represent an as-machined sample due to lattice strain re-distribution following material removal in the near-surface region, which alters the peak breadth despite the grain size remaining unchanged. A simple mathematical model for detecting white layers using the peak broadening method can instead be proposed based on the theory of XRD and the ultimate theoretical resolution capability of the XRD- Δ PB method can then be defined. Specifically, a

theoretical curve can be generated to describe the probability of detecting white layers with different thicknesses.

The intensity of x-rays after travelling a distance L through a material is described by Eq. 8.1 where μ is the linear attenuation coefficient and I_0 is the intensity of x-rays at the surface of the material.

$$\frac{I(L)}{I_0} = e^{-\mu L} \quad \text{Eq. 8.1}$$

In XRD Bragg-Brentano geometry, the incident x-rays and diffracted x-rays are not perpendicular to the sample surface [275]. As such, the path length L travelled by the diffracted x-rays through the material is greater than the depth x from which the x-rays were diffracted and is described by Eq. 8.2. This is illustrated in Figure 8.16 (a).

$$L = x \left(\frac{1}{\sin(\theta)} + \frac{1}{\sin(\theta)} \right) \quad \text{Eq. 8.2}$$

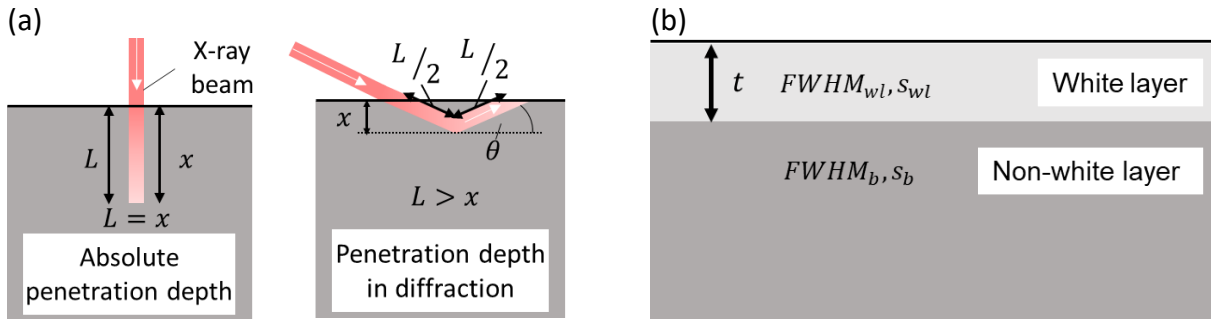


Figure 8.16 – Schematics outlining (a) the concept of x-ray penetration depth and (b) a simplified representation of a surface with a white layer.

The intensity fraction of the x-rays reaching the detector from diffraction at a depth of x , relative to the x-rays incident on the surface, can be found by substituting Eq. 8.2 into Eq. 8.1, resulting in Eq. 8.3. As such, the fraction of x-rays diffracted beneath the depth x has to be $I(x)/I_0$, with the remainder of the x-rays $(1 - I(x)/I_0)$ diffracted above this depth.

$$\frac{I(x)}{I_0} = e^{-\frac{2\mu x}{\sin(\theta)}} \quad \text{Eq. 8.3}$$

To simplify the model, it can be assumed that a machined surface can be represented as two distinct layers, a white layer on top of non-white layer material, as shown schematically in Figure 8.16 (b), with each layer possessing an average peak breadth, $FWHM$, and a standard deviation of peak breadths, s . For x-rays reaching a depth greater than the white layer thickness, t , a proportion of the x-rays will be

diffracted in the white layer, by substituting the white layer thickness into Eq. 8.3 the fraction of x-rays diffracted outside the white layer can be determined. Thus the relative weightings of the x-rays diffracted in the white layer, w_{wl} , and outside the white layer, w_b , can be found from Eq. 8.4.

$$w_{wl} = 1 - e^{-\frac{2\mu t}{\sin(\theta)}}, \quad w_b = e^{-\frac{2\mu t}{\sin(\theta)}} \quad \text{Eq. 8.4}$$

If we assume that the XRD peak corresponding to a particular crystallographic plane can be described by a Gaussian distribution centred on the diffraction angle, 2θ , as shown in Figure 8.17 (a), then the resulting diffraction peak, which arises from the interaction of x-rays with both layers, is another Gaussian distribution in the diffraction angle domain. The variance, σ^2 , in the diffraction domain of the new distribution when summing two Gaussian distributions is equal to the square sum of the individual variances from each layer, in this case, the white layer and the non-white layer material. The *FWHM* of a Gaussian distribution is equivalent to 2.355σ . As such, the peak breadth of the new resultant Gaussian distribution in the diffraction domain can be obtained through Eq. 8.5. Here, $FWHM_r$ is the resultant average peak breadth for the machined surface being inspected, $FWHM_{wl}$ is the average peak breadth of surfaces containing a white layer and $FWHM_b$ is the average from non-white layer samples.

$$\begin{aligned} FWHM_r &= 2.355 \times \sigma_r = 2.355 \times \sqrt{w_{wl}\sigma_{wl}^2 + w_b\sigma_b^2} \\ &= \sqrt{w_{wl}(2.355\sigma_{wl})^2 + w_b(2.355\sigma_b)^2} \\ &= \sqrt{\left(1 - e^{-\frac{2\mu t}{\sin(\theta)}}\right)(FWHM_{wl})^2 + e^{-\frac{2\mu t}{\sin(\theta)}}(FWHM_b)^2} \end{aligned} \quad \text{Eq. 8.5}$$

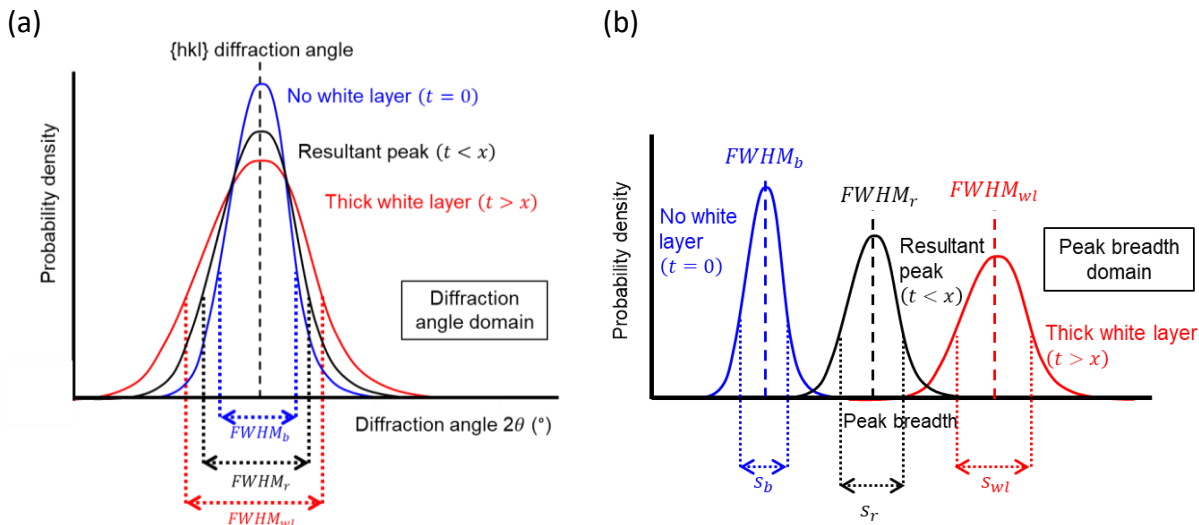


Figure 8.17 – Schematics showing (a) Gaussian distributions for an XRD peak corresponding to a particular (hkl) plane in the diffraction angle domain and (b) the distributions of peak breadths for different types of surface in the peak breadth domain.

$FWHM_r$ is shown as a single value in Figure 8.17 (a), however, there is actually a distribution of possible values for resultant peak breadth in the peak breadth domain, as highlighted in Figure 8.17 (b), in the same sense that there is a distribution of peak breadths for surfaces with white layer and those without, in the diffraction angle domain. If a Gaussian distribution is assumed, this time in the peak breadth domain rather than the diffraction angle domain, then the standard deviation for the resultant breadth in the peak breadth domain, s_r , is the weighted sum of the standard deviations of peak breadths in each layer, s_{wl} and s_b respectively, as described by Eq. 8.6. These standard deviations can be obtained from measurements in machined surfaces.

$$s_r = \sqrt{w_{wl}(s_{wl})^2 + w_b(s_b)^2} \quad \text{Eq. 8.6}$$

In an NDT test for white layers there are four possible results:

- True positive – a white layer is present and is detected
- False negative – a white layer is present but is not detected
- True negative – a white layer is not present and is not detected
- False positive – a white layer is not present but is detected

These results are shown in Figure 8.18 (a) and (b).

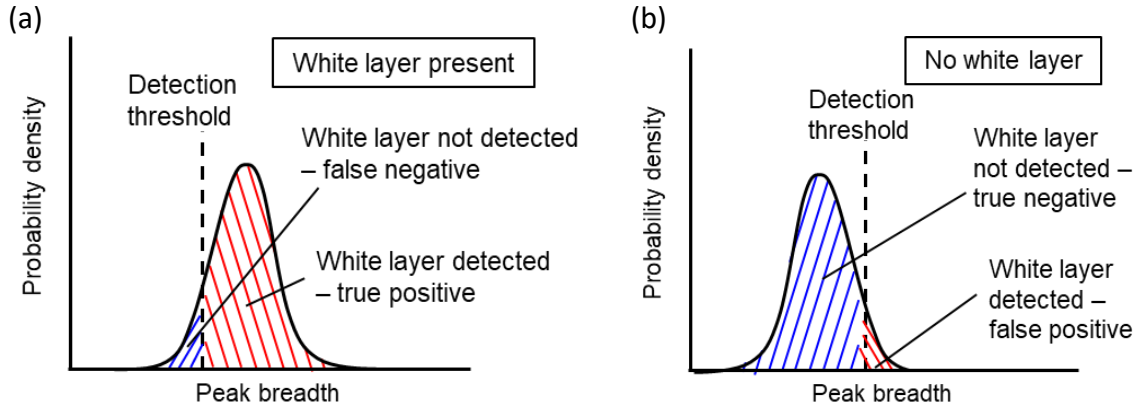


Figure 8.18 –Schematics highlighting (a) the case where a thin white layer is present on the surface but the distribution of possible peak breadths means there is a chance of a false negative and (b) the case where no white layer is present but the peak breadth distribution means a false positive is possible.

To estimate the probability of detecting a white layer as a function of the white layer thickness and the selected peak breadth threshold value, β , the area under the Gaussian distribution above the threshold value can be calculated from the cumulative distribution function (*CDF*) as shown in Eq. 8.7. The error function (*erf*) in Eq. 8.7 arises from the integration of the normal distribution when obtaining the area

underneath the curve. The triggering of the detection threshold can occur due to a true positive or a false positive test, as indicated by Figure 8.18 (a) and (b).

$$\begin{aligned}
 POD(t, \beta) &= 1 - CDF(t, \beta) \\
 &= 1 - \frac{1}{2} \left[1 + \operatorname{erf} \left(\frac{\beta - FWHM_r}{s_r \sqrt{2}} \right) \right] \\
 &= 1 - \frac{1}{2} \left[1 + \operatorname{erf} \left(\left(\beta - \sqrt{\left(1 - e^{-\frac{2\mu t}{\sin(\theta)}} \right) (FWHM_{wl})^2 + e^{-\frac{2\mu t}{\sin(\theta)}} (FWHM_b)^2} \right) / s_r \sqrt{2} \right) \right]
 \end{aligned}
 \tag{Eq. 8.7}$$

As highlighted in the previous chapter, the minimum detectable feature size is often defined by the size, $t_{90/95}$, for which there is a 90% POD with 95% confidence [279]. To calculate the 95% confidence POD curve, the lower 95% confidence interval was calculated for the white layer samples, as this represents the worst-case separation between the peak breadths. Note that the upper 95% confidence interval could also be found for the samples without a white layer and used in combination with the lower 95% white layer interval, however, this was seen to increase the POD, therefore reducing $t_{90/95}$. This unexpected result was due to a higher number of false positives, so only the 95% white layer confidence interval was used in the determination of $t_{90/95}$.

To calculate the confidence interval, Eq. 8.8 was used, where n is the number of samples in the sample set and $tdist$ is the value from the t -distribution used to account for a sample set smaller than 30 samples [278].

$$FWHM_{95\%} = FWHM_{95\%} - \frac{tdist \times s}{\sqrt{n}} \tag{Eq. 8.8}$$

The 95% confidence interval describes the range of values for which there is a 95% confidence that the true population mean is contained within the range. The need for a confidence interval arises from the assumption that the average peak breadth calculated from the white layer samples generated in this project, is the true mean. If all trials were repeated the mean would likely change, however, 95% of the time this new mean would lie in the 95% confidence interval. This new 95% peak breadth was used to calculate the 90/95 POD curve using Eq. 8.7 for each material based on the initial experimental results, as shown in Figure 8.19 (a). The minimum detectable white layer thickness can be found from the POD curves. The ideal threshold gives a high POD with a low probability of false positive (POFP), however, as the POD

increases so does the POFP. Consequently, to select a threshold, a maximum POFP, in this case 1%, must be defined and then the threshold peak breadth tuned up to this limit. This can be visualised by considering the plot in Figure 8.19 (b), which can be used to find the optimum threshold and therefore smallest $t_{90/95}$ for different POFP limits.

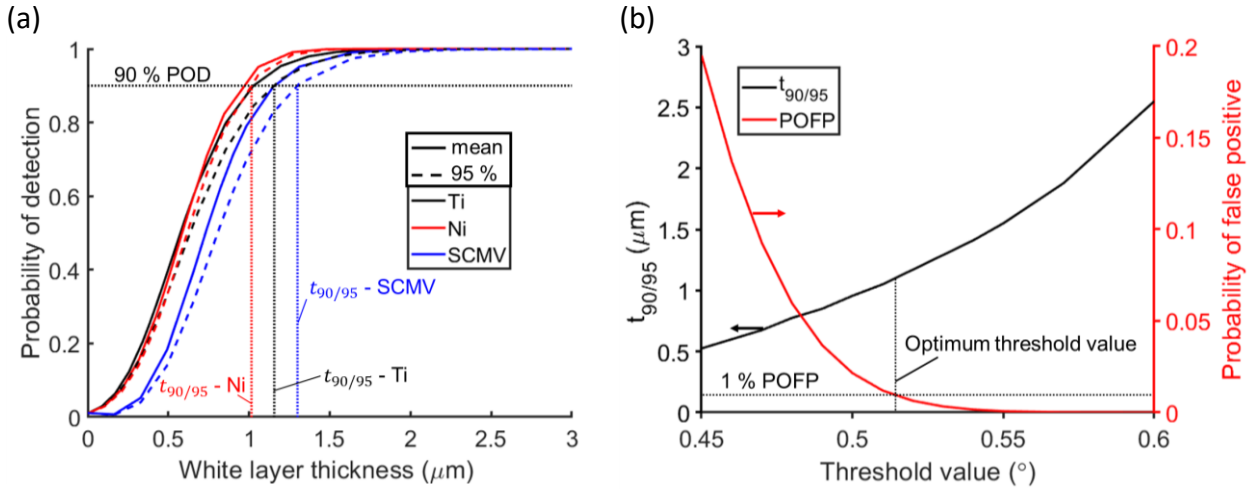


Figure 8.19 – POD curves for each material calculated from the proposed mathematical model and (b) a plot displaying the POFP and minimum detectable thickness on the XRD- ΔPB threshold values.

The input parameters for the mathematical model are shown in Table 8.6. Knowledge of the composition of each alloy in combination with a database of mass attenuation coefficients [221] was used to find the composition weighted μ in all three materials.

Table 8.6 – Values used to generate theoretical POD curves and the corresponding minimum detectable white layer thickness in each aeroengine alloy in this study.

Material	μ (cm^{-1})	$FWHM_{wl}$	$FWHM_b$	s_{wl}	s_b	β	$t_{90/95}$ (μm)
Ti-6Al-4V	926.2	0.732°	0.431°	0.037°	0.043°	0.513°	1.15
IN-718	1191.8	1.105°	0.732°	0.001°	0.0522°	0.854°	1.02
SCMV	2352.6	1.337°	1.009°	0.095°	0.0633°	1.154°	1.30

It can be seen from Table 8.6, that each alloy has a different minimum detectable white layer thickness. This is due to their different linear attenuation coefficients, which dictate the depth of penetration of x-rays in each material, in addition to the separation in the peak breadth domain between samples with and without a white layer obtained from existing measurements on as-machined samples. Focusing on just Ti-6Al-4V, the minimum detectable white layer thickness as predicted by the mathematical model according to the 90/95 criteria is approximately 1.15 μm . By contrast, producing an experimental POD curve from the results of the EP white layers predicts a minimal detectable white layer thickness of at least 3 μm , as

shown in Figure 8.20 (a). However, by evaluating the mathematical model specifically for EP surfaces with white layers, rather than as-machined surfaces, another theoretical POD curve can be produced which shows a closer agreement with the experimental results, as shown in Figure 8.20 (b).

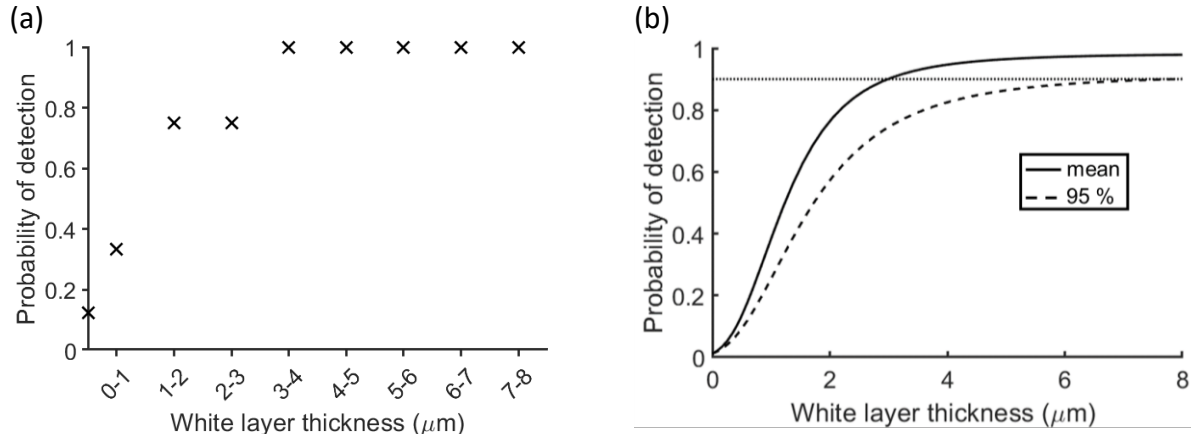


Figure 8.20 – A POD curve (a) measured from the EP Ti-6Al-4V white layer samples and theoretical POD curves (b) based on the proposed mathematical model using data from the EP Ti-6Al-4V samples.

The discrepancy between the POD curves for the measured EP samples and the mathematical model based on data from as-machined samples is to be expected since the model uses several assumptions that simplify the real-world case. These assumptions are:

- The distributions of peak breadths for white layer and non-white layer samples are Gaussian
- The XRD peaks have a Gaussian distribution
- The material beneath the white layer has a peak breadth equivalent to the bulk material or a machined surface with a good SI.
- The EP process for removing material results in a surface representative of a machined surface with the same white layer thickness

The true distribution of the peak breadths for white layer and non-white layer samples is not known due to the limited number of samples investigated. XRD peaks do not typically have a Gaussian distribution in the diffraction angle domain, instead, they are often best represented by a weighted combination of Gaussian and Lorentzian distributions [276]. However, this would increase the complexity of the mathematical model but would have only a minimal impact on the predicted resolution, as this is affected the most by the distribution of $FWHM_{wl}$ and $FWHM_b$ in the peak breadth domain. It is not possible to measure the peak breadth from the region beneath the white layer without removing the white layer

through a technique such as electropolishing, as used in this study, however, it has been shown already that there is typically a large swept grain region beneath a white layer formed through SPD. This swept grain material may result in additional peak broadening over the bulk material, due to grain refinement, as shown through SEM images in the swept grain region, and lattice strain, as was shown from the iXRD peak breadth profiles. Additionally, it has also been highlighted that the EP samples are not entirely representative of as-machined samples and the POD curve here is calculated using a limited set of samples whereas, typically, a full POD study requires 27 samples per bin and this was not possible with the resources available. As such the discrepancy between the theoretical POD curve from the as-machined samples and that measured from the EP samples is to be expected. It should also be noted that the mathematical model did not adjust for instrumental peak broadening, however, this broadening shifts all peaks equally so would not affect the POD.

The similarity between the experimental and theoretical POD curves for EP samples shown in Figure 8.20 (a) and (b), indicates that the mathematical model is a suitable simplification under the correct input conditions. As such, the 95% confidence level predictions for the minimum detectable thickness may be achievable with appropriate as-machined samples. However, to verify this experimentally with statistical confidence, a large number of machined surfaces with white layers 1-5 μm thick would be required and this is beyond the resources of this project.

8.2 A texture-sensitive XRD method for white layer detection (XRD- Δ IR)

In this section, the results from texture-sensitive XRD- Δ IR measurements on machined surfaces are presented before discussing the results in the context of the physical properties of white layers and assessing the methods against the XRD- Δ PB method.

8.2.1.1 XRD- Δ IR white layer detection in Ti-6Al-4V

The IR for a large number of Ti-6Al-4V machined surfaces with and without white layers, as well as samples of as-received material with different crystallographic textures, is shown in Figure 8.21 (a). The IR was larger in surfaces with a white layer compared to those without, irrespective of as-received texture. According to the graph, a threshold of the IR can be set to approximately 3 to detect a surface containing a white layer in all samples investigated, while an IR value less than 2 would be an indicator of a surface without a white layer.

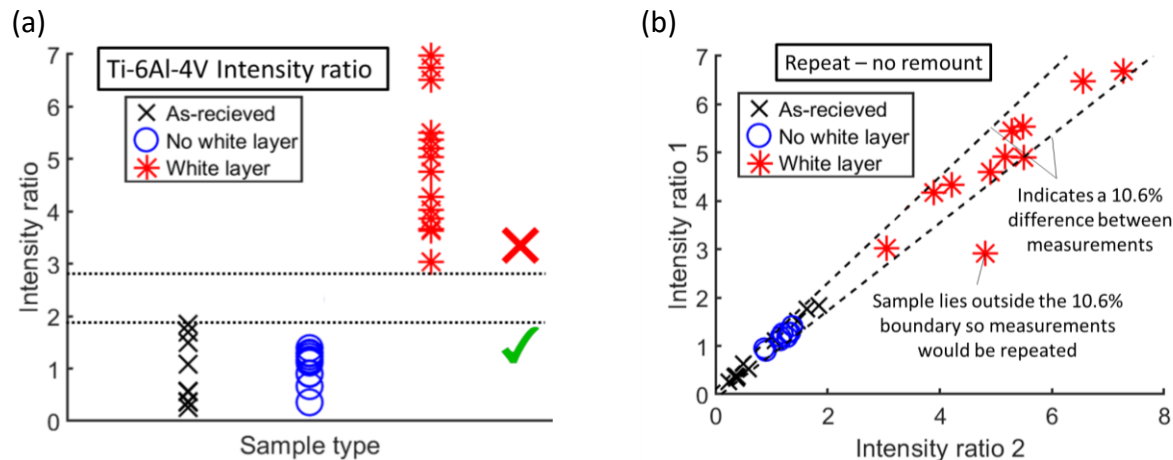


Figure 8.21 – (a) The IR for samples with and without white layer as well as as-received material with different textures and (b) repeat IR measurements used to identify incorrect sample mounting.

As with the XRD- Δ PB measurements described in section 8.1.2.1, each machined surface was measured twice, with the surface remounted between each measurement to assess the errors associated with running the technique, particularly those linked to sample mounting. For these measurements, a 10.6% threshold criterion was adopted for repeating the measurements as this corresponds to the 3σ confidence level for the intensity ratio measurements in repeats of the same surface. Samples lying outside the 10.6% threshold, such as that highlighted in Figure 8.21 (b), were judged to have been mounted incorrectly and the measurements were repeated.

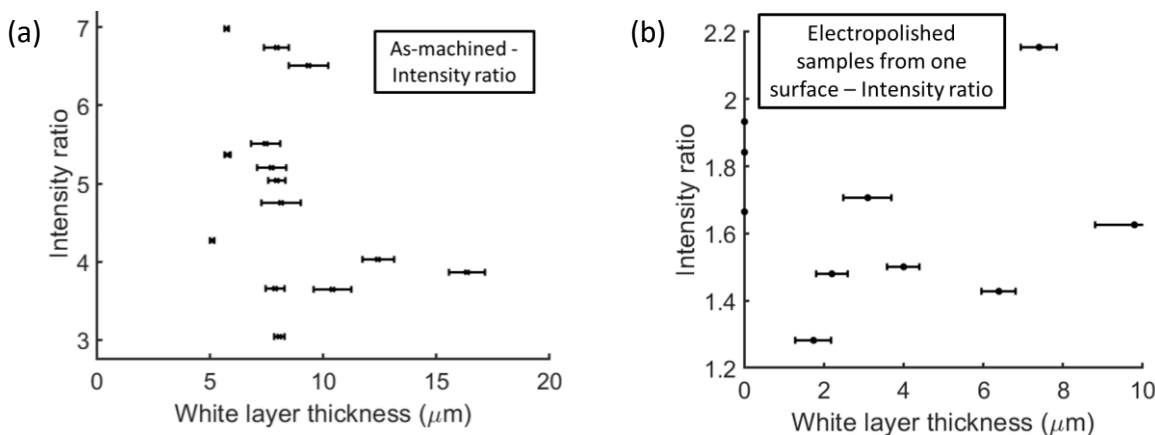


Figure 8.22 – (a) the IR plotted against white layer thickness for as-machined surfaces and (b) the IR plotted against white layer thickness for EP samples from a single machined surface. Error bars represent the standard error of the mean.

The dependence of IR on the white layer thickness for as-machined surfaces and EP surfaces with artificially thinned white layers is shown in Figure 8.22 (a) and (b) respectively. No relationship was found between the calculated IR and the white layer thickness in the as-machined surfaces as the white layer thickness was greater than the x-ray penetration depth in all samples. In the EP samples, where the x-ray

penetration depth was smaller than the white layer thickness, the considerable variation in the measurement points in Figure 8.22 (d) shows that the IR is still independent of the white layer thickness ($R^2=0.01$).

8.2.1.2 XRD- Δ IR white layer detection in IN-718 and SCMV

The IR for IN-718 and SCMV surfaces is shown in Figure 8.23 (a) and (b), respectively, for machined surfaces with and without white layers. Figure 8.23 (a) shows that IR is three times larger in IN-718 surfaces containing a white layer compared to a surface without a white layer. An IR > 12 was indicative of a surface containing a white layer in all IN-718 samples tested and an IR < 4 would appear to be indicative of surface without a white layer. It can be seen from Figure 8.23 (b) that when a white layer is present in a SCMV surface the IR is lower, however, there is no clear separation between surfaces with and without white layers, as measured for the other alloys investigated. The significantly larger IR measured for all SCMV surfaces compared to IN-718 and Ti-6Al-4V surfaces is due to the dominant {110} peak in the diffraction pattern for SCMV, as shown in Figure 8.3 (b), which has a far higher intensity than all other peaks.

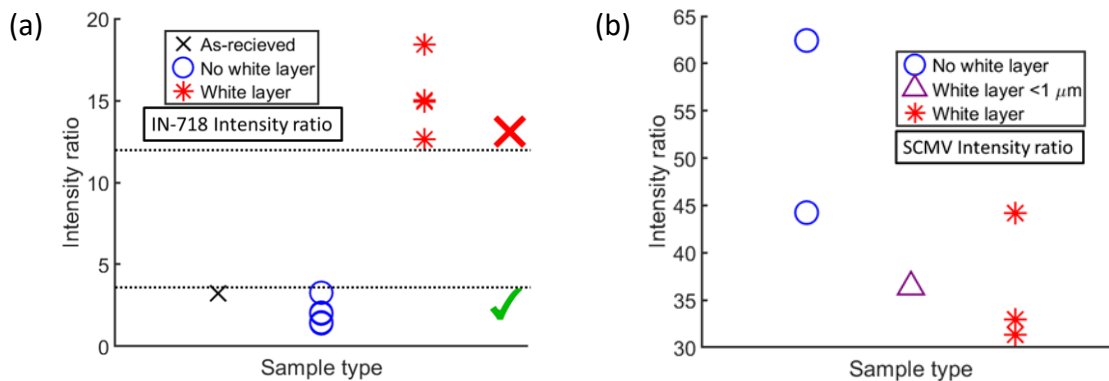


Figure 8.23 – Plots of IR for (a) IN-718 and (b) SCMV surfaces.

8.2.2 Discussion and assessment of the grain-size sensitive XRD- Δ IR method for white layer detection

Results indicate that the XRD- Δ IR method can be used to identify machined surfaces containing a white layer in Ti-6Al-4V and IN-718 due to large changes in XRD peak intensity relative to a surface with no white layer. In Ti-6Al-4V these changes in IR could be directly associated with crystallographic re-orientation of the white layer relative to non-white layer material, as identified in section 6.1.3. No significant change in texture type or texture index was associated with white layer formation in IN-718 and SCMV, however the XRD patterns from phase analysis in IN-718, as shown in Figure 6.5 (c), indicate changes in intensity, indicating that although the texture type is the same, the texture is rotated slightly. These changes in

intensity in the simple XRD pattern during phase analysis are not immediately apparent from pole figure measurements because a pole figure describes the results from intensity measurements from all directions on a hemisphere. By contrast, the simple XRD pattern shows only one point on a hemisphere. As such, it is possible to observe differences in the simple diffraction pattern, which are not immediately apparent in the pole figures. The XRD- Δ IR method cannot be applied to SCMV, as the change in intensity of the crystallographic texture in the white layer is not large enough.

The absence of a significant link between the IR and the white layer thickness in the electropolished Ti-6Al-4V samples with white layers thinner than the x-ray inspection depth can be explained by considering that the texture index of the pole figure ODF for surfaces with a white layer is not proportional to the white layer thickness, as shown in Table 6.3. As such, it would not be possible to determine the size of white layers using this XRD- Δ IR method as the texture is not dependent on the white layer thickness.

Development of the XRD- Δ IR method expands significantly on previous work in which a change in peak height during was recorded during XRD of machined titanium [142] and attributed to metallurgical machining damage. The XRD- Δ IR method is capable of white layer detection in a number of alloys, whereas previous work was limited to the detection of general damage in one alloy, Ti-6Al-4V [142]. Additionally, the underlying basal crystallographic texture change in titanium upon which the XRD- Δ IR method for this material is based, has been identified, whereas this was not investigated by Patton et al. [142].

Since the crystallographic texture is not intrinsic to white layers and their formation in all materials, the reliance on a particular texture within the white layer for detection using the XRD- Δ IR method is less robust than the reliance on intrinsic properties such as the grain size or lattice strain, as in the XRD- Δ PB method. It is reasonable to expect a white layer formed by the phase transformation mechanism could possess a significantly different texture to that formed by SPD and therefore would produce different results under XRD- Δ IR measurements. Therefore, the XRD- Δ IR test is only effective in certain circumstances when white layers are formed by SPD through machining with extremely worn inserts in nickel and titanium alloys, but is not suitable for identification of white layers formed by different mechanisms or processes in all materials, as evidenced by its ineffectiveness for SCMV surfaces. As such, the IR method offers no improvement over the XRD peak breadth technique for the detection of white layers.

8.3 A texture-sensitive SAW method for white layer detection (SRAS)

In this section, the results from texture-sensitive SRAS measurements on machined surfaces are presented before discussing the results in the context of the physical properties of white layers and assessing the method against the XRD- Δ PB method.

8.3.1 SAW velocity and orientation maps

SRAS was used to obtain velocity maps (plots of SAW propagation velocity for different locations across a surface) for a sample which contained a white layer in only one region of the surface after the polishing that was required to remove the surface roughness. The dependence of the SAW propagation velocity on the underlying near-surface microstructure is shown in Figure 8.24, where it can be seen that the presence of a white layer appears to result in an increase in the SAW velocity for certain propagation directions during SRAS. For the 130° scan, the high velocity (red) band at the top of the image can be attributed to the 7 μ m white layer present in this region. The lower velocity in other regions of this scan corresponds to swept grain material with no white layer. However, for the perpendicular 40° scan the velocity distribution changes and there appears to be no underlying link to the white layer region. The difference in velocity maps for perpendicular directions must be attributed to a non-uniform velocity circle. It should be noted that the penetration depth of the SAW during these SRAS methods was 24 μ m and therefore the velocity distribution will be significantly affected by the swept grain material in all cases.

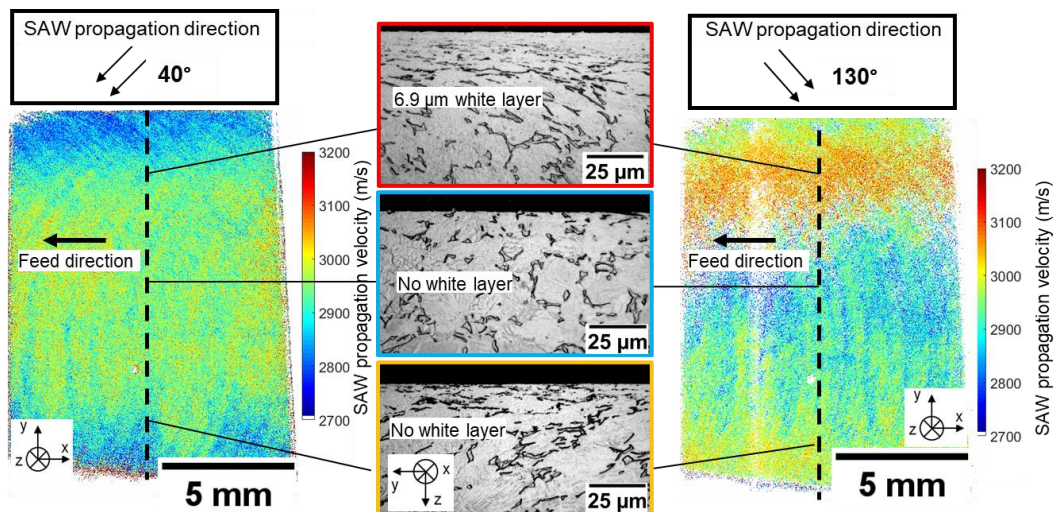


Figure 8.24 – Velocity maps for two orthogonal SAW propagation directions and corresponding cross-sectional micrographs of a surface containing a white layer (40 m/min, 0.5 mm VB).

It is apparent from Figure 8.24 that the velocity distribution only varies in a direction perpendicular to the feed direction, i.e. across the width of the machined shoulder. This is unsurprising as the underlying microstructure did not change either, along a direction parallel to the feed direction. This reason for this band structure is discussed in section 8.3.4.

Figure 8.25 shows the SAW velocity maps for the as-received material, a machined surface with good SI and a machined surface with a white layer. For both the as-received material and a machined surface with good SI, Figure 8.25 (a) and (b), the velocity maps reveal macrozones spread across the surface. These macrozones contain material with a similar crystallographic orientation in which the measured SAW velocity is comparable between neighbouring measurement points. By contrast, for a surface containing a white layer, Figure 8.25 (c), the macrozones are not present and are replaced by a more uniform velocity distribution across the surface. It should be noted that the sample with the regions of highest velocity is the as-received material, rather than the surface with a white layer.

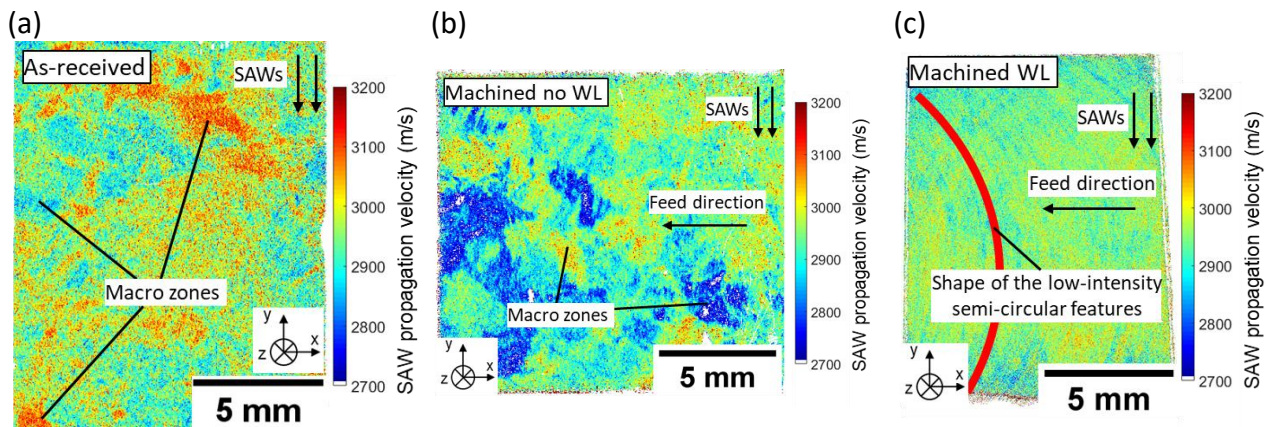


Figure 8.25 – SRAS velocity maps showing the SAW velocity distribution for (a) as-received material, (b) a machined surface with no white layer (20 m/min, 0 mm VB) and (c) a machined surface that contains a white layer (40 m/min, 0.5 mm VB).

In Figure 8.25 (c), low-intensity semi-circular features are faintly visible. These features are believed to correspond to the effect of the worn tool interacting with the workpiece near-surface, although they cannot be attributed to the physical feed marks that appear on the machined surface. This point is shown more clearly in Figure 8.26 (a) and (b), where an optical micrograph of a machined surface after 5 μm of material removal and a SRAS velocity map of the same surface after 20 μm of material removal are shown, respectively. It can be seen that the physical feed marks that are present on the machined surface in Figure 8.26 (a) are in the opposite direction to the semi-circular features that are visible in the SRAS scan, Figure 8.26 (b). As such, the features in the SRAS scan cannot be caused by surface roughness. Instead, they must

be attributed to subtle changes in the underlying near-surface crystallographic orientation in the workpiece during machining on multiple tool passes, which causes small changes in velocity. A discussion around the reason for the difference in appearance between the physical feed marks and the semi-circular features in the SRAS velocity map is given in section 8.3.4.

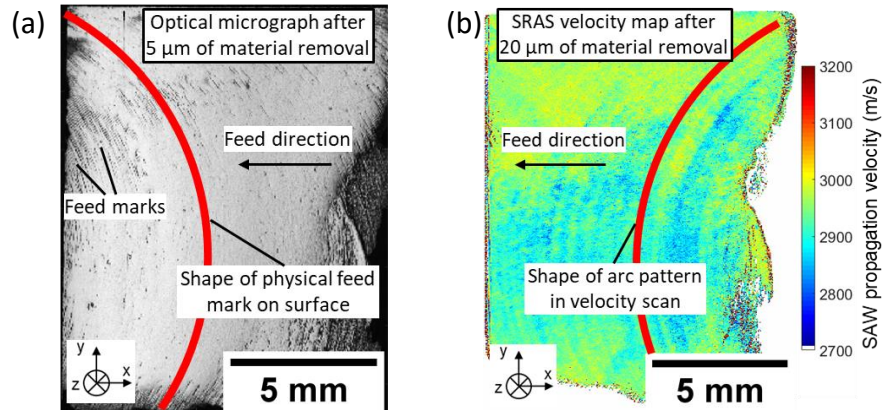


Figure 8.26 – (a) an optical image of a machined surface (80 m/min, 0.5 mm VB) after 5 μm of material removal in the centre and (b) A SRAS velocity map for the same surface after approximately 20 μm of material removal. The semi-circular features in each image are highlighted.

SRAS orientation maps for surfaces with a good SI and poor SI are shown in Figure 8.26 (a) and (b) respectively, these are produced by combining the results from SRAS velocity maps from two perpendicular scans and inferring the crystallographic texture. Due to the use of only two scans, only the basally orientated (white) and non-basally orientated (non-white) material can be identified. For a surface with poor SI, the orientation appears non-basal, despite the presence of a basally orientated white layer within the SRAS inspection depth. The absence of this texture in the orientation maps for the white layer surface can be attributed to the larger size of the SRAS inspection volume compared to the white layer thickness and this is explored further in the discussion section. In the surface with good SI, the orientation varies across the map with a basal orientation visible in some macrozones, in stark contrast to the surface with poor SI which shows no evidence of comparable macrozones and a largely uniform orientation distribution.

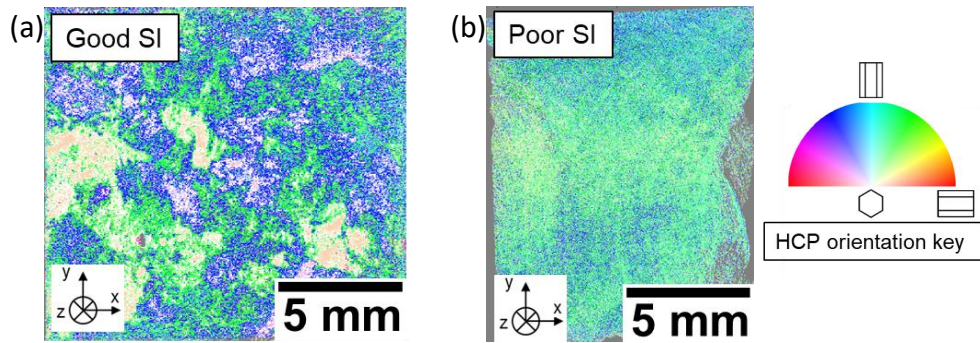


Figure 8.27 – SRAS orientation maps of (a) a machined surface with no white layer (20 m/min, 0 mm VB) and (b) a machined surface that contains a white layer (40 m/min, 0.5 mm VB). The colour coding shows the crystallographic orientation.

8.3.2 The dependence of the average SAW velocity on surface integrity

The average SAW velocity for samples with different SI is shown in Figure 8.28 (a). For each surface the average velocity is calculated using all four SRAS scan angles (0° , 40° , 90° and 130°) and the error bars in the plot correspond to the standard error of the mean. It can be seen from Figure 8.28 (a) that the average velocity is greater for surfaces that possess a poor SI, (i.e. the raw machined surface contained a white layer before any of the polishing needed to prepare samples for SRAS) than for the as-received material and those surfaces possessing good SI. The variation in average SAW velocity with the direction of SAW propagation is highlighted in Figure 8.28 (b). It can be seen that the 0° and 130° scans allow separation between good and poor SI in terms of velocity for the majority of samples, however the as-received material cannot be separated for the 0° scan. The 40° and 90° scans reveal less information on the underlying SI than the other scan directions, with considerable overlap in SAW velocity between samples with different SI.

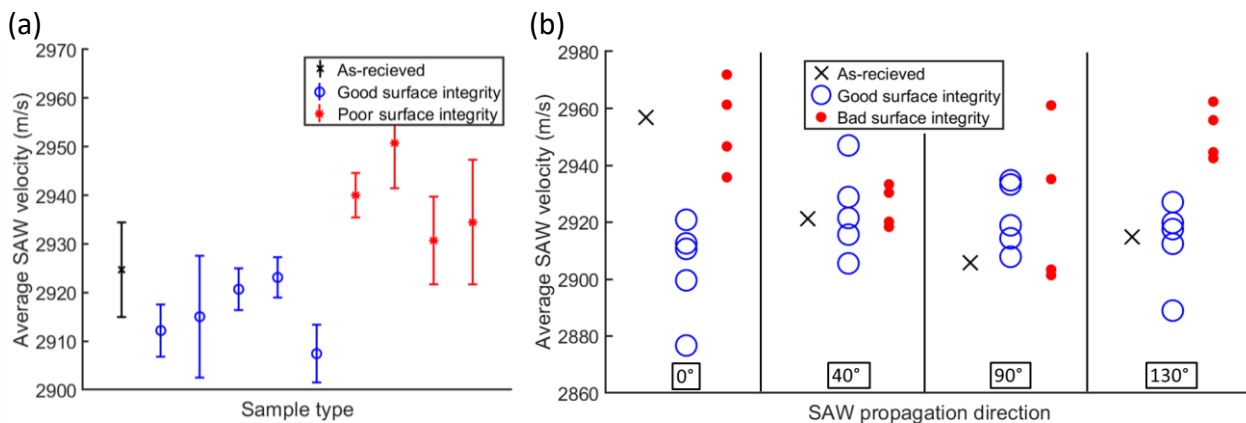


Figure 8.28 – A plot (a) highlighting the average SAW propagation velocity from SRAS scans in different samples and a plot (b) showing the dependence of average SAW velocity on the SAW propagation direction during a SRAS scan. Error bars represent the standard deviation of the mean.

8.3.3 SRAS depth-profiling

To determine how the appearance of the SRAS scan changes with the depth beneath the machined surface, from the white layer region through to the bulk material, SRAS measurements were undertaken at successive depths on a machined surface that originally contained an 8 μm thick white layer, as described in section 7.3. This depth-profiling allows the sensitivity of SRAS to different regions to be assessed, starting in the white layer then moving through the swept grain region to the bulk material. Figure 8.29 shows the SRAS velocity maps at different depths along with the corresponding white layer and swept grain thicknesses.

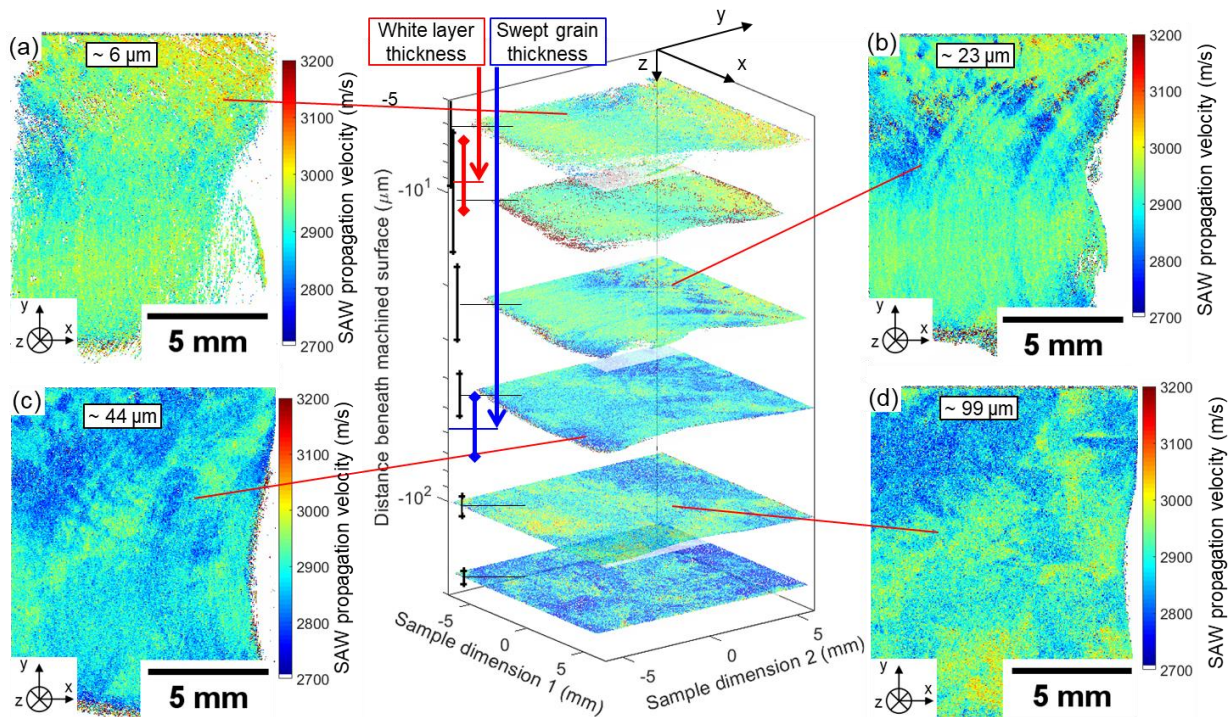


Figure 8.29—SRAS velocity maps at increasing depths (a) 6 μm , (b) 23 μm , (c) 44 μm and (d) 99 μm beneath the machined surface for a sample which originally contained an 8 μm white layer (80 m/min, 0.5 mm VB). The central portion of the figure shows a vertical stack of the surfaces on a logarithmic depth scale with the white layer and swept grain regions highlighted. The error bars on the stack plot highlight the standard error in the depth measurements.

It can be seen from Figure 8.29 (a) that the measured velocity of the immediate sub-surface of the sample, with only 6 μm of material removal, is largely amorphous with no macrozones visible, similar to the map shown previously in Figure 8.25 (c). For a SRAS scan taken after 23 μm of material removal, Figure 8.29 (b), and therefore containing no white layer, low-intensity macrozones and tool impression marks are the only distinguishable features. After 44 μm of material removal, Figure 8.29 (c), approximately half of the swept grain region is removed and macrozones can be more clearly resolved in the velocity map but

there are still tool impression marks visible in the velocity scan, indicating a machining-affected region. Beyond this depth, in the velocity map after 99 μm of material removal, Figure 8.29 (d), the macrozones observed in the as-received material become fully visible and the tool impression marks vanish, as would be expected considering the swept grain material has been removed leaving only the bulk microstructure.

To investigate how the SRAS method performs against the XRD- ΔPB method, XRD measurements were also undertaken on the depth-profiling samples. The XRD peak breadth, together with the average SRAS velocity are shown in Figure 8.30 for increasing depths beneath the machined surface. The white layer region (red) and swept grain region (blue), as quantified from cross-sectional microscopy inspection in an adjacent sample from the same machined surface, are shown at the top of the figure. Only the XRD- ΔPB method was compared against SRAS, as it has already been identified that the XRD- ΔIR is less suited to white layer detection.

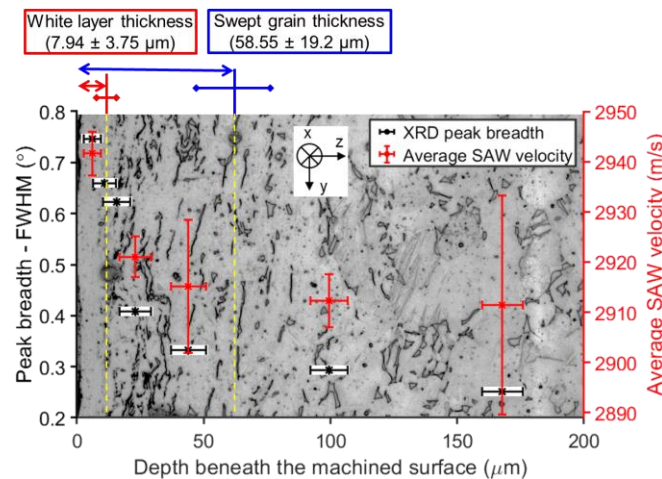


Figure 8.30 – The change in XRD peak breadth and average SRAS velocity with depth in a sample with a white layer (40 m/min, 0.5 mm VB). The plot is superimposed on a representative cross-sectional image used to illustrate the white layer thickness. Error bars represent one standard error of the mean.

Figure 8.30 shows that both methods are sensitive to a change in the properties of the machined surface with depth, as indicated by a decay in the peak breadth and the SAW propagation velocity with increasing depth beneath the original machined surface. It should be noted that there are fewer SRAS measurements than XRD measurements, because two of the SRAS scans, at material removal levels between 6 μm and 23 μm , were of poor quality due to a focusing issue and so they are not included in the plot. However, the absence of these scans does not affect the results from SRAS inspection at different depths. The variation of the XRD peak breadth measurement from the white layer to the bulk (approximately 100%) is much larger than one measured for the average SAW velocity (around 6%),

relative to the range of expected values for a SAW in the tested material. Additionally, the standard error of the mean for the SRAS measurements is greater than for the XRD- Δ PB measurements, which had an error smaller than 0.02° due to the high repeatability of XRD, as highlighted previously.

8.3.4 Discussion and assessment of the texture-sensitive SRAS method for white layer detection

It is apparent from the results presented here that SRAS is sensitive to the presence of a white layer and thick swept grain regions within the SAW interaction volume. In these surfaces with a poor surface integrity, isolated regions of material possessing a similar SAW propagation velocity were absent. These macrozones represent regions of different crystallographic texture in the workpiece material that are retained from the prior processing route in surfaces with a good SI. In surfaces with a white layer or a thick swept grain region, the macrozones are destroyed by the high levels of SPD when machining with worn inserts, resulting in a velocity map that appears more uniform. It should be noted that the absence of macrozones does not reveal whether a surface would fail SI inspection on the presence of a white layer or a thick swept grain region specifically, only that it would fail overall.

In addition to the velocity maps, the orientation maps shown in Figure 8.27 also indicate that the presence of macrozones can be associated with a good SI. It is important to notice that the orientation maps for a surface with poor SI do not show a basal orientation, despite the presence of a white layer at the surface, which has been shown through pole figure measurements to possess a strong basal texture. The absence of a basal texture visible in the orientation map is thought to be due to the larger penetration depth of the SAW ($24\text{ }\mu\text{m}$) compared to the white layer thickness (smaller than $10\text{ }\mu\text{m}$). The swept grain material, which possesses a shear texture, therefore makes up a larger proportion of the inspection volume and so the resulting average texture of the material in SAW interaction volume is non-basal. This means that when two perpendicular SRAS scans are compared, as shown in Figure 8.24, the spatial distribution of propagation velocities are different because material with a non-basal texture has a non-uniform velocity circle, due to the anisotropy of the HCP crystal [39]. This is evidenced by the angular dependence of the average SAW velocity that can be seen across SRAS scans with different propagation directions, as shown in Figure 8.28 (b).

In addition to the absence of macrozones observed in the velocity maps for a surface with a poor SI, the average SAW velocity across four SRAS scan directions was measured to increase relative to a surface

with good SI. The small increases in velocity, despite the white layers being thinner than the penetration depth of the SAW, can be explained by considering the white layer as a distinct layer on top of the non-white layer material, as was described previously when discussing the XRD- Δ PB measurements. The white layer and non-white layer regions each have distinctive SAW properties, as highlighted in Figure 8.31, with a higher propagation velocity for the white layer, due to its basal orientation. Note that in reality there will not be a sudden change in SAW velocity when moving from white layer to non-white layer material. SAWs propagate with a high velocity in all directions on the basal plane compared to any other orientation of the HCP crystal. The resulting average SAW velocity for a surface is, therefore, a weighted combination of the velocities of these two regions that will be related to the relative fraction of the inspection depth that each region occupies. Consequently, despite the white layer making up less than 50% of the penetration depth in all samples tested, the average velocity for a surface that contains a white layer is increased relative to a surface containing only swept grain material.

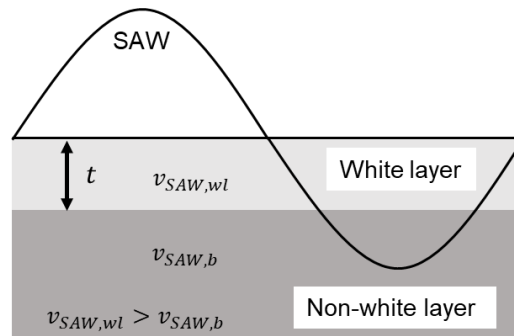


Figure 8.31 – A schematic of SAW propagation on a surface showing the different acoustic properties of the two layers.

The ability to also distinguish machined surfaces with a good or poor SI based on the average SAW velocity obtained from a single SRAS scan direction, as shown in Figure 8.28 (b) is a consequence of the contrasting texture directions for each surface. The 0° and 130° scans facilitate separation based on SI whereas 40° or 90° scans do not. The shear texture in a surface machined with worn inserts is the opposite direction to a surface machined with new inserts, as identified during destructive characterisation in section 6.1.3. Due to their opposite textures, the velocity circle is therefore also opposite for these two types of surface and, as such, if the propagation velocity were fast in a particular SAW direction for a surface with poor SI, then it would be expected that the velocity perpendicular to this would be slower for a surface with poor SI. This relationship is highlighted in Figure 8.32, obtained by averaging the results for good and poor SI surfaces from Figure 8.28 (b) and presenting them alongside representative pole figures.

The surfaces with a poor SI have a higher average SAW velocity overall, due to the influence of the basally orientated white layer within the SAW penetration depth.

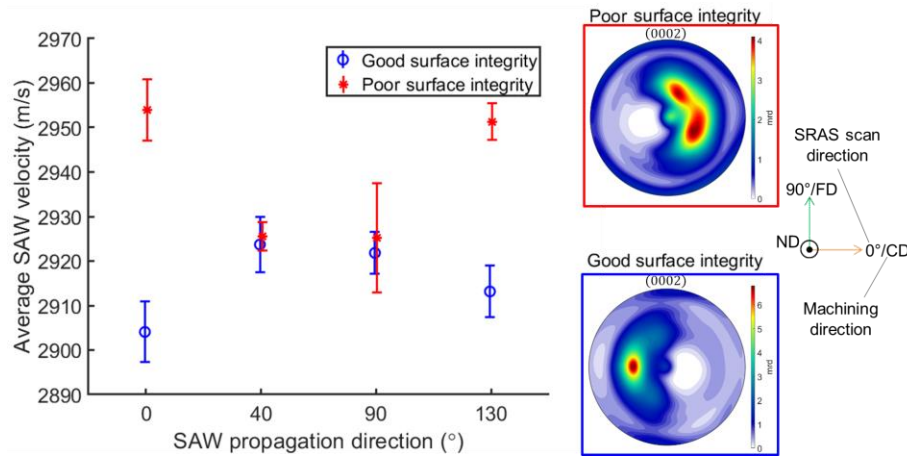


Figure 8.32 – A plot highlighting the contrasting relationships between SAW velocity and propagation direction depending on SI state and corresponding pole figures. Error bars represent one standard deviation of the mean.

The semi-circular features visible in the velocity maps for surfaces with poor SI, as shown in Figure 8.25 (c) and Figure 8.26 (b), are believed to correspond to the path of the cutting tool over the surface and, as such, directly evidence the deformation caused by machining. They cannot be a product of retained surface roughness as these features are still visible in the velocity map, as shown in Figure 8.26 (b), of a surface after approximately 20 μm of material had been removed through polishing, thereby erasing the feed marks. Additionally, the features are in the opposite direction to the physical feed marks visible on top of the machined surface, as shown in Figure 8.26 (a). The semi-circular features in the velocity maps must therefore be representative of the residual deformation in the machined surface, which appears to have resulted in subtle changes in the measured SAW velocity for regions machined on different tool passes.

The change in the shape of the arc at different depths can be explained by revisiting the concept introduced previously in section 6.2.1 to account for the contrasting shear texture between surfaces with a thick swept grain region and surfaces with good SI. In the central region of the machined shoulder, the cutting tool passes over the surface in one direction during the first part of the tool rotation, when forming a chip, and then rubs in the opposite direction during the second half, as outlined in Figure 8.33 (a). As such, the top surface, where the physical feed marks are visible, is dominated by the rubbing on the second half of the tool rotation, but at greater depths the deformation can be in the opposite direction, due to

SPD of the workpiece sub-surface during the first half of the workpiece rotation. This is highlighted in the cross-sectional optical micrograph in Figure 8.33 (b) in which grain distortion in opposite directions is visible at different depths. Consequently, the semi-circular features in SRAS maps can be in the opposite direction to the feed marks on the surface.

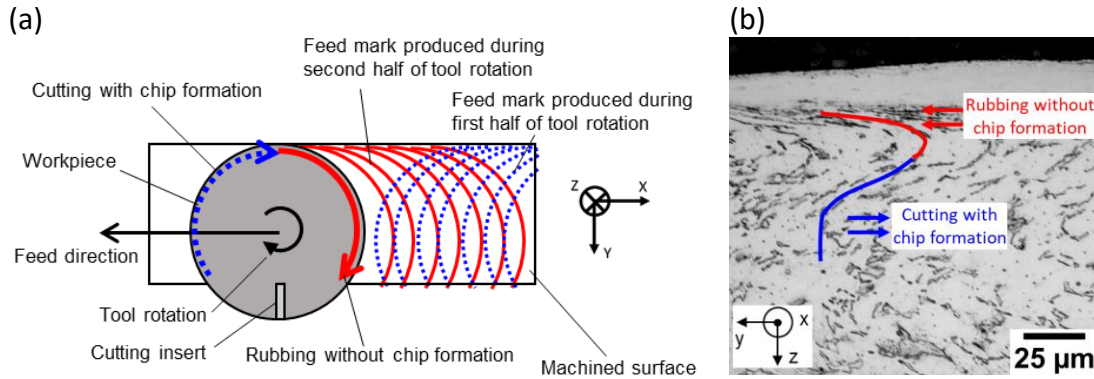


Figure 8.33 – A schematic (a) of the cutting operation highlighting the two arcs produced by the cutting tool during the milling operation and an optical micrograph showing deformation in one direction due to rubbing and deformation in another due to chip formation on the other half of the cutting arc.

One of the main shortcomings of the SRAS technology employed in this study is the need to mechanically polish samples to obtain the optically flat surface required for the detection laser. This mechanical removal of material affects the geometrical configuration of the machined surfaces which are slightly curved, both due to elastic relaxation after machining and a change in cutting tool deflection under the varying forces at different points around the cutting arc. This variation in surface height across the machined surface, caused by the curvature, was on a scale of approximately 10 μm . When polishing this curved surface, the level of material removal was dependent on the position across the width of the machined shoulder, as shown in Figure 8.34 (a). As such, in the SRAS velocity maps of these polished surfaces, distinct bands were found at the top and bottom of the images corresponding to regions of lower material removal, as shown in Figure 8.34 (b) and when these bands were higher velocity, it was typically because these regions contained more white layer material as shown in Figure 8.24.

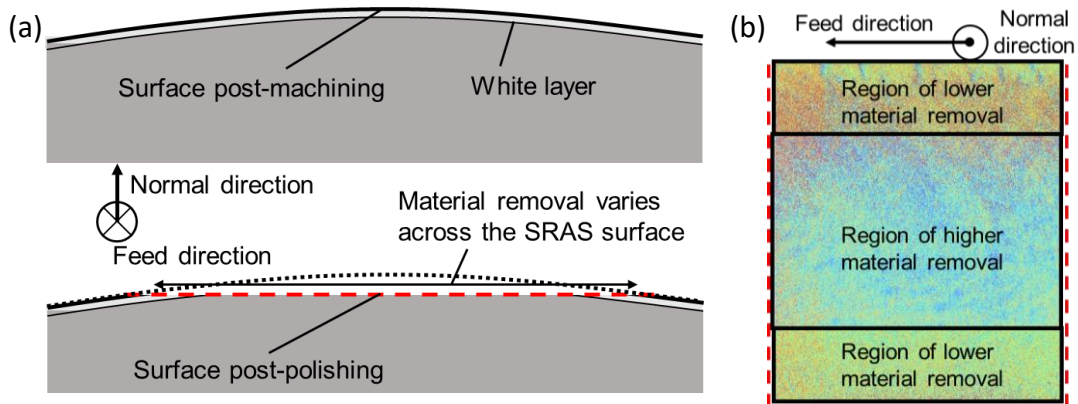


Figure 8.34 – A schematic (a) highlighting the slight curve to the machined surface post-machining and the corresponding bands (b) visible in velocity maps of some surfaces.

The SRAS and XRD- Δ PB depth profiling measurements indicate that the XRD- Δ PB method is more suitable than SRAS as a technique for the detection of white layers. The sensitivity of the XRD- Δ PB method to the intrinsic properties of the white layer, in addition to the improved repeatability, means that the relative difference in the measured property between a surface with and without a white layer is greater and any pass-fail result from inspection has greater statistical significance than for SRAS. Additionally, considering the inherently small penetration depth of x-rays compared to surface acoustic waves in aeroengine metals, XRD can be used to detect thinner white layers compared to the current class of SRAS equipment. It should also be noted that the difference in average SAW propagation velocities between good and poor SI samples is smaller than the standard error in the measurements. As such, the specific detection of white layers, as opposed to general machining damage, does not seem feasible with the current resolution capability of the SRAS, especially as the XRD- Δ PB method is more capable for this task.

Although SRAS is less suited for white layer detection, it may offer more potential as a method for the detection of other aspects of SI, such as swept grain, by associating the presence of arcs in the velocity map with machining damage. This is evidenced in the depth profiling measurements by the ability to resolve tool impression marks in the velocity maps in the swept grain region that are not visible for surfaces with a good SI. The XRD- Δ PB measurements on the same sample, by contrast, have only a slightly higher peak breadth at this depth than within the bulk material, but the peak breadth value is comparable to other machined surfaces with good SI, so would not facilitate swept grain detection in this case. Although the depth profiling was destructive, the same results could be expected if the tool impression marks detected after 44 μ m of material removal had instead been the machined surface, thus facilitating non-destructive inspection. It may also be possible to size the region of machining damage by comparing SRAS

scans at different wavelengths through changing the spacing of the fringe pattern that forms the SAW on the workpiece surface to facilitate inspection at depths greater than 24 μm . XRD, by contrast, is limited to small penetration depths, far smaller than the total thickness of the machining affected region, regardless of the x-ray radiation or diffraction angle used.

The SRAS method for the detection of anomalous machined surfaces offers a number of advancements over previous SAW research carried out by Patton et al. [142], in which changes in the angle of maximum backscatter during SAW testing of a machined test piece submerged in water was measured to change with surface integrity. This previous work [142] identified that the incident probe angle was 1° lower for a damaged surface and attributed this to a change in elastic modulus that increased the SAW velocity. SRAS is similarly dependent on the surface integrity through changes in velocity, however, it also allows detection through the visualisation of the macrostructure in the near-surface region in velocity maps, thereby offering an additional level of detection over the backscatter method. As well as offering more options for anomalous surface detection, this study has also identified the underlying crystallographic texture responsible for the change in elastic modulus that most likely allowed Patton et al. [142] to identify an anomalous surface. Although both methods are non-contact, the requirement for the component to be submerged in water, or another liquid, in the backscatter method means that SRAS may be easier to implement within a machining centre in an on-machine inspection method, if the surface roughness limitation can be overcome.

8.4 A residual stress-sensitive method for detecting white layers in ferromagnetic materials

In this section, the results from BN inspection of SCMV turned surfaces are presented before these results are discussed in the context of the physical properties of the white layers and the other NDT methods reported in this chapter.

8.4.1 Basic Barkhausen noise measurements

The results from basic BN measurements on machined surfaces using a magnetizing frequency of 180 Hz, a voltage of 16 V_{pp} and a 200 kHz – 450 kHz band-pass filter are shown in Figure 8.35. Each point on the plot is the average of 5 BN measurements at different locations across the surface, with the vertical and horizontal error bars representing one standard error of the mean for the BN and the white layer thickness measurements. The magnetizing frequency and voltage were selected as they resulted in a

higher amplitude signal, compared to other parameters, with BN greater than 100 mp in all samples. These basic BN measurements represent the default measurement mode of the Rollscan 350 unit used. No relationship between Barkhausen noise and white layer thickness was found and this basic measurement mode could not be used to separate samples with white layer from those without white layer. Consequently, the previously outlined MCS and MFS methods were investigated.

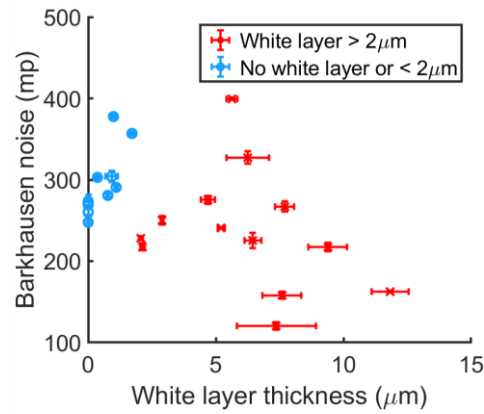


Figure 8.35 – Barkhausen noise plotted against the white layer thickness, error bars represent the standard error of the mean.

8.4.2 Magnetizing frequency sweeps

Results from MFS measurements at 16 V_{pp} are shown in Figure 8.36. Samples with thick white layers (>2 μm) are highlighted in red and samples with thin (<2 μm) or no white layers in blue. It can be seen from the high proportion of red peaks at lower magnetizing frequency that samples with a thick white layer demonstrate a lower f_{max} compared to the other samples which feature a MFS peak at higher frequencies.

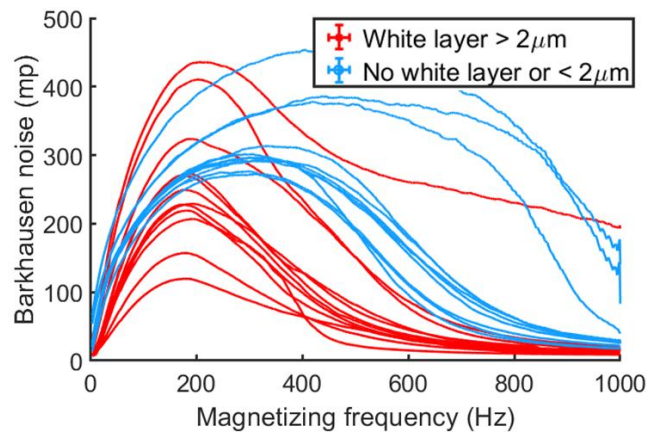


Figure 8.36 – Barkhausen noise plotted against the magnetizing frequency from MFS measurements.

Figure 8.37 (a) plots the f_{max} in relation to the white layer thickness, wherein the samples can be grouped based on the white layer thickness. The blue markers represent samples surfaces with good SI or

those with less than 2 μm of white layer and the red data points show samples with a white layer thicker than 2 μm . It can be seen that a lower f_{max} is associated with surfaces containing a thicker white layer. In contrast to f_{max} , the BN_{max} in a MFS appears to offer no benefit over the basic BN measurement, as both plots, Figure 8.35 and Figure 8.37 (b) respectively, have a similar scatter of points.

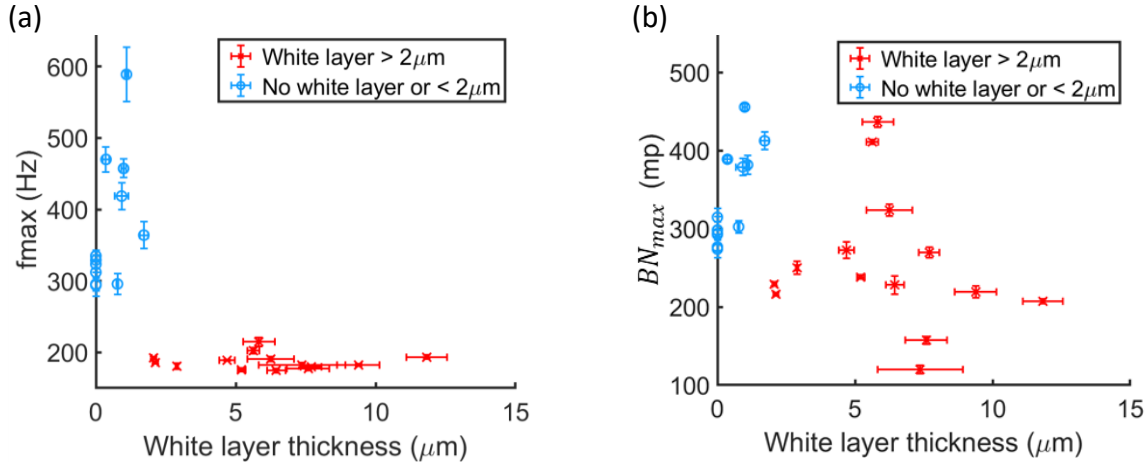


Figure 8.37 – Plots of (a) f_{max} and (b) BN_{max} plotted against white layer thickness, error bars represent the standard error of the mean.

It should be noted that, although the variable f_{max} allows samples to be grouped depending on the presence of a thick white layer, the measured value appears to be independent of the actual thickness, i.e. the difference between the f_{max} for a 12 μm or 3 μm white layer is not significant. Consequently, a mechanical property of the surfaces with white layers, which is not proportional to the white layer thickness, must be facilitating the distinction. The effects of key properties such as hardness, residual stress and surface roughness on the measured f_{max} are outlined in section 8.4.4.

To determine the optimum peak-to-peak voltage for magnetisation, MFS measurements were carried out at three additional V_{pp} levels. The percentage f_{max} difference between samples with thick white layers and all other samples is outlined in Table 8.7. It was found that larger peak-to-peak voltages facilitated a greater f_{max} difference between the sample groupings. Additionally, there was an overlap between sample groupings at 7 V_{pp} , meaning that a number of surfaces would be falsely classified if white layer assessment was made based on f_{max} measurement. The consistent increase in f_{max} and BN_{max} with peak-to-peak voltage can be used to infer that the surfaces under inspection were not magnetically saturated during the measurements, as such further improvement may be offered by increasing the peak-to-peak voltage, however, the Rollscan 350 is limited to a maximum of 16 V_{pp} .

Table 8.7 – Peak-to-peak voltage and the corresponding separation in f_{max} between surfaces with a thick white layer and all other surfaces at a bandpass filter frequency of 200-450 kHz. The error given is the standard deviation of the mean.

Voltage peak-to-peak (V_{pp})	f_{max}/f_{max} (%)	Overlap between sample groupings
7	55 (± 70)	Yes
10	54 (± 36)	No – 5 Hz difference
13	84 (± 42)	No – 52 Hz difference
16	98 (± 47)	No – 81 Hz difference

The effect of filter frequency, at 16 V_{pp} , on the ability to distinguish a surface with a thick white layer from other surfaces, is outlined in Table 8.8. The percentage difference in f_{max} between different sample types decreases with increasing filter frequency, however the spread of results decreases, meaning that the chance of wrongly classifying a surface is decreased at higher frequencies as the sample groupings are further from overlapping. Due to the smaller inspection depth at high filter frequencies, which restricts the measurement to the near-surface, in addition to a lower chance of a false positive reading because of the increased separation between sample sets, the results from inspection at 16 V_{pp} with a 200-450 kHz filter were selected as the optimum BN parameters for further investigation.

Table 8.8 – Filter frequency and the corresponding separation in f_{max} between surfaces with a thick white layer and all other surfaces at 16 V_{pp} . The error given is the standard deviation of the mean.

Filter frequency (kHz)	f_{max}/f_{max} (%)	Overlap between sample groupings	Inspection depth (μm)
10-70	148 (± 96)	No – 52 Hz difference	Up to 340
70-200	115 (± 76)	No – 74 Hz difference	Up to 130
200-450	98 (± 47)	No – 81 Hz difference	Up to 75

8.4.3 Magnetizing current sweeps

Results from MCS measurements at 100 Hz magnetizing frequency on the testing set of SCMV surfaces are shown in Figure 8.38. As with the MFS measurements, the plots for samples with a thick white layer have a different shape than the other samples. In this case, samples with thick white layers (red curves) were found to yield a curve with a lesser gradient, on average compared to other samples (blue curves).

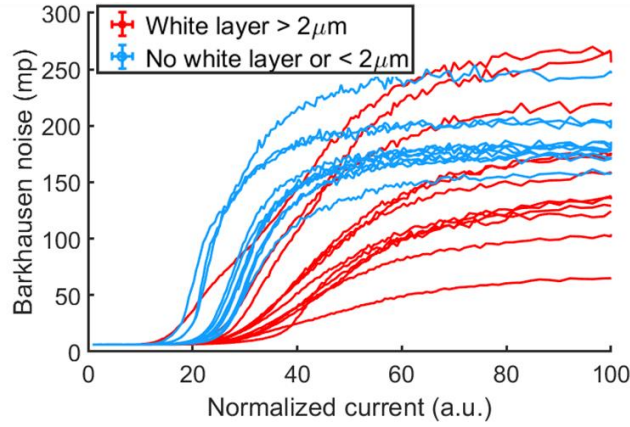


Figure 8.38 - Barkhausen noise plotted against the normalised current from MFS measurements.

The dependence of the four MCS properties outlined previously (the maximum slope S_1 , the current at which this occurs NC_1 , the maximum derivative of the slope S_2 and current at which this occurs NC_2) on the white layer thickness is shown in Figure 8.39. It can be seen that the MCS sweep features do not offer the same level of distinction between samples containing a thick white layer and other samples, compared to the MFS property, f_{max} . The properties, S_1 and S_2 decrease in samples with a thick white layer, while NC_1 increases for thicker white layers, however the separation between the two types of samples was smaller for all of these MCS features than for f_{max} . NC_2 is the worst performing MCS feature as it offers no distinction between sample types.

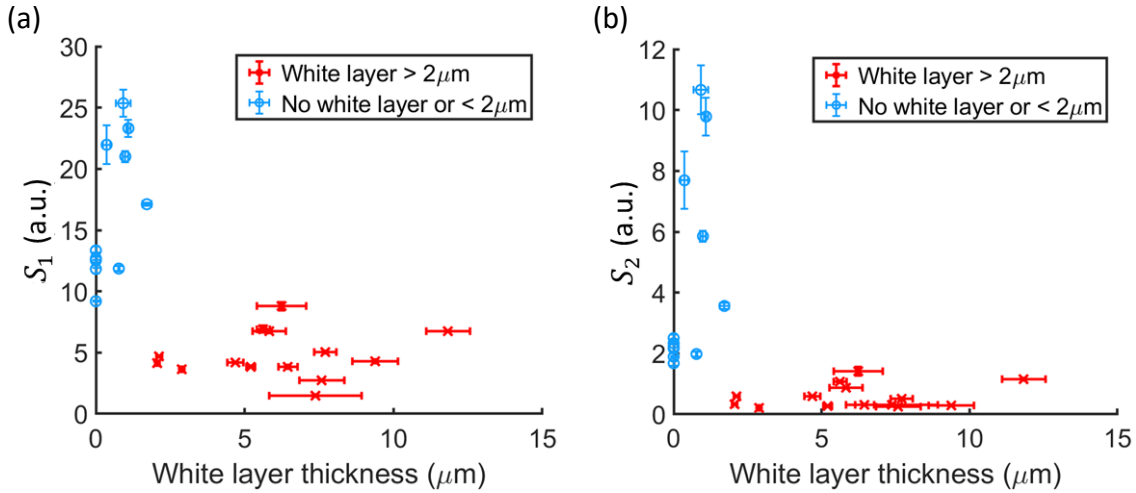


Figure 8.39 continues on the next page

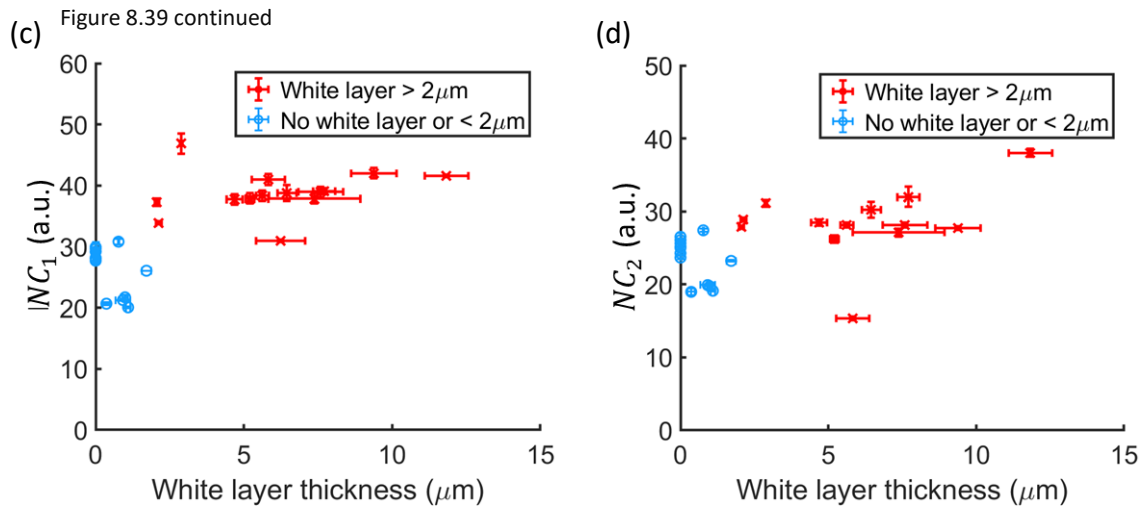


Figure 8.39 – MCS features (a) S_1 , (b) S_2 , (c) NC_1 and (d) NC_2 plotted against the white layer thickness. Errors bars represent one standard error of the mean.

Six different magnetizing frequencies were investigated for the MCS and the results are summarised in Table 8.9. The MCS method is only valid for all samples within a narrow region of magnetizing frequencies (75-150 kHz), as the maximum gradient in the MCS plot was not reached outside of this range, before the maximum current was applied. Even for magnetizing frequencies in this 75-150 kHz range, there was no statistically significant separation between the measured values for thick and thin white layers. Therefore, no threshold value would allow separation between the groups of samples without giving high false positives rates or failing to detect surfaces with a thick white layer.

Table 8.9 – The separation between measured feature values for the two types of surfaces at different magnetizing frequencies during MCS measurements. The error given is the standard deviation of the mean.

Magnetizing frequencies (Hz)	Percentage difference between sample groups (%)			Absolute minimum difference between sample groups (a.u.)		
	S_1 (a.u.)	S_2 (a.u.)	NC_1 (a.u.)	S_1 (a.u.)	S_2 (a.u.)	NC_1 (a.u.)
75	225 (± 162)	47 (± 25)	646 (± 680)	None	0.31	None
100	229 (± 165)	47 (± 25)	618 (± 697)	0.41	0.26	0.15
150	147 (± 131)	150 (± 24)	664 (± 840)	None	None	None
50, 200, 300	* Maximum gradient not reached in all samples within voltage sweep.					

The poor performance of the MCS features compared to f_{max} for the application of identifying anomalous surfaces, makes f_{max} the only suitable choice for further investigation into the relationship between the BN and the physical properties of the machined surfaces.

8.4.4 The relationship between mechanical and surface texture properties and Barkhausen noise measurements

The measured f_{max} is independent of white layer thickness in all surfaces with thick white layers, as such, a secondary property of the material within the BN inspection volume must facilitate the separation of white layer and non-white layer material. It has already been identified that surface roughness, hardness and residual stress can affect the BN signal and, as such, the relationships between these mechanical and surface texture properties and the measured f_{max} were investigated. As outlined previously, the nanohardness and residual stress measurements were weighted in this BN analysis to approximate the enhanced sensitivity of BN to the near-surface region. Nanohardness and residual stress measurements could only be carried out for a subset of samples from the full data set, due to the prohibitive cost of these techniques. The subset was chosen so that samples with a range of f_{max} values and machined with cutting parameters encompassing the full range of cutting speeds and tool wear levels. The as-received material was also incorporated into this analysis.

Surface roughness measurements plotted against white layer thickness, as presented in Figure 8.40 (a), exhibit the same trend as f_{max} that was shown previously in Figure 8.37 (a) and, as such, the surface roughness cannot be said to be significantly affecting the Barkhausen noise measurements. Figure 8.40 (b) shows the variation of f_{max} with the weighted hardness within the BN inspection volume. Although the white layer is known to be harder than the bulk, no significant relationship can be established between f_{max} and hardness as there is a significantly different f_{max} measured for samples with comparable hardness. The low R^2 values from linear regression analysis of the dependence of f_{max} on hardness ($R^2=0.12$) and surface roughness ($R^2=0.18$) suggests that neither property is having a significant effect on the results of BN inspection.

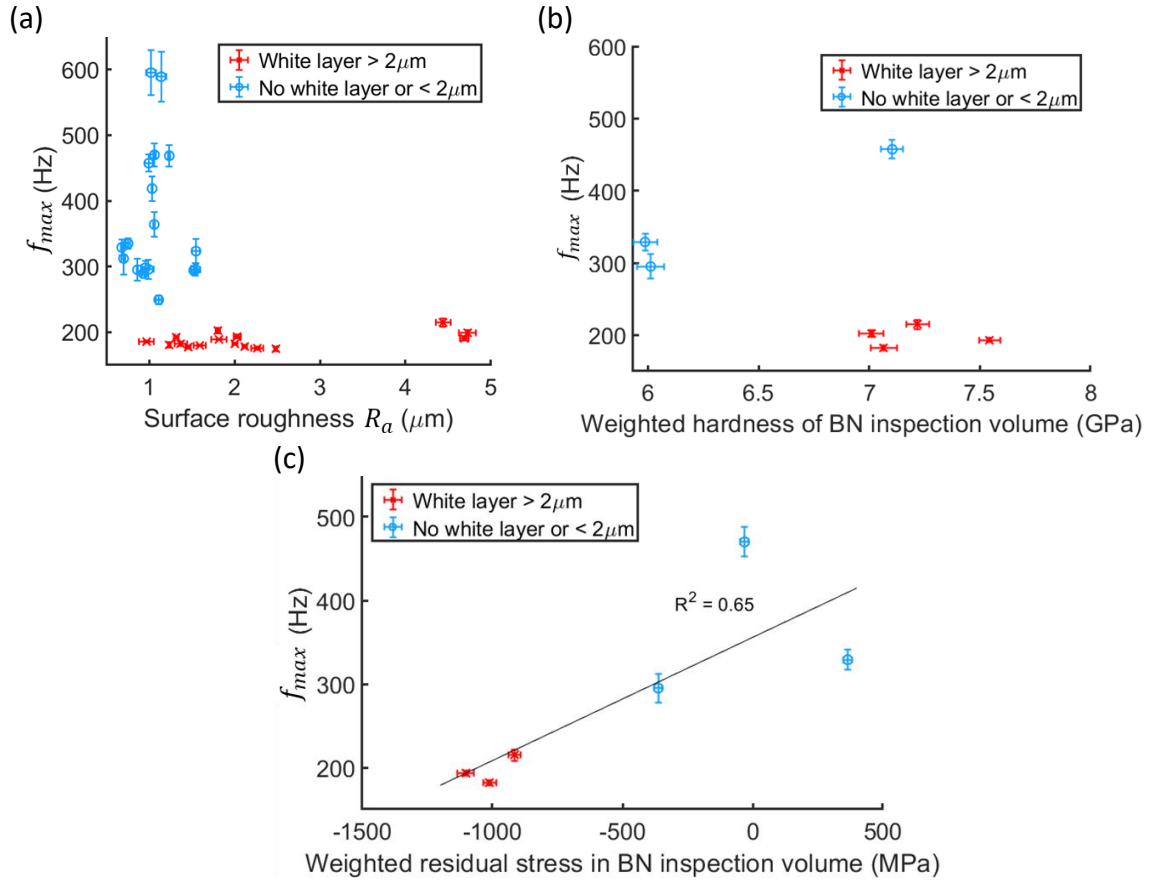


Figure 8.40 – The variation of f_{max} with (a) the surface roughness, (b) the average hardness in the white layer and (c) the average residual stress in the Barkhausen noise inspection volume, error bars represent the standard error of the mean.

Weighted residual stress in the BN inspection volume, by comparison, appears to have a direct impact on the Barkhausen noise measurements, as shown in Figure 8.40 (c). The higher R^2 from linear regression analysis can be used to infer that there is a statistically significant relationship between the measured f_{max} and the residual stress state in the BN inspection volume. A larger compressive residual stress was measured to result in a lower value of peak frequency, i.e. the effect of a compressive near-surface residual stress state is change the shape of the MFS curve.

It should be noted that the grain size within the BN inspection volume may also contribute to the measured f_{max} however, it was not possible to measure the difference in grain size between different samples with a white layer without undertaking a significant number of TEM measurements, which fell beyond the scope and resources of this study. The measurement of XRD peak broadening could be used not give a quantitative estimate of any grain size differences in the BN samples, due to the competing broadening effect of lattice strain known to be present in these surfaces, as outlined previously when discussing the XRD-ΔPB method .

8.4.5 Discussion and assessment of the residual stress-sensitive method for white layer detection

Results from BN- f_{max} inspection of machined surfaces with and without white layers show that surfaces can be sorted into a group containing those surfaces with a white layer thicker than 2 μm and a group containing all other surfaces, according to the MFS property f_{max} . The lack of a relationship between f_{max} and the white layer thickness, suggests that the BN- f_{max} method is not solely sensitive to a property of the white layer itself, such as grain size. A small grain size would increase the BN, due to the increased grain boundary area which causes more pinning [219] and thus a greater jump in magnetisation following de-pinning. However, the white layer occupies only a small fraction of the BN inspection volume, so grain size cannot be solely responsible for the change in f_{max} . By comparing f_{max} against key properties of the machined surface, it was shown that the hardness of the material in the BN inspection volume and the surface roughness were not significantly influencing the BN measurements.

Surfaces with a white layer showed a high surface roughness due to the artificially worn inserts used in the trials. When using these worn inserts a significant level of smearing of the workpiece material in the feed direction occurred, as highlighted in Figure 8.41 (a), due to the friction between the workpiece and the large wear land on the ground inserts. This resulted in large, regular deviations of the machined surface profile from the mean line. Smearing did not occur to noticeable levels for the unworn inserts, as shown in Figure 8.41 (b). The occurrence of high surface roughness alongside white layer formation and the absence of a significant relationship to the value of f_{max} indicated in Figure 8.40 (a) suggests that the surface roughness is not significantly influencing the BN- f_{max} measurement.

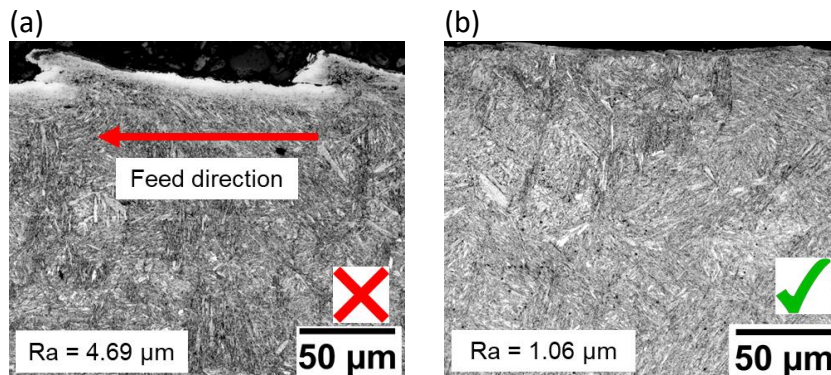


Figure 8.41 – Cross-sectional micrographs showing the contrast in surface topography for surface machined with (a) a worn insert and (b) an unworn insert. The surface in (a) would fail SI inspection due to the roughness and the white layer.

The ability of BN inspection to detect hardness changes relies on compositional change [209], so the absence of a relationship between mechanical hardness in the white layer and f_{max} in these turned SCMV

samples is unsurprising. This is because the phase composition of the white layers in these samples is unchanged, as shown previously in Figure 6.5 (c).

Of the three mechanical and topological properties investigated, the average residual stress in the BN inspection volume had the most significant effect on the BN measurements, as shown in Figure 8.40 (c). A larger compressive residual stress state resulted in a larger measured f_{max} . This observation aligns with the ferromagnetic domain theory outlined in the previous chapter. The decrease in f_{max} for surfaces with a larger compressive residual stress can be attributed to the suppression of high-frequency BN. For materials with positive magnetostriction, such as most iron-carbon alloys [199], the energy required for a domain wall to overcome de-pinning, and thus generate BN, is greater under a compressive stress state [201]. The effect of this change in domain-wall pinning on f_{max} can be understood by considering the time lag for domain wall motion following a rapid increase in magnetic field strength as described previously in Eq. 4.5.

The time lag for wall motion increases with the domain wall activation energy, E_{WM} , and this is larger when the material is under a compressive residual stress state, for a material with a positive magnetostriction constant. As such, at higher magnetic field frequencies, when there is less time for domain walls to move before the field reverses, BN is reduced for a compressive residual stress state, as a larger number of domain walls do not move during a magnetizing cycle due to the increased time lag. This suppression of high-frequency BN, which is apparent from the curves shown in Figure 8.36, gives rise to a decrease in the f_{max} .

The compressive residual stress state also influences the f_{max} measured at different BN filter frequencies. The increased percentage difference in f_{max} between samples with a thick white layer and a thin or no white layer at lower filter frequencies, as shown in Table 8.8, can be attributed to the depth to which residual stress is observed. In surfaces with a white layer the compressive residual stress state extends beyond the BN inspection depth at all filter frequencies, to depths of up to 800 μm , as shown previously in Figure 6.20 (a). By contrast, in samples with a thinner white layer or no white layer, the compressive residual stress decays at a greater rate with depth and reaches values close to zero within 200 μm . Therefore, the difference in residual stress between sample types over the whole BN inspection

volume is greater at lower filter frequency, as the BN interaction volume is inversely proportional to the filter frequency.

The BN- f_{max} measurement using commercially available BN equipment presented here adds to the previous BN study [213], highlighted in the literature review, which also investigated white layer detection. This is because the BN- f_{max} can be used to identify anomalous surfaces without the need for custom laboratory-based equipment. Additionally Stupakov et al. investigated only phase transformation white layers [213], whereas the BN- f_{max} method has been shown to facilitate the identification of surfaces containing a white layer formed by the SPD mechanism. It is difficult to directly compare the resolution capability of the BN- f_{max} method for white layer detection with the previous work, as Stupakov et al. [213] did not quantify white layer thicknesses for each sample tested. From the plots presented in [213], it cannot be said whether the white layers were thin or not present, at low levels of tool wear. The BN- f_{max} method could be argued to possess a resolution around 2 μm , due to the ability to distinguish white layers of this thickness from all other samples. However, it should be considered that the technique was sensitive to the residual stress rather than an intrinsic property of the white layer and as such, the true resolution, in terms of the interaction of the magnetic field with the test piece material is related to the BN inspection depth, which is approximately 50-75 μm at the highest filter frequency.

This lack of sensitivity to an intrinsic feature of the white layer means that the BN- f_{max} method is less optimal than the method introduced by Stupakov et al. [213], if it can be proven that their method is sensitive to SPD white layers too. However, the properties that were said to facilitate white layer detection by Stupakov et al. [213] were the presence of retained austenite and a change in magnetic hardness. These features are not intrinsic to the SPD white layers generated here in SCM-V, so detection of SPD white layers may not be achievable using the method of Stupakov et al. [213]. Due to its reliance on the contribution of residual stress to the resultant signal rather than a fundamental property of the white layer itself, the BN- f_{max} method has limited applicability as a general test for white layers. However, it may be applicable in certain situations, such as that presented in this study when machining with worn inserts but the method cannot be recommended for white layer detection when compared with the XRD- ΔPB , due to its limitations to ferromagnetic alloys.

8.5 Comparison of NDT methods to the existing destructive inspection method and other NDT methods

The current SI inspection process used in industry involves destructive sectioning of machined surfaces followed by optical microscopy [160]. For white layers thinner than 5 μm , higher resolution SEM imaging is undertaken to verify the presence of a white layer, up to a minimum detectable thickness of around 1 μm . These detection limits arise due to the effect of edge-rounding during sample preparation, inconsistent etching and edge-effect during SEM imaging.

XRD- Δ PB measurements can be used to detect white layers in all machined surfaces examined in this study, regardless of the alloy under inspection. The results from the inspection of EP samples with artificially thinner white layers suggest it is possible to detect white layers that are smaller than 5 μm . Mathematical modelling of the technique suggests a minimum detectable thickness of between 1-2 μm depending on the aeroengine alloy under inspection. Consequently, XRD offers an improved resolution to the current, destructive, optical microscopy inspection method. The technique may even be able to offer a similar resolution capability to a SEM, if the tests can be carried out on a greater number of as-machined surface samples possessing a thin white layer. It is not possible to size white layers using the proposed XRD- Δ PB methodology; however, a white layer is not tolerated at any thickness on critical aeroengine components so sizing is not a priority. Consequently, XRD offers a rapid and non-destructive pass/fail inspection alternative for white layers without a compromise in resolution when compared with current destructive techniques used in industry.

It should be noted that the surfaces examined in this study had a simple geometry, being either flat or cylindrical with a radius of curvature above 60 mm. Most surfaces in an aeroengine, including the shafts, discs and fan blades, are flat or have a large radius of curvature and as such, the technique is immediately applicable. However, in surfaces such as drilled holes and in corner radii, the radius of curvature will be smaller and surface access will be an issue. It is to be expected that as the radius of curvature decreases, for the same x-ray illumination area on the sample, the instrument peak broadening will increase, possibly to the point of masking the peak broadening due to the white layer. This is because, for tight radii, the curvature of the surface will increase the range of diffractometer angles for which a proportion of the incident x-rays are at the required angle, relative to the tangent of the surface at a point around the radius,

for coherent interference. However, to combat the effect of sample curvature, a smaller illumination area could be used with the penalty of increased measurement times.

SRAS in its current form does not offer true non-destructive inspection, however, as highlighted previously, SRAS inspection without the requirement for a polished surface is feasible with appropriate advances in equipment. If truly non-destructive SRAS can be realised, then this test may offer an alternative to the XRD- Δ PB method for white layer inspection. However, it is apparent that improvements to the depth resolution capability using smaller wavelengths, without a concurrent, significant increase in the signal-to-noise ratio, are necessary to be able to inspect white layers of the order of several micrometres in thickness. SRAS may be more applicable as a general test for poor SI, due to its sensitivity to regions of swept grain material. In swept grain samples the deformation of near-surface material under the action of machining is directly detectable through the appearance of semi-circular features in velocity maps.

Based on the results presented, the BN- f_{max} method of white layer detection offers improved resolution compared to optical microscopy in that some white layers smaller than 5 μm can be detected. However, the lack of sensitivity to an intrinsic property of the white layer means the method cannot be recommended as a test for white layers due to the potential for false positives and false negatives.

Compared to the optical scattering NDT method for white layer detection that was identified in the literature review [238], the XRD- Δ PB method offers more reliable non-destructive testing across different materials. The optical scattering method has only been applied to four samples in one material, so it requires many additional measurements to be validated. Additionally, there is no key feature of the white layer, that can be attributed to its detection using optical scattering, indicating that the results may be influenced by a secondary feature not accounted for in the study [238]. Both XRD and optical scattering are non-contact methods, however, XRD requires shielding due to the use of ionizing radiation, unlike optical scattering, so it would be more difficult to implement on-machine.

The use of AET for monitoring white layer formation during machining [68] was highlighted in the literature review. AET offers a different type of solution to white layer detection than the other NDT methods investigated in that it is only an in-process method and therefore cannot be applied to components after machining. As such, AE monitoring equipment would be required for every machining

centre, whereas the XRD- Δ PB method offers the option of inspection of surfaces from different machining centres in a dedicated testing cell if in-process monitoring is not a priority. Further work is required to validate the use of AET for white layer detection in different machining operations, as the previous study only investigated turning [68], where AE signals are less complex than for milling or grinding. More information is required to assess the impact of material type, surface roughness and tool edge sharpness on the ability to use AET for white layer detection. Despite the current shortcomings of AET, the use of XRD- Δ PB post-machining in combination with AET for condition monitoring in-process may offer the optimum solution for white layer detection in a smart factory. This is because these methods could provide data on the surface condition throughout the machining operation as well as a definitive decision on the SI of the component after machining.

Although not investigated experimentally in this study, the potential for the use of PAS for white layer detection was highlighted in the literature review. Previous studies [142, 239] have shown that PAS is sensitive to general machining damage, however, the XRD- Δ PB method allows detection of white layer specifically and also appears to be sensitive to swept grain material. It is apparent that both PAS and XRD- Δ PB are sensitive to an enhanced number of dislocations in the machined surface, through broadening of the gamma-ray spectrum and lattice strain peak broadening, respectively, however the x-ray method is also sensitive to grain size, an intrinsic property of the white layer that is not shared with the swept grain material. Therefore XRD is more applicable to the specific detection of white layers. It should be noted that x-rays can have a much lower penetration depth than that of positrons, [239] so they are more suited to the detection of thinner surface layers. Additionally, portable XRD kits are commercially available and can be lower cost than PAS equipment. Both techniques require shielding, however, the gamma photons produced in PAS are more ionizing than x-rays, due to their higher energy and therefore PAS requires more shielding. As such, from the results presented for the XRD- Δ PB method and the limitations identified for PAS, it is predicted that PAS will not be able to offer improvements over the XRD- Δ PB technique.

To facilitate a simple comparison between the destructive and non-destructive white layer detection methods discussed in this section, the penetration depth and the minimum detectable white layer thickness for each technique are shown in Table 8.10.

Table 8.10 – The penetration depth of different white layer detection methods and predicted minimum detectable thickness.

Technique	Penetration depth (μm)	Minimum detectable WL thickness (μm)	Comments
Optical microscopy	N/A cross-sectional	5 μm	Destructive test, limited by etching quality
SEM	N/A cross-sectional	1 μm	Destructive, limited by the edge-effect
XRD-ΔPB	5-10 μm	1-2 μm	Larger penetration depths achievable with different x-ray radiation
XRD-ΔIR	5-10 μm	5 μm	Applicable to fewer materials than the ΔPB method
SRAS	24 μm	5 μm	Detection of white layers cannot be separated from swept grain.
BN- f_{max}	50-75 μm	2 μm (<50 μm)	Relies on detection of residual stress state rather than directly detecting white layer.
Optical scattering	N/A top surface	Unknown	Not enough evidence of capability
PAS	29-159 μm	<29 μm	Penetration depth depends on positron source.
AET	μm - mm	5-10 μm	Penetration depth depends on filter frequency used.

8.6 Concluding remarks

Following the analysis of the applied techniques in the detection of white layers in aerospace alloys, the following conclusions can be made. XRD-ΔPB measurements can be used to detect surfaces with a white layer in all three aeroengine materials and should be applicable to a white layer formed via SPD or phase transformation. The technique possesses the highest reliability and resolution capability of all the techniques investigated and may be capable of detecting layers as small as 1-2 μm, although, it cannot be used to size white layers at this point. The XRD-ΔIR method is only applicable to white layers formed in Ti-6Al-4V and IN-718 via the SPD mechanism and as such offers no improvements over the XRD-ΔPB technique. SRAS can be used to detect Ti-6Al-4V surfaces containing a white layer through an increase in propagation velocity and the absence of macrozones observable in velocity maps for these surfaces. The resolution of this method is lower than XRD, due to the larger inspection depth, and detection through velocity measurement may not be appropriate for inspecting other aeroengine alloys, as the basal texture of the white layer is only seen in HCP alloys. The BN MFS inspection method allows surfaces with thick white layers to be distinguished from all other surfaces in SCMV, however the method is sensitive to residual stress state in the machined near-surface region, rather than an intrinsic property of the white layer. Additionally, the technique is only applicable to ferromagnetic materials, so it offers no improvements over the XRD-ΔPB technique.

9 Conclusions

In this study, the key properties of machining induced white layers have been investigated using materials characterisation methods. Based on the measured properties, several non-destructive evaluation methods have been proposed, which are known to be sensitive to variations in the key properties associated with white layers and the surfaces containing these features. A novel non-destructive evaluation method for detecting white layers in aeroengine materials, using x-ray diffraction, was developed. Additional work also highlighted the capability of spatially resolved acoustic spectroscopy and Barkhausen noise inspection for the detection of anomalous machined surfaces.

The key findings of the work, which can be broken down into two topics, white layer characterisation and white layer detection, are summarised below.

9.1 White layer characterisation

- White layers in Ti-6Al-4V are formed by severe plastic deformation with continuous dynamic recrystallisation when machining with worn inserts due to the high strain rates imposed during machining and the high strain (typically exceeding 0.7 true strain) imparted into near-surface by the cutting tool.
- White layers in titanium, formed under the applied machining conditions, are composed of ultra-fine equiaxed grains with an average diameter of less than 100 nm. Both the α and β phases are present.
- Large compressive forces when machining with worn inserts lead to a strong basal texture in Ti-6Al-4V white layers. This basal texture arises due to activation of the $\{10\bar{1}2\}$ tensile twinning mode and this results in a change in the elastic modulus of the white layer, due to the elastically anisotropic HCP structure.
- White layers formed when machining IN-718 and SCM435 with worn inserts possess a similarly ultrafine grain size, increased hardness, and compressive residual stresses. However, no distinct change in crystallographic texture was recorded relative to a surface with good surface integrity.
- The white layer is up to 15%, 25% and 45% harder than the bulk material in Ti-6Al-4V, IN-718 and SCM435 respectively.

- XRD phase analysis suggests that the white layers observed in this study did not form via an $\alpha \rightarrow \beta$ phase transformation mechanism in titanium, austenitisation in SCMV or phase transformation in IN-718.
- Residual stress profiling measurements beneath the machined surface indicate that there is no single residual stress level that can be associated with the presence of a white layer in Ti-6Al-4V, however, in general, a compressive residual stress beneath the white layer was always present. By contrast, in SCMV a large compressive residual stress was measured for all surfaces with a white layer.
- Measurement of strain in the machined surface has shown that white layer formation is enhanced at higher levels of tool wear due to the rubbing effect of the flank wear land and the ploughing effect of a larger cutting edge radius.
- A white layer was only formed in regions, during orthogonal milling of Ti-6Al-4V, where the cutting edge radius was larger than the instantaneous chip thickness. Plastic strain was particularly large for the case of rubbing with zero chip thickness.

9.2 White layer detection

- White layers can be reliably detected through x-ray diffraction peak breadth measurements. The small crystallite size and inhomogeneous lattice strain in a white layer give rise to a significant broadening of XRD peaks, compared to samples with good SI.
- Peak breadth is 40-85% larger, depending on the alloy under inspection, for surfaces containing a white layer when an appropriate diffraction peak is selected.
- By setting a threshold peak breadth value for a particular material, surfaces with a white layer can be identified without the need for operator interpretation and with an inspection time of under 5 minutes, considerably shorter than the current destructive inspection process.
- For white layers thinner than the depth of penetration of x-rays in the sample, the peak breadth is reduced due to a contribution from the material beneath. A mathematical model was created to predict a minimum detectable white layer thickness of 1-1.6 μm for the three aeroengine alloys investigated.

- The similarity, in terms of the key properties, between machining-induced white layers and those in other fields e.g. the rail industry, means that the peak breadth method has applications beyond this project's scope.
- White layers in Ti-6Al-4V and IN-718 could also be detected through XRD- Δ IR measurements, due to changes in crystal orientation relative to a surface with good SI. However, this crystallographic texture is not a fundamental property of the white layer, given that white layers formed by phase transformation or different machining methods may have a different texture, meaning this XRD method is less widely applicable.
- Spatially resolved acoustic spectroscopy can be used to identify surface with poor SI in Ti-6Al-4V due to the destruction of macrozones relative to a surface with good surface integrity.
- The average SAW propagation velocity for a surface with a poor SI was greater than for a surface with a good SI, this was attributed to the basal orientation of white layers.
- SRAS is less sensitive to white layers than XRD peak broadening, although it offers potential to detect swept grain due to the destruction of macro zones and the presence of arcs in the velocity map, which result from the deformation introduced during machining.
- Magnetizing frequency sweeps using Barkhausen noise testing equipment could be used to segregate ferromagnetic samples with a white layer thicker than 2 μm from all other samples.
- This detection was facilitated by the compressive residual stress state in the inspection volume, for samples with a white layer. As such, the technique was not detecting a fundamental property of the white layers themselves, so it has limited applicability when compared to the XRD peak breadth method.

10 Future work

In this chapter, areas for further research are suggested based on the findings of this project. Topics for further work have been divided into three categories; white layer characterisation, white layer detection, and detection of other aspects of surface integrity.

10.1 White layer characterisation

- In this study, only white layers formed through the SPD and dynamic recrystallisation method were generated, despite attempts to generate phase transformation white layers through machining at high cutting speeds with new inserts. It would be appropriate to investigate the cutting conditions required to form a phase transformation white layer in titanium if such a mechanism is possible for this material.
- The success of the micro-grid strain analysis experiments was limited by the comparatively thin white layers that were generated when compared to the pitch of the micro-grids. Therefore, it would be appropriate to carry out strain measurements using the micro-grid method for another alloy, such as IN-718, that tends to form thicker white layers. This would facilitate higher resolution measurements of plastic strain, due to the comparatively large thickness of the white layer that can be generated relative to the micro-grid size.
- Attempts should be made to quantify the grain size, using TEM, for white layers having undergone heat treatments. This would improve the understanding of the relative weightings of grain size and lattice strain effects when using the XRD- Δ PB method.

10.2 White layer detection

- Attempts were made to source machined surface samples from industry, to validate the XRD method with samples from real-world components, however, these could not be obtained. It is therefore important to work with industry to acquire such samples, as this validation will be required before any adoption of the technique.
- In combination with this testing of real-world components, parts with a small radius of curvature should be tested to establish the capability of the XRD- Δ PB method with respect to sample curvature.

- Electropolishing was used to create thinner white layers to allow the effect of white layers thinner than the XRD inspection depth to be determined. However, electropolished surfaces are not representative of as-machined surfaces, so to validate the method for the detection of thin white layers, additional machined samples with thin white layers should be generated and tested. This could be achieved by machining with tools with lower levels of artificial wear, or by machining with new inserts up until tool failure and extracting test specimens at regular intervals as the tool wears.
- Only SPD white layers were tested with the XRD- Δ PB method in this study, due to the lack of phase transformation white layers formed during the machining trials. Future work should look to validate the technique on thermal white layers.
- White layers, or features that exhibit similar properties but possess a different name, are also found in the rail industry in rail wheels and tracks, wire electric discharge machined surfaces and components produced through shaping and forming operations. The applicability of the XRD peak breadth method for the inspection of these surfaces should be validated, particularly in the rail industry where white layers can lead to fatigue failure of the rail or wheel.
- Grazing incidence XRD peak breadth measurements could be investigated as a higher resolution, but slower, NDT method that may show increased sensitivity to thin white layers, due to the thin penetration depth when compared to conventional Bragg-Brentano geometry XRD used in this study.
- Due to the current limitations of the SRAS equipment, it was not possible to achieve high-quality scans for SAW wavelengths smaller than 24 μ m. SRAS scans of white layers with a smaller wavelength should be carried out when the SRAS equipment is capable of such measurements. With smaller wavelengths, the signal from the white layer will be enhanced since this feature will make up a larger proportion of the SAW interaction volume, meaning it should be easier to detect white layers.
- Similarly, the current roughness limitations for the SRAS equipment meant the technique was not truly non-destructive due to the need for a mirror surface finish, which required the removal of a portion of the white layer, hence making the feature harder to detect. Recent developments in SRAS laser detection hardware have facilitated measurements on rough surfaces, so existing samples should be re-inspected on this equipment.

- SRAS inspection of steel and nickel superalloy samples with a white layer would provide insight into the broader applicability of the technique.

10.3 Detection of other surface integrity features

- Preliminary analysis of industry-supplied Udimet-720 samples that contained a swept grain region, but not a white layer, indicated that peak breadth increases with the maximum depth of swept grain in this material. As such further work should attempt to detect swept grain in addition to white layers. Swept grains are the other key SI feature, aside from white layers, which requires parts to be destructively sectioned, for SI inspection. If swept grain could be detected, it may be possible to circumvent destructive inspection.
- The relative intensities of different XRD diffraction peaks may allow the detection of as-machined surfaces containing high levels of swept grain due to possible subtle changes in the crystallographic orientation or the strength of the shear texture.
- Increased sensitivity to swept grains through XRD may be afforded by a machine learning approach, looking at the features of multiple XRD peaks.
- A similar investigation of swept grain surfaces with SRAS would also be appropriate due to the ability to resolve a feature such as cutting arcs in SRAS velocity maps, which directly evidence grain distortion.

11 Industrial recommendations

11.1 White layer characterisation

The investigation into the physical properties of white layers in Ti-6Al-4V has shown that a sub-100 nm grain size, an increased hardness relative to the bulk microstructure and compressive residual stresses can be associated with this feature. These properties are similar to those previously identified for machining-induced white layers in steels and superalloys when SPD and recrystallisation was the primary mechanism. Additionally, Ti-6Al-4V white layers were found to possess a compressive texture but their formation could not be avoided by altering the initial workpiece texture. White layers were only formed when machining with tools beyond existing tool life criteria, as such, machining using tools within current thresholds for flank wear and cutting edge radius will prevent the systematic formation of white layers. However, with a drive to enhance productivity, these limits may be pushed, so the ability to detect the features non-destructively becomes increasingly important.

11.2 Non-destructive white layer detection

Inspection of machined surfaces using the XRD- Δ PB method allows white layers to be detected in all aeroengine alloys, due to the sensitivity of the diffraction peak breadth to the small grain structure and inhomogeneous lattice strain in the white layer. The XRD-IR method also facilitates the detection of machining-induced white layers, however, it is only applicable to Ti-6Al-4V and IN-718, with the method unable to detect a white layer in SCMV. As such, the XRD- Δ PB method is recommended over the XRD-IR method, due to its lack of material restrictions. It has already been shown that the XRD- Δ PB method can be carried out in a truly non-destructive manner, without any sectioning of the component required. It should be noted that x-ray methods use ionizing radiation so they require the implementation of shielding.

It has been demonstrated that SRAS measurements can be also used to detect anomalous Ti-6Al-4V surfaces containing a white layer and a large swept grain region. However, several limitations should be addressed before this method can be adopted in industry as a SI inspection technique. The SRAS technique used in this study can be defined as a pseudo-NDT technique, as the surface roughness features must be removed through polishing to be able to obtain a good quality measurement. As highlighted previously, the inspection of rough surfaces using SRAS has been developed [195], however, currently the inspection time for rough surfaces is significantly longer, due to the added complexities. Even on polished surfaces,

SRAS scan time is much longer than the inspection time for the XRD method, largely because in SRAS a velocity map is captured by raster-scanning over the surface, whereas during the XRD- Δ PB method the data is captured for the full area under inspection at once. Currently, the SRAS technique is less robust than XRD as it is sensitive to subtle variations in the set-up, which can result in poor quality scans. The SRAS method, however, does offer increased potential for sizing white layers and the swept grain region, compared to XRD, due to the ability to change the SAW wavelength to examine a range of different volumes of material. Additionally, SRAS is already a semi-automated inspection process, with the workpiece raster-scanned beneath the laser using a controllable stage. As such, this method has the potential to be implemented in a machining centre to facilitate on-machine inspection. It should be noted that due to the high-energy laser used to generate the SAW, laser shielding is required for SRAS.

The Barkhausen noise f_{max} inspection method facilitates the identification of surfaces with white layers thicker than 2 μm , due to the compressive residual stress state in the samples that have been tested. The dominant effect of residual stress on the Barkhausen noise measurement, over intrinsic properties of the white layer such as grain size, means this technique is a poor candidate as a white layer inspection method. The technique could be expected to produce very different results if there was a tensile residual stress state, as is common for phase transformation white layers. This lack of sensitivity to a key property of the white layer, in addition to the restriction to just ferromagnetic alloys means the technique is not recommended as an alternative to the current destructive inspection process. However, it should be added that BN is the only technique of the three examined here that does not require any form of shielding.

Of all the NDT techniques trialled in the course of this study, XRD- Δ PB offers the optimal solution for detection of machining induced white layer due to its high repeatability, its sensitivity to the intrinsic properties of the material in the white layer and the shorter inspection time relative to the current destructive process. As such, it is the recommended NDT method to take forward for machining-induced white layer inspection in industry.

11.2.1 XRD- Δ PB method validation considerations

To validate the XRD- Δ PB method for industrial implementation, further trials including blind inspection or gauge repeatability and reproducibility (Gauge R&R) studies are recommended. In a blind inspection

trial, several operators are tasked with identifying parts containing anomalous features from a set of samples, without prior knowledge of their SI, as such these blind trials are representative of real-world application of the NDT technique. Gauge R&R studies aim to quantify the variation in the measurement system, from both the instrumentation and the operator point-of-view. Gauge R&R studies, by necessity, involve blind trials, which assess reproducibility, i.e. consistency between operators, but expand on them to assess instrument variation, i.e. the repeatability.

It is expected that the XRD- Δ PB method for white layer detection should perform well when assessed in a Gauge R&R study, as no operator interpretation of the results is required; the peak breadth either exceeds the defined threshold value or it does not. Currently, the mounting of the sample and test setup is the main source of variation in repeated measurements in a large diffractometer like the Xpert³ or portable kit such as the iXRD, but variations can be minimised through the adoption of a standard operating procedure. A full probability of detection study is not necessary for validation of the technique, providing equivalency can be shown between the capabilities of the NDT method and the current destructive method of inspection.

11.2.1.1 Proposed inspection procedure

To simplify the inspection process, a table of threshold peak values for the alloys of interest on the chosen XRD equipment is required. These thresholds would be selected from measurements on a baseline set of samples, similar to the measurements carried out in this study. If inspecting a different alloy of the same type of material, for example, Ti-834 when data for Ti-6Al-4V exists, then a sample set containing at least 3 machined surfaces with a white layer and 3 without a white layer may be sufficient, however, larger sample sets are desirable if possible. This data set would ideally be contained within digital databases embedded within a piece of software that carries out the peak breadth calculation and returns a pass/fail response. A summary of the envisioned inspection procedure is given in Figure 11.1.

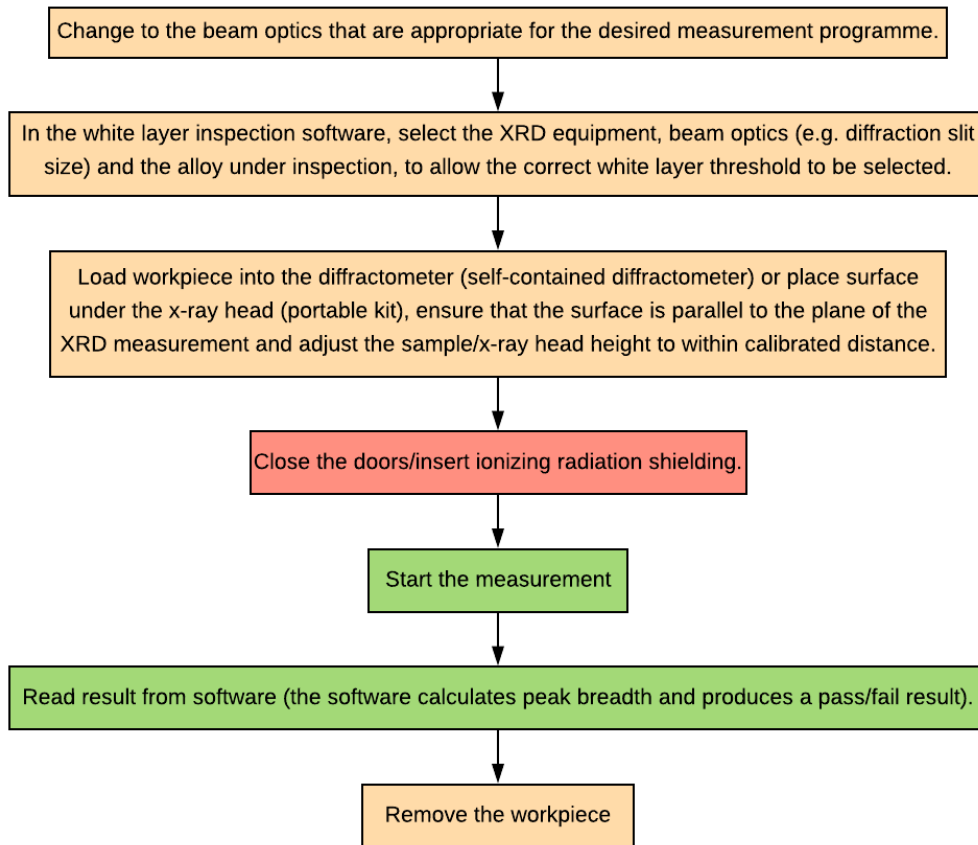


Figure 11.1 – The envisioned inspection procedure for the XRD-APB white layer detection method.

The second step of the proposed inspection procedure will be equipment specific, i.e. the most appropriate method for loading the workpiece and consistently setting up the measurement equipment will become more apparent as the exact equipment is defined. The key process variables, which must be controlled to ensure a predictable and consistent output from the XRD-APB method, can be listed as follows:

- X-ray radiation and accelerating tube voltage and current must be kept constant.
- Beam optics must be unchanged (diffraction and anti-scatter slits).
- Pulse height distribution limits must be consistent for the detector.
- Working distance and tilt of the workpiece surface must be kept constant.
- Consistent post-processing is applied to the raw data before the FWHM measurement.
- The method of FWHM calculation, including any interpolation method, is constant

11.2.1.2 Implementation considerations

Although the underlying theory and proof-of-concept of the XRD- Δ PB method for the detection of white layers have been described in this study, there are further considerations that must be addressed from an industrial perspective, before the technique could be implemented on production components. This is because, an NDT method should be comparable to, or better than, the existing destructive inspection method with respect to a cost/benefit analysis, in order to be implemented. The key comparison considerations are outlined in Table 11.1 for the existing destructive methods of white layer assessment and those of the novel non-destructive method.

Table 11.1 – The key considerations for each method of white layer inspection summarised.

Technique	Optical microscopy	SEM	XRD-ΔPB
Type	Destructive	Destructive	Non-destructive
Contact/non-contact	Non-contact	Non-contact	Non-contact (i.e. not limited by a probe size. Beam area can be changed by altering the size of the beam masks and slits)
Resolution	$\sim 5 \mu\text{m}$	$\sim 1 \mu\text{m}$	$< 5 \mu\text{m}$ ($1\text{-}2 \mu\text{m}$ theoretically possible)
Evidence for capability	Industrial surface integrity inspection	Industrial surface integrity inspection	Measurement on research samples
The physical property(s) that facilitate white layer detection	White layer etches differently to bulk material.	White layer etches differently to bulk material and has a smaller grain size.	White layers possess a small crystallite size and inhomogeneous lattice strain causing peak broadening.
Equipment	Sectioning machine, grinding and polishing machine, fume cupboard (etching), optical microscope, PC.	Sectioning machine, grinding and polishing machine, fume cupboard (etching) - optional, SEM, PC	XRD diffractometer (portable or self-contained), PC
Equipment cost	£10,000's	£100,000's	£10,000's - £100,000's
Health and Safety	Chemical etching	Chemical etching (optional)	Ionizing radiation necessitates shielding
Footprint	Requires a sample preparation laboratory $\sim 16 \text{ m}^2$.	Requires a room for the SEM $\sim 8 \text{ m}^2$ and a laboratory for sample preparation $\sim 16 \text{ m}^2$.	Currently requires space for a self-contained diffractometer and cooling $\sim 4 \text{ m}^2$ or portable kit 4 m^2 . The equipment could fit inside a machining centre with further research.

Table 11.1 continues on the next page

Table 11.1 continued

Sample preparation time	2+ hours to take the sample through the micropreparation steps. It can take much longer depending on the sectioning.	2+ hours to take the sample through the micropreparation steps. It can take much longer depending on the sectioning.	None
Sample size/shape limitations	30-40 mm or smaller, requires sectioning to reveal a flat cross-sectional surface	30-40 mm or smaller, requires sectioning to reveal a flat cross-sectional surface	iXRD - no upper limitation, Empyrean – 400 mm upper limit, Xpert ³ – 30-40mm. Custom equipment – no upper sample size limits. Lower size limit is dictated by the smallest slit available to restrict the XRD beam area. Applicability to highly curved surfaces is dictated by the smallest size of the XRD beam area. Access is required to transmit the XRD beam to the surface and receive it at a detector.
Effect of surface roughness	Cross-sectional measurement so unaffected	Cross-sectional measurement so unaffected	Unaffected by the levels of roughness that would allow a part to pass roughness inspection.
Effect of surface contaminant	Cross-sectional measurement so unaffected	Cross-sectional measurement so unaffected	XRD peaks from planes corresponding to a surface contaminant are likely to be at a different diffraction angle to the PB measurement. If the contaminant is thicker than the XRD penetration depth in the contaminant, then white layer detection will not be possible.
Measurement time	5 minutes	30 minutes	< 5 minutes. Measurement time will increase with a smaller XRD beam area, due to the lower diffracted beam intensity.
Analysis time	< 5 minutes	< 5 minutes	< 5 minutes
Overall time from machining to receiving results	1 day – several weeks due to the numerous steps.	1 day – several weeks due to the numerous steps.	< 10 minutes achievable
How is the result analysed?	Requires interpretation from an operator.	Requires interpretation from an operator.	Interpretation only required when defining the thresholds.

The benefits, in terms of inspection time and resolution, have been discussed previously but additional considerations such as the equipment cost and footprint, outlined in Table 11.1, have not been introduced. The predicted equipment cost of XRD is greater than that for optical microscopy, however, most XRD equipment is designed for laboratory research, so it contains many advanced features unnecessary for peak breadth measurements. An XRD testing kit designed solely for white layer detection, without the usual additional functionality, could be made for a lower cost, however, it may still not compete directly with optical microscopy on equipment costs. Further work, in collaboration with an XRD manufacturer, would be required to develop such a bespoke machine. In addition to equipment costs, the floor-space and safety features required for the equipment must be considered. XRD requires shielding to prevent users from ionizing radiation; self-contained diffractometers are fully shielded but portable diffractometers require portable shielding or protection of the room in which they are located. If the technique was implemented in a machining centre, lead-lining or equivalent shielding within the machine would be necessary to allow the equipment to be utilised on the factory floor.

Depending on the XRD equipment, the XRD- Δ PB method is subject to sample size limitations. Enclosed diffractometers, such as the Empyrean are restricted to the inspection of workpieces that can fit within the confines of the machine. By contrast, a portable kit such as the iXRD can be used on any workpiece, providing the surface can be located in the path of the beam. It should be noted that the inspection of drilled holes with this method would be difficult due to the need to transmit the XRD beam to the machined surface and receive the diffracted beam. The XRD- Δ PB method is not sensitive to levels of surface roughness on aeroengine components that would pass roughness inspection but the technique may be sensitive to surface contamination, for example rust, if this contaminant were thicker than the penetration depth of x-rays. For thin contaminant layers where the x-rays can penetrate through to reach the base alloy, the diffraction of x-rays in the contaminant is unlikely to occur at the same diffraction angle as the peak of interest in the peak breadth measurement, so white layer detection would still be achievable.

An XRD inspection method could be implemented into a modern manufacturing process in one of the following three ways:

- Automated inspection of the workpiece on-machine, using equipment contained within the machining centre (possibly controlled using the in-built computer numerical control).
- Semi-automated inspection of the workpiece on-machine using a portable kit which is placed on top of the workpiece or reaches into the machining centre using, for example, a robotic arm.
- Semi-automated inspection of the workpiece outside of the machining centre in a dedicated testing cell.

Table 11.2 – The key considerations for the three different options for the implementation of XRD-APB inspection.

Technique	Automated inspection on-machine	Semi-automated inspection on-machine	Semi-automated inspection in a testing cell
Operator required	No	Yes	Yes
Equipment/resources required	Computer numerical control, XRD equipment, shielding.	XRD equipment, Robotic arm, PC, trolley to move between machines, shielding.	Test cell, XRD equipment, shielding.
Research cost	Highest	Middle	Lowest
Location	Contained within a machining centre so no additional floor space required.	If a portable kit is placed in the machine then 1 m ² is needed to store the equipment. 1-4 m ² would likely be required if a small robotic arm was used.	Requires a separate testing cell 5-10 m ² on or near the factory floor.
Units required	1 unit per machining centre.	1 unit could be used in multiple machines.	1 unit could be used to test parts from different machines.
Measurement time (including set-up)	Shortest	Middle	Longest
Lights-out process	Yes	No	No

The advantages and limitations of these methods are compared in Table 11.2. The first proposed inspection method is the only option that allows for a truly automated inspection process and thus has several benefits over the semi-automated processes, which require an operator to correctly position the XRD kit or move the workpiece. Automated non-destructive SI assessment could be readily implemented as part of Industry 4.0 concepts in a smart factory. By carrying out surface assessment non-destructively

without operator input, the manufacturing route from machining through to part validation can be accelerated and carried out as part of a lights-out production method. This automation can be facilitated by building a digital twin of the component, using information from sensors during the machining and subsequent non-destructive assessment. The digital twin can be used to make decisions on what further machining is required and whether the component passes SI inspection or not.

11.3 Strategies to increase the sensitivity of the technique

To further improve the sensitivity of the XRD- Δ PB technique to facilitate the detection of thinner white layers than predicted from the mathematic model, the use of grazing incidence XRD geometry can be used to restrict the inspection depth to a thin surface region ($< 1 \mu\text{m}$). If the incident angle is below the critical angle for the material being inspected, then total external reflection occurs and the penetration depth of the x-ray beam will be of the order of nanometres [289]. The use of incident angles greater than the critical angle but smaller than 5° will facilitate inspection beneath $1 \mu\text{m}$, thereby offering increased resolution over the existing technique. However, this increase in resolution typically comes at the cost of longer scan times. Additionally, because the incident beam is at a lower angle, the illumination area on the sample will be increased, for the same beam mask, relative to conventional XRD. Therefore, to inspect a smaller region, narrower diffraction slits are required and this will further increase inspection time.

Another strategy to increase the sensitivity of the technique is to expand the peak breadth analysis to multiple diffraction peaks, rather than relying on a single peak. Individual peaks, when employing conventional XRD geometry, correspond to different incident beam angles and therefore slightly different penetration depths. By using a machine learning pattern recognition approach, it may be possible to predict the size of any white layer by comparing peak breadth across a full diffraction pattern. Again, it should be noted that obtaining a full diffraction pattern will considerably increase measurement time relative to the inspection of only one peak. The anisotropic peak broadening measured in this study would have to be investigated further to be able to utilise multi-peak analysis.

11.4 Other aspects of surface integrity

Although it has been shown that white layers can be detected non-destructively, other aspects of SI can still only be detected through cross-sectional microscopy, so components will require destructive characterisation to capture these other aspects of SI. It could be argued that swept grains are just as critical

as white layers, as they also necessitate destructive cut-ups during the inspection process and are critical to the assessment of machined surface integrity. Features such as alpha-case, in titanium alloys, and strain banding, while detrimental to functional performance, are either material-specific or lead to fewer inspection fails and, as such, are a lower priority SI feature. Additionally, while features such as cracks are unacceptable in an aeroengine component, they can be readily captured by existing NDT techniques so they are therefore a low priority feature in this assessment.

A swept grain layer is formed to some level in all chip-forming machining operations, regardless of the workpiece material, due to the influence of the primary shear zone, which extends beyond the tool tip. However, the presence of swept grain is not an immediate SI inspection fail, unlike white layer formation. The pass/fail criterion for the swept grain region is defined by its thickness, on a material and processing route basis. Consequently, any NDT technique must be able to size the swept grain thickness to compete with the current optical inspection method. The accuracy to which swept grain thickness must be measured by NDT is difficult to define. Microscopy measurement of swept grain is highly subjective, as it requires the inspector to judge grain deformation, which typically decays gradually with depth, and distinguish this from the bulk grain structure. NDT measurement of swept grain, which produces a quantitative swept grain thickness based on physical properties, could be a more appropriate method than optical inspection if operator interpretation is removed from the inspection procedure.

The NDT techniques that may be suitable for swept grain detection are XRD, USAWT and PAS. It has already been shown, from preliminary results, that swept grain may be detectable by the XRD- Δ PB method, at least in a nickel superalloy. Further work would be required to investigate whether this technique can size the feature, or if it would simply facilitate a present/not present result, similar to the inspection white layers. Swept grain material, has typically not undergone phase transformation or recrystallisation, as with SPD white layer, so crystallite broadening will not be present due to the larger grain size in swept grain (>500 nm). However, peak broadening due to lattice strains could be large enough to observe peak broadening. The ability to size a swept grain region will be reliant on the gradient of the inhomogeneous lattice strain beneath the machined surface increasing for thicker swept grain regions. This is because the acceptable thicknesses of swept grain layers are typically greater than the XRD penetration depth in most alloys.

Surface acoustic wave inspection methods, specifically SRAS, could be a more feasible option for NDT and sizing of swept grain material. It has already been shown that SRAS is sensitive to swept grain material due to crystallographic texture differences. Additionally, other indicators of surface damage include distorted material visible in SRAS velocity maps as arcs that correlate to the tool path and the destruction of macrozones relative to as-received material. Sizing of swept grain regions with SRAS is more realizable, due to the comparatively large inspection depth which is of the order of the swept grain layer thickness and can also easily be altered by changing the separation of the fringe pattern used to generate the SAW. By comparing the results from scans at different SAW wavelengths, it may be possible to estimate the swept grain thickness.

As highlighted previously, there should be an increased dislocation density in the swept grain region relative to the unaffected material beneath due to the deformation of the workpiece. This increase in the number of dislocations may be detectable using positron annihilation spectroscopy due to the sensitivity of the technique to these lattice defects. The ability to size swept grains will be dependent on the existence of a relationship between the thickness of swept grain, the amount of distortion with depth and the dislocation density. The degree of distortion does appear to decrease with depth and thicker swept grain regions have greater distortion, as shown from the various optical micrographs in this study, so the existence of this relationship would be unsurprising. Previous studies, highlighted in the literature review, have already shown that PAS is sensitive to machined surface damage but the major limitation of this technique is the high cost compared to x-ray and ultrasonic techniques. As such, due to the expertise gained in the application of other NDT methods during the course of this study, it is recommended that XRD and SRAS methods be the focus for future detection of swept grain.

Detection of white layers and swept grain with a single NDT technique is more complex than the detection of just one of these SI features. In the XRD- Δ PB method, the presence of a thin white layer or a swept grain region would both give an increase in peak breadth relative to a surface with good SI, although this increase would be much smaller than for a surface with a white layer thicker than the inspection depth. As such, it may not be possible to detect one feature in particular, rather the technique may only be able to detect a general anomalous surface. The same difficulty in separating the two key features would be expected with PAS. SRAS is the only NDT method that could facilitate the detection of both white layers and swept grain individually, using the same measurement. This is because the presence of a thick white

layer results in an increase in average velocity, whereas the presence of swept grain can be seen through the cutting arcs visible in velocity maps, which indicate distortion of the underlying microstructure.

Non-destructive detection of alpha-case through XRD phase analysis may be possible, if the β -phase is present in sufficient quantities to be detectable in the as-received material, however, further research into the properties of this SI feature is required to make an informed recommendation. PAS may be sensitive to the presence of strain bands, which will coincide with an increase in dislocation density, compared to a sample without strain banding. Finally, redeposited material may be detectable using the XRD- Δ PB method, as this material will have undergone enhanced deformation relative to the material on which it is deposited. However, detection via this method is reliant on raster-scanning the whole component, if the re-deposited material is discontinuous rather than a layer across the whole surface.

To guide future work aiming to offer a fully non-destructive alternative to SI inspection, suggestions have been made in Table 11.3 as to NDT methods that may be applicable for the detection of the key SI features. These suggestions are summarised graphically in Figure 11.2. The features have been assigned a priority based on their impact on the functional performance of a component, the limitations of the inspection method currently used to identify these features and their relationship to other SI.

Table 11.3 – Anomalous surface integrity features and how they can currently be detected, in addition to non-destructive methods that may be applicable for their detection with further work.

Anomalous feature (colour indicates priority)	White layer	Swept grain	Alpha-case	Redeposited material	Laps	Flaking	Plucking	Softening/Hardening	Strain	Cracks	Residual stress	Surface roughness	Foreign material	Scores/Scratches
Currently assessed through destructive microscopy	✓	✓	✓	✓	✓	✓	✓		✓ (strain bands)	✓			✓	
Could be assessed through non-destructive (surface) microscopy (case dependent)					✓	✓	✓							✓
Currently assessed non-destructively								✓		✓	✓	✓		
Could be detectable using the current WL detection form of XRD	✓	✓		✓ (if significantly deformed)										
Could be detectable / measurable using a form of XRD (Phase analysis, texture, peak broadening, residual stress)	✓	✓	✓	✓					✓		✓		✓	
Could be detectable using another form of NDT (based on the knowledge acquired during the EngD)	✓ (BN, USAWT, PAS)	✓ (USAWT, PAS)	(needs literature review)	✓ (USAWT)	✓ (AET)	✓ (AET, OS)	✓ (AET, OS)	✓ (BN)	✓ (PAS)	✓ (UT, UTSAW, DPI, MPI)	✓ (ECT, BN)	✓ (OS)	✓ (ECT, UT)	✓ (OS)

AET – Acoustic emission testing

BN – Barkhausen noise

DPI – Dye-penetrant inspection

ECT – Eddy current testing

MPI – Magnetic particle inspection

OS - Optical scattering

PAS – Positron annihilation spectroscopy

USAWT – Ultrasonic surface acoustic wave testing

UT – conventional ultrasonic testing

Priority

High – This anomalous feature cannot currently be detected except through destructive microscopy and is either widely encountered or highly detrimental to a surface

Medium – This feature is either alloy specific, detectable by methods other than destructive microscopy, or is a secondary feature which is influenced by the presence of the feature with higher priority

Low – This feature is observable through non-destructive surface microscopy or may be picked up during existing NDT inspection (e.g. cracks detected through DPI or MPI)

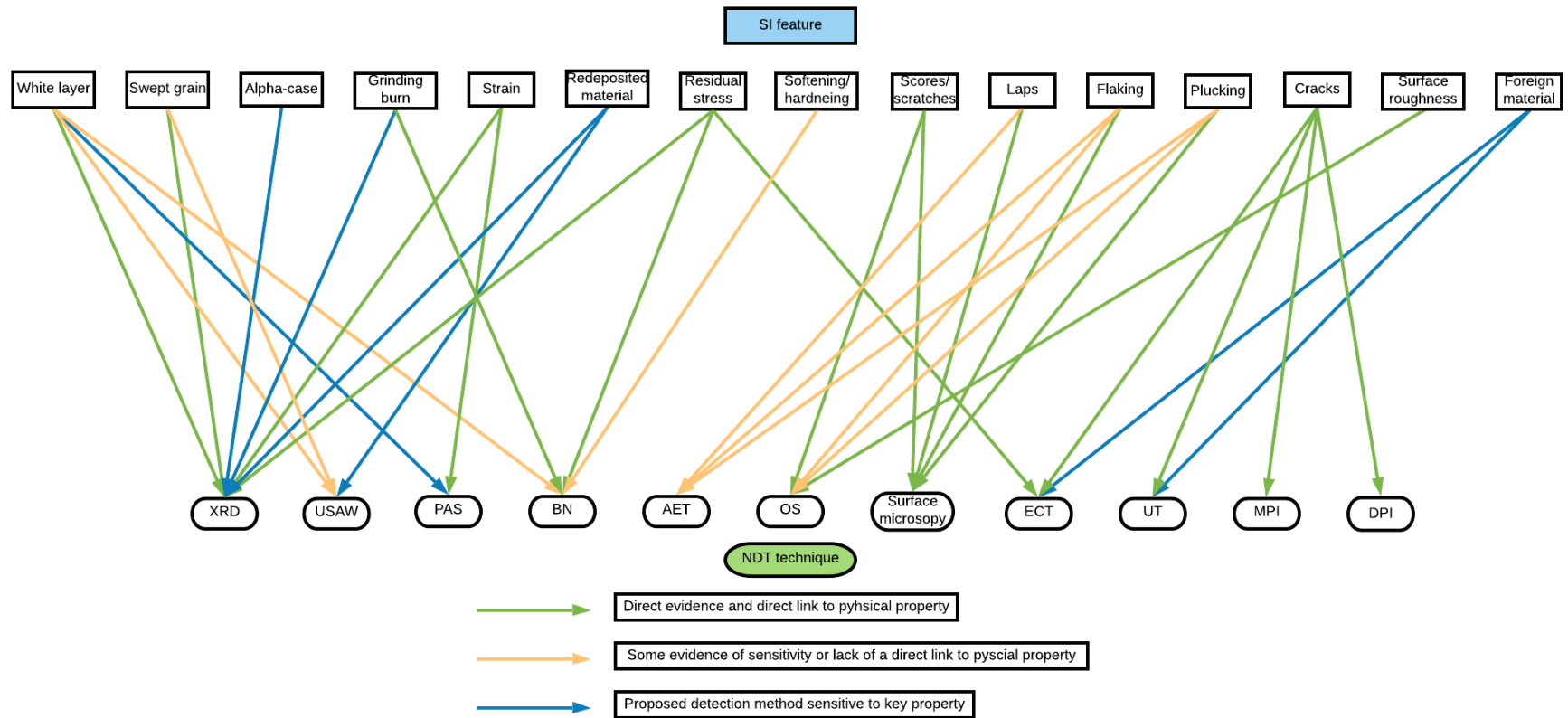


Figure 11.2 – Graphical illustration of the current and proposed NDT methods for assessing surface integrity.

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13 Appendix

13.1 Patent applications

Primary inventor on UK Patent Application No 1906037.5, NON-DESTRUCTIVE DETECTION OF SURFACE AND NEAR SURFACE ABNORMALITIES IN A METALLIC PRODUCT, The University of Sheffield – AMRC, [filed - 30/04/19].

Primary inventor on International Patent Application No PCT/GB2020/050795. NON-DESTRUCTIVE DETECTION OF SURFACE AND NEAR SURFACE ABNORMALITIES IN A METALLIC PRODUCT, The University of Sheffield - AMRC, [filed - 25/03/20].

13.2 Publications

13.2.1 Peer-reviewed journal articles

M. Brown *et al.*, "Quantitative characterisation of machining-induced white layers in Ti–6Al–4V," *Materials Science and Engineering: A*, vol. 764, p. 138220, 2019/09/09/ 2019, doi: <https://doi.org/10.1016/j.msea.2019.138220>

M. Brown *et al.*, "Destructive and non-destructive testing methods for characterisation and detection of machining-induced white layer: A review paper," *CIRP Journal of Manufacturing Science and Technology*, 2018, doi: <https://doi.org/10.1016/j.cirpj.2018.10.001>

13.2.2 Peer reviewed conference proceedings

M. Brown *et al.*, "Non-destructive detection of machining-induced white layers in ferromagnetic alloys", *Procedia CIRP*, vol. 87, pp 420-425, doi: <https://doi.org/10.1016/j.procir.2020.02.065>

13.2.3 Conference presentations

M. Brown *et al.*, "Non-destructive detection of machining-induced white layers in ferromagnetic alloys", 5th CIRP Conference on Surface Integrity, 3rd-5th June 2020, Vitoria-Gasteiz, Spain.

M. Brown *et al.*, "Towards a non-destructive evaluation method for detection of machining induced white layers in steels", EUROMAT 2019 – European congress and exhibition on advanced materials and processes. 1st-5th September 2019, Stockholm, Sweden.

13.3 Machining parameters

Table 13.1 – Cutting conditions for the square shoulder milling trials in Ti-6Al-4V.

Experiment No.	Run order	Cutting Speed V_c (m/min)	Inset wear VB (mm)	a_p (mm)	a_e (mm)	Feed per tooth f_t (mm/tooth)	Coolant
1	19	20	0	2	16	0.12	No
2	1	20	0	2	16	0.06	No
3	11	200	0	0.5	16	0.06	No
4	17	20	0	2	16	0.12	No
5	8	100	0	2	16	0.12	No
6	20	200	0	0.5	16	0.12	No
7	7	50	0	2	16	0.06	No
8	10	60	0	2	16	0.09	No
9	12	100	0	2	16	0.12	No
10	13	120	0	0.5	16	0.09	No
11	4	40	0	0.5	16	0.12	No
12	6	40	0	0.5	16	0.06	No
13	9	60	0	2	16	0.09	No
14	5	200	0	0.5	16	0.12	No
15	3	120	0	0.5	16	0.09	No
16	14	40	0	0.5	16	0.12	No
17	2	20	0	2	16	0.06	No
18	16	40	0	0.5	16	0.06	No
19	18	200	0	0.5	16	0.06	No
20	15	100	0	2	16	0.06	No
21.a	21	200	0	0.5	16	0.06	No
21.b	22	200	0	0.5	16	0.06	No
22.a	23	200	0	0.5	16	0.06	No
22.b	24	200	0	0.5	16	0.06	No
23	25	40	0.8	0.5	16	0.12	Flood
24	40	40	0.8	0.5	16	0.06	Flood
25	33	20	0	2	16	0.06	Flood
26	27	20	0.5	2	16	0.06	Flood
27	41	40	0.5	0.5	16	0.06	Flood
28	36	20	0.5	2	16	0.06	Flood
29	29	20	0	2	16	0.06	Flood
30	26	40	0.8	0.5	16	0.06	Flood
31	38	40	0	0.5	16	0.06	Flood
32	31	40	0.5	0.5	16	0.06	Flood
33	37	40	0	0.5	16	0.06	Flood
34	28	20	0.8	2	16	0.06	Flood
35	30	20	0.8	2	16	0.06	Flood
36	34	80	0.5	0.5	16	0.06	Flood
37	35	40	0.5	2	16	0.06	Flood
38	25	80	0.5	0.5	16	0.06	Flood
39	34	40	0.5	2	16	0.06	Flood
40	32	40	0.8	2	16	0.06	Flood

Table 13.2 – Cutting conditions for the turning trials in SCMV.

Experiment No. / Run order	Cutting Speed V_c (m/min)	Inset wear VB (mm)	Depth of cut w (mm)	Feed per tooth f_t (mm/tooth)	Coolant
1	60	0	0.5	0.15	Jetstream*
2	120	0.25	0.5	0.15	Jetstream
3	60	0.15	0.5	0.15	Jetstream
4	120	0.25	0.5	0.15	Jetstream
5	180	0.5	0.5	0.15	Jetstream
6	180	0	0.5	0.15	Jetstream
7	60	0.5	0.5	0.15	Jetstream
8	180	0.15	0.5	0.15	Jetstream
9	60	0	0.5	0.15	Jetstream
10	60	0.15	0.5	0.15	Jetstream
11	60	0.5	0.5	0.15	Jetstream
12	180	0.5	0.5	0.15	Jetstream
13	180	0.15	0.5	0.15	Jetstream
14	180	0	0.5	0.15	Jetstream
15	120	0.25	0.5	0.15	no
16	180	0.15	0.5	0.15	no
17	60	0.15	0.5	0.15	no
18	60	0.5	0.5	0.15	no
19	60	0	0.5	0.15	no
20	180	0	0.5	0.15	no
21	60	0	0.5	0.15	no
22	60	0.15	0.5	0.15	no
23	120	0.25	0.5	0.15	no
24	180	0.5	0.5	0.15	no
25	180	0	0.5	0.15	no
26	180	0.5	0.5	0.15	no
27	180	0.15	0.5	0.15	no
28	60	0.5	0.5	0.15	no

* Jetstream is the Seco Tools' through-tool high-pressure coolant delivery system.

Table 13.3 – Cutting conditions for the square shoulder milling trials in IN-718.

Experiment No.	Run order	Cutting Speed V_c (m/min)	Insert wear VB (mm)	a_p (mm)	a_e (mm)	Feed per tooth f_t (mm/tooth)	Coolant
1	8	10	0	0.5	16	0.03	Flood
2	9	20	0.5	0.5	16	0.03	Flood
3	1	20	0.5	0.5	16	0.03	Flood
4	10	20	0	0.5	16	0.03	Flood
5	20	10	0	0.5	16	0.03	Flood
6	16	10	0	0.5	16	0.03	Flood
7	7	20	0.5	0.5	16	0.03	Flood
8	17	10	0.5	0.5	16	0.03	Flood
9	6	10	0.5	0.5	16	0.03	Flood
10	14	20	0	0.5	16	0.03	Flood
11	4	20	0.5	0.5	16	0.03	Flood
12	12	10	0	0.5	16	0.03	Flood
13	18	20	0.5	0.5	16	0.03	Flood
14	15	20	0	0.5	16	0.03	Flood
15	5	20	0	0.5	16	0.03	Flood
16	3	10	0	0.5	16	0.03	Flood
17	19	100	0	0.5	16	0.03	Flood
18	13	200	0	0.5	16	0.03	Flood
19	11	150	0	0.5	16	0.03	Flood
20	2	100	0	0.5	16	0.03	Flood

Table 13.4 – Cutting conditions for the effect-of-texture trials in different Ti-6Al-4V workpieces.

Experiment No. \ Run order	Workpiece	Cutting Speed V_c (m/min)	Insert wear VB (mm)	a_p (mm)	a_e (mm)	Feed per tooth f_t (mm/tooth)	Coolant
1	U/D ND	40	0.5	2	16	0.06	Flood
2	U/D RD	40	0.5	2	16	0.06	Flood
3	U/D TD	40	0.5	2	16	0.06	Flood
4	U/D TD	40	0.5	2	16	0.06	Flood
5	Cross-rolled	40	0.5	2	16	0.06	Flood
6	Cross-rolled	40	0.5	2	16	0.06	Flood
7	FAST – radial	40	0.5	2	16	0.06	Flood
8	FAST – axial	40	0.5	2	16	0.06	Flood
9	U/D ND	40	0.5	2	16	0.06	Flood
10	FAST – radial	40	0.5	2	16	0.06	Flood
11	U/D RD	40	0.5	2	16	0.06	Flood
12	FAST – axial	40	0.5	2	16	0.06	Flood
13	FAST – radial	40	0.5	2	16	0.06	Flood
14	FAST – radial	40	0.5	2	16	0.06	Flood
15	FAST – axial	40	0.5	2	16	0.06	Flood
16	FAST – axial	40	0.5	2	16	0.06	Flood

Table 13.5 – Cutting conditions for the Ti-6Al-4V orthogonal milling trials.

Experiment No. \ Run order	Cutting Speed V_c (m/min)	Insert wear VB (mm)	Cutting edge radius (mm)	a_p (mm)	a_e (mm)	Feed per tooth f_t (mm/tooth)	Climb/ conventional milling
1	20	0.265	137	8	0.5	0.5	Climb
2	20	0.265	137	8	0.5	0.5	Conventional
3	20	0	30	8	0.5	0.5	Climb
4	20	0	30	8	0.5	0.5	Conventional
5	200	0	30	8	0.5	0.5	Climb
6	200	0	30	8	0.5	0.5	Conventional
7	20	0.5	86	8	0.5	0.5	Climb
8	20	0.5	85	8	0.5	0.5	Conventional
9	80	0.5	11	8	0.5	0.5	Climb
10	80	0.5	13	8	0.5	0.5	Conventional
11	80	0.5	86	8	0.5	0.5	Climb
12	80	0.5	93	8	0.5	0.5	Conventional
13	20	0.5	13	8	0.5	0.5	Climb
14	20	0.5	13	8	0.5	0.5	Conventional
15	20	0.265	137	8	0.5	0.5	Climb
16	20	0.265	137	8	0.5	0.5	Conventional

13.4 Cutting tools and inserts

Full details of the cutting tool and insert geometry.

Table 13.6 – Details of the cutting inserts.

Insert	Clearance angle (°)	Rake angle (°)	Corner radius (mm)	Insert shape code	Insert hand	Grade	Coated
XOEX120408R-M07 MP2050	15	20.3	0.8	XOEX	Right	Carbide	PVD
DNMG150608-M3 TP2501	0	0	0.8	D	Neutral	Carbide	PVD
TPUN160308 HX	11	0	0.8	T	Neutral	Carbide	No

Table 13.7 – Details of the cutting inserts.

Insert	Rake angle radial (°)	Rake angle orthogonal (°)	Rake angle axial (°)	Tool cutting edge angle	Tool hand
R217.69-2020.0-12-2AN	-11.8	-11.8	2.8	90	Right
R220.17-0040-16	0	0	0	90	Right
PDJNL2020K15JETL	-6	-6	-6	93	Left

13.5 Micro-grid strain measurement theory and image analysis procedure

13.5.1 Deformation theory

To explain how strain is measured from the micro-grids, deformation theory must first be introduced. The change in shape between an undeformed object and a deformed object is described by its deformation. Mathematically, deformation is the rate of change of shape with respect to position, i.e. the gradient of the shape. The deformation gradient $\mathbf{F}$ (Eq. 13.1) describes the rotations and stretches required to transform between the initial and final conditions.

$$F_{ij} = \frac{\partial v_i}{\partial V_j} = \begin{bmatrix} \frac{\partial v_1}{\partial V_1} & \frac{\partial v_1}{\partial V_2} \\ \frac{\partial v_2}{\partial V_1} & \frac{\partial v_2}{\partial V_2} \end{bmatrix} \quad \text{Eq. 13.1}$$

Where, $\mathbf{v}$ is a vector used to describe the deformed condition and $\mathbf{V}$ is a vector used to describe the undeformed condition. For the particular case of strain measurement from a micro-grid, as described by [157], the undeformed ($\mathbf{V}$) and deformed ($\mathbf{v}$) vectors take the form highlighted in Figure 13.1 and Eq. 13.2.

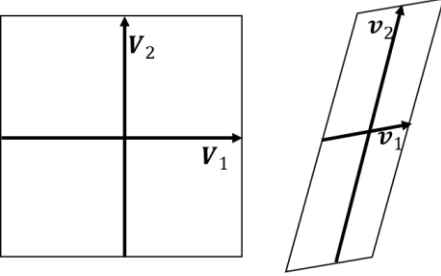
$$\begin{aligned} \mathbf{V}_1 &= A\mathbf{i} + B\mathbf{j}, & \mathbf{v}_1 &= a\mathbf{i} + b\mathbf{j} \\ \mathbf{V}_2 &= C\mathbf{i} + D\mathbf{j}, & \mathbf{v}_2 &= c\mathbf{i} + d\mathbf{j} \end{aligned} \quad \text{Eq. 13.2}$$


Figure 13.1 – Schematic showing the vectors $\mathbf{V}_1$ and $\mathbf{V}_2$ for the undeformed grid, and vectors $\mathbf{v}_1$ and $\mathbf{v}_2$ for the deformed grid.

The deformation gradient maps the undeformed configuration to the deformed configuration, as described mathematically in Eq. 13.3.

$$\mathbf{v} = \mathbf{F} \cdot \mathbf{V} \quad \text{Eq. 13.3}$$

$$\mathbf{F} = \begin{bmatrix} a & c \\ b & d \end{bmatrix} \begin{bmatrix} A & C \\ B & D \end{bmatrix}^{-1}$$

$\mathbf{F}$ can be decomposed into rotation $\mathbf{R}$ and stretch $\mathbf{U}$ components (Eq. 13.4).

$$\mathbf{F} = \mathbf{R} \cdot \mathbf{U} \quad \text{Eq. 13.4}$$

The stretch tensor gives rise to stress and strain due to deformation so it is this component which is of interest in strain analysis, whereas in contrast, rotation involves no change in shape and therefore no strain. Polar decomposition is used to find $\mathbf{U}$, as the transpose of the rotation matrix is equivalent to the inverse, by definition. $\mathbf{U}$ is symmetric, therefore, the stretch tensor can be written as in Eq. 13.5.

$$\begin{aligned}\mathbf{F}^T \cdot \mathbf{F} &= \mathbf{U}^T \cdot \mathbf{R}^T \cdot \mathbf{R} \cdot \mathbf{U}, & \mathbf{R}^T \cdot \mathbf{R} &= \mathbf{R}^{-1} \cdot \mathbf{R} = \mathbf{I} \\ \therefore \mathbf{F}^T \cdot \mathbf{F} &= \mathbf{U}^T \cdot \mathbf{U} \cdot \mathbf{I} = \mathbf{U}^2 \\ \mathbf{U} &= (\mathbf{F}^T \cdot \mathbf{F})^{0.5}\end{aligned}\tag{Eq. 13.5}$$

True strain, ϵ_T , was chosen to represent strain in these trials as it is geometrically symmetric, meaning an extension of x times the original length and a compression of $1/x$, give the same value of true strain, but with opposite signs. It should be noted that engineering strain $\epsilon = \mathbf{U} - \mathbf{I}$, so to find the true strain from the stretch tensor, $\mathbf{U}$ must be diagonalised by calculating principal strains and then the natural logarithm of $\mathbf{U}$ computed. The principal strain angle is found through Eq. 13.6.

$$\theta_p = \frac{1}{2} \tan^{-1} \left(\frac{2U_{12}}{U_{11} - U_{22}} \right)\tag{Eq. 13.6}$$

The transformation matrix $\mathbf{Q}$ (Eq. 13.7) can then be used to diagonalise $\mathbf{U}$ through Eq. 13.8 to arrive at the principal strain.

$$\mathbf{Q} = \begin{bmatrix} \cos(\theta_p) & \sin(\theta_p) \\ -\sin(\theta_p) & \cos(\theta_p) \end{bmatrix}\tag{Eq. 13.7}$$

$$\mathbf{U}_{diag} = \mathbf{Q} \cdot \mathbf{U} \cdot \mathbf{Q}^T\tag{Eq. 13.8}$$

The diagonalised true strain matrix is calculated through Eq. 13.9.

$$\epsilon_{T,diag} = \ln \mathbf{U}_{diag}\tag{Eq. 13.9}$$

True strain in the original coordinate system can then be found through a transformation using $\mathbf{Q}$ once more, as outlined in Eq. 13.10

$$\epsilon_T = \mathbf{Q} \cdot \epsilon_{T,diag} \cdot \mathbf{Q}^T\tag{Eq. 13.10}$$

Where

$$\epsilon_T = \begin{bmatrix} \epsilon_{T,11} & \epsilon_{T,12} \\ \epsilon_{T,21} & \epsilon_{T,22} \end{bmatrix}$$

13.5.2 Image analysis

Measurement of the strain following machining was achieved by capturing SEM images of the micro-grids and comparing them to undeformed grids. To allow for faster and more automated strain analysis,

13.5.3 Validation

To validate the image processing and strain calculation algorithm, a series of images of deformed grids from published research were analysed with the algorithm and compared to the original results. One such comparison is shown in Figure 13.3. A plot of true strain YY from an unpublished figure from previous research in the field [154] and a strain map, created from the deformed image using the algorithm described in this chapter, are presented in Figure 13.3 (a) and (b) respectively. It can be seen that there is a close agreement between the two sets of results in terms of both the maximum and minimum strain values as well as their locations on the surface. The colouring of the images differs subtly due to the different colour scales adopted. By moving towards automation of the grid coordinate extraction, the time taken from image capture, through to the calculation of the strains is reduced, without a compromise in validity.

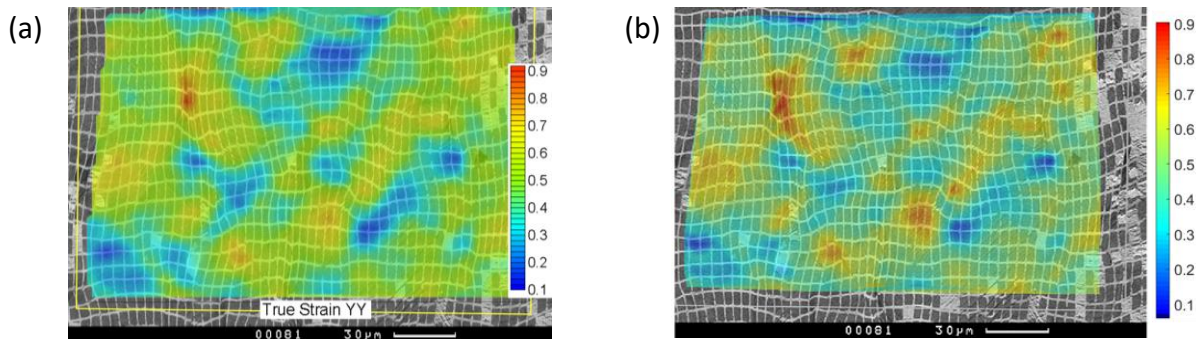


Figure 13.3 - Strain plots from tensile testing obtained using micro-grid methods: (a) an image from a previous study [154] (b) an image obtained using the image processing algorithm developed for faster strain analysis.

13.6 Tool contact time for diffusion of cutting tool elements

The tool contact area is approximately 0.5 x 2 mm due to the 0.5 mm wear land and its 2 mm width. The cutting speed for the surface machined with the longest contact time was 40 m/min and the tool diameter was 20 mm. The distance travelled during one tool rotation is calculated from Eq. 4.6.

$$0.02 \times \pi = 0.0628 \text{ m} \quad \text{Eq. 13.11}$$

The fraction of the tool rotation for which the cutting insert was in contact with certain region of workpiece is found from Eq. 13.12.

$$\frac{\text{width of contact patch}}{\text{total distance travelled in rotation}} = \frac{0.0005 \text{ m}}{0.0628 \text{ m}} = 0.00796 \quad \text{Eq. 13.12}$$

The rotation speed can then be calculated from Eq. 13.13.

$$\frac{40 \text{ m/min}}{0.02\pi \text{ m}} = 636.6 \text{ rpm} \quad \text{Eq. 13.13}$$

The time for one tool rotation is given by Eq. 13.14

$$\left(\frac{636.6 \text{ rpm}}{60\text{s}}\right)^{-1} = 0.0942 \text{ s} \quad \text{Eq. 13.14}$$

The time for which the wear land and a region of the workpiece are in contact per tool rotation is found from Eq. 13.15.

$$0.00796 \times 0.0942 \text{ s} = 0.000750 \text{ s} \quad \text{Eq. 13.15}$$

In the central region of the machined shoulder, the tool contact time is the longest due to the length of the wear land and numerous passes of the tool. The cutting tool contacts each workpiece region multiple times due to the length of the contact patch (2mm) and the feed (0.06 mm/rev) as well as contact on the second half of the cutting arc. Therefore, the total contact time can be found from Eq. 13.16.

$$\frac{2 \text{ mm}}{0.06 \text{ mm/min}} \times 0.000750 \text{ s} \times 2 = 0.05\text{s} \quad \text{Eq. 13.16}$$

13.7 Eddy current c-scans

During the first year of the EngD, time was spent at the Rolls-Royce NDT offices and laboratories investigating the application of eddy current testing for identifying anomalous machined surfaces in titanium. The key results are shown here for completeness. The technique was unsuccessful in detecting features such as swept grain and white layer due to an overriding sensitivity to surface roughness through lift-off effects.

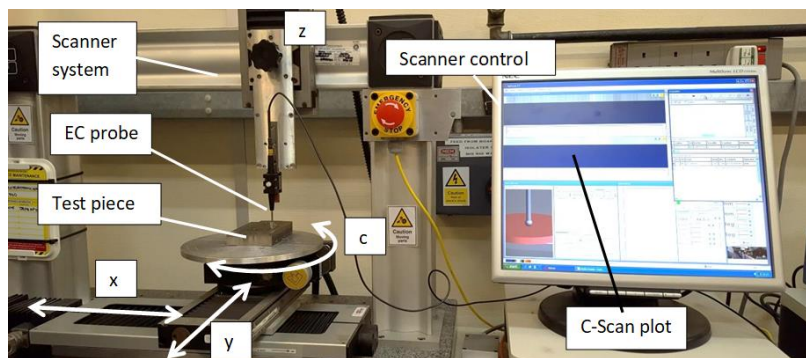


Figure 13.4 – The test rig for the experiments using the ELOTEST B300 ECT system

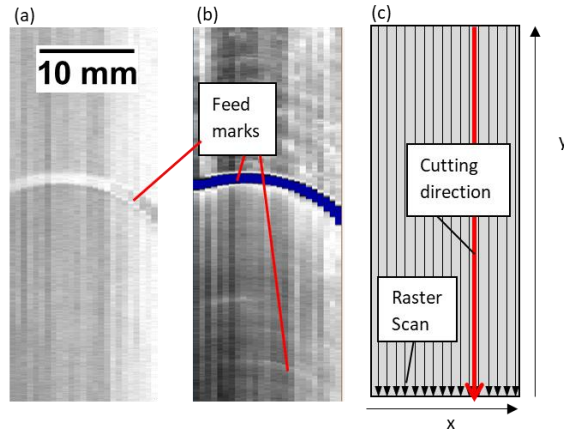


Figure 13.5 – (a) C-scan of the machined surface coloured by the y-value of impedance (changes largely independent of lift-off). (b) C-scan of the machined surface coloured by the x-value of impedance (lift-off effects). A darker colour corresponds to a larger x-impedance and therefore larger lift-off. (c) Schematic showing the scan over the machined surface that produces the c-scan images.

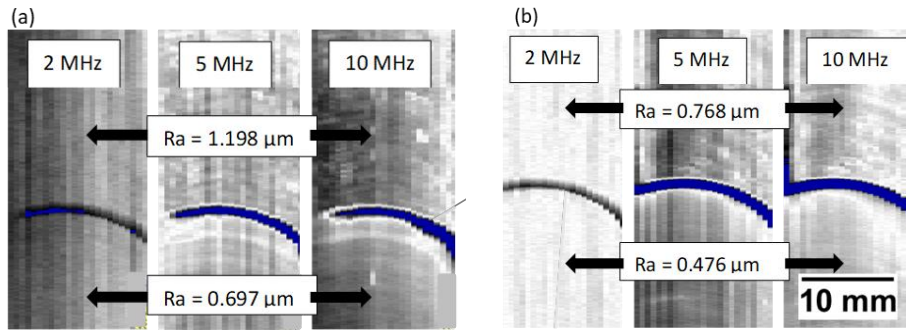


Figure 13.6 – y-impedance c-scan images of the machined surfaces from (a) trial 40 (40 m/min, 0.8 mm VB, 0.5 mm a_e) and (b) trial 30 (40 m/min, 0.8 mm VB, 0.5 mm a_e). The surface roughness of the machined surface before and after the large feed mark caused by tool failure is outlined for all surfaces.

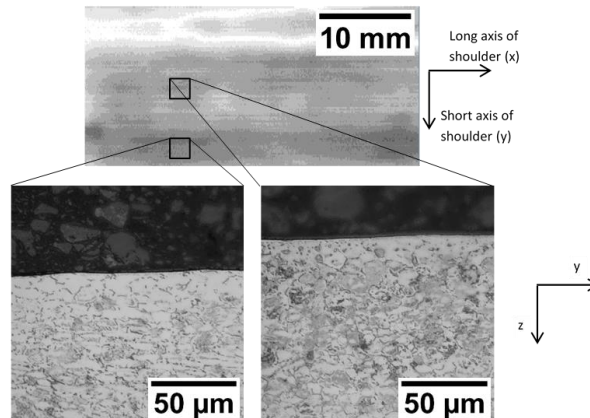


Figure 13.7 – Comparison between an ECT c-scan and cross-sectional optical microscope images from different locations across the width of the machined shoulder from trial 19 (200 m/min, 0 mm VB). The optical microscope images are shown with their relative location across the short axis of the insert.

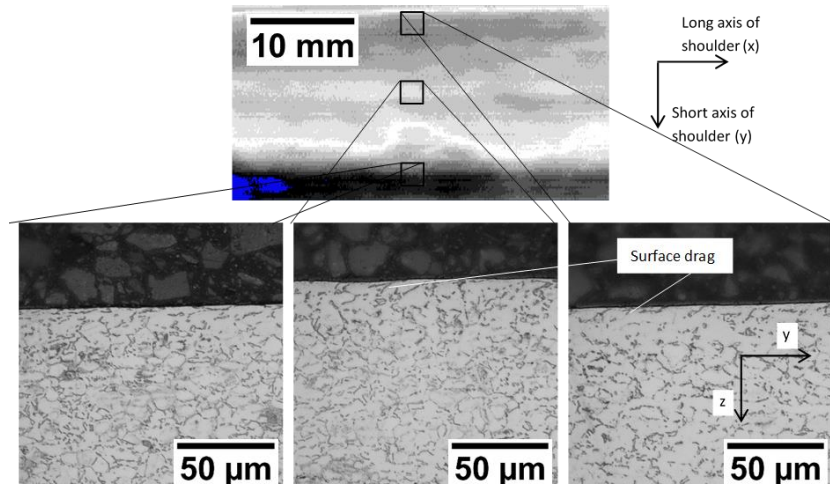


Figure 13.8 – ECT c-scan and optical microscopy images for the machined surface from trial 17 (20 m/min, 0 mm VB).

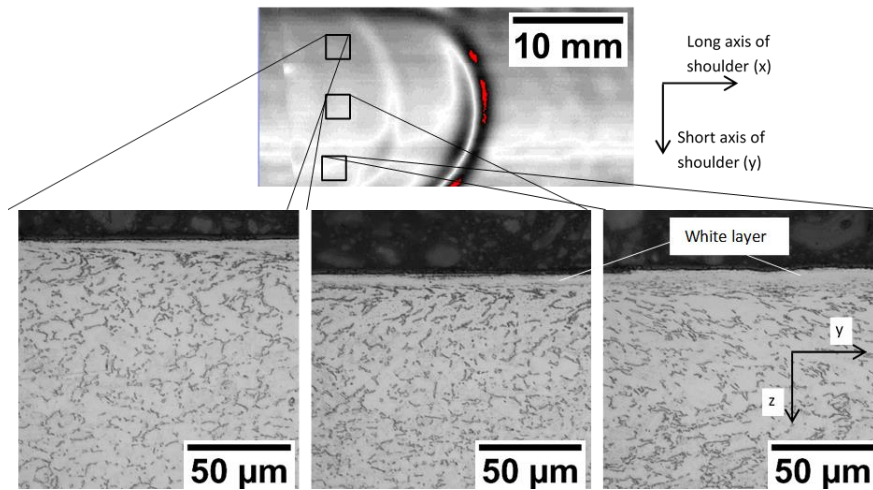


Figure 13.9 – ECT c-scan and optical microscopy images for the machined surface from trial 40 (40 m/min, 0.8 mm VB).

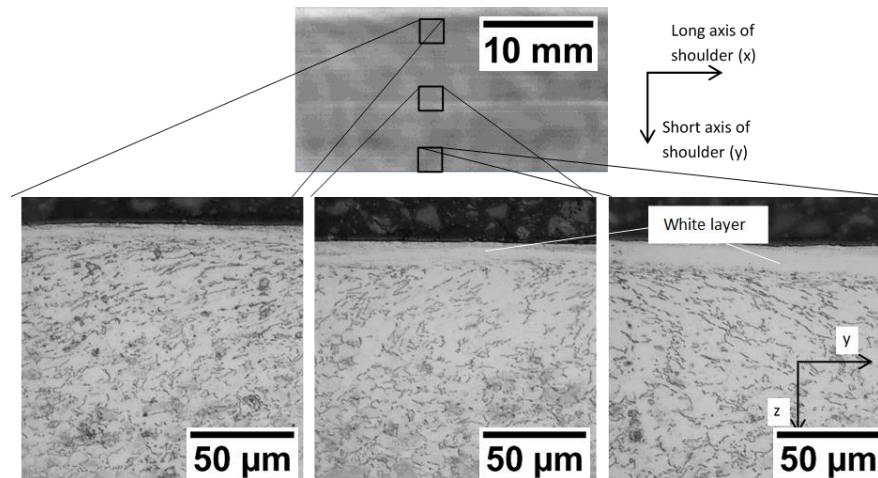


Figure 13.10 – ECT c-scan and optical microscopy images for the machined surface from trial 37 (40 m/min, 0.5 mm VB).

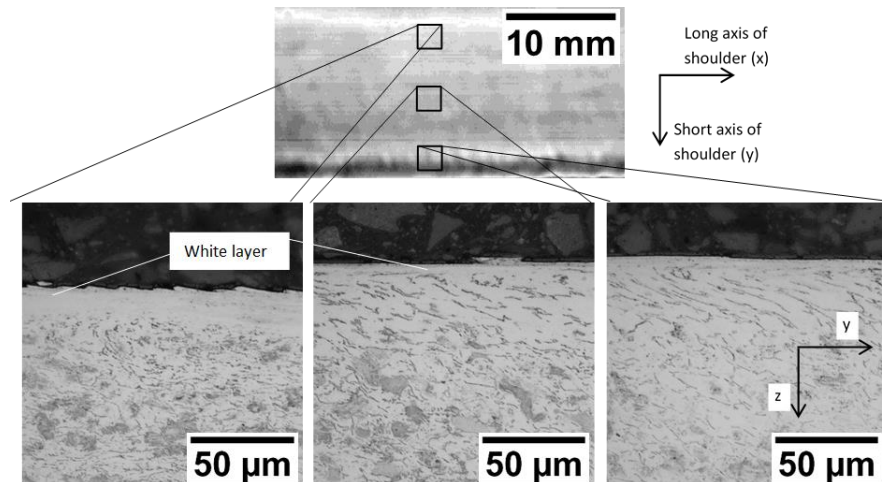


Figure 13.11 – ECT c-scan and optical microscopy images for the machined surface from trial 36 (80 m/min, 0.5 mm VB).

13.8 SRAS velocity maps k-means cluster analysis

Attempts were made to utilise k-means clustering to identify macrozones in filtered SRAS velocity maps, however, these attempts proved unsuccessful as a numerical measure to distinguish the clusters in one image from those in another could not be identified. k-means clustering failed to produce images which were distinct within one surface type, as shown in Figure 13.12 (a-d), where it can be seen that two surfaces with poor SI appear very dissimilar and the macrozone (cluster) sizes are larger than good SI surfaces in one case and smaller in the other. The same result was encountered when increasing the number of clusters, as such this approach was taken no further. For automated macrozone identification to achieve reliable results, it is thought that a larger number of samples are required from which data set can be built up and used to train a machine learning classifier algorithm.

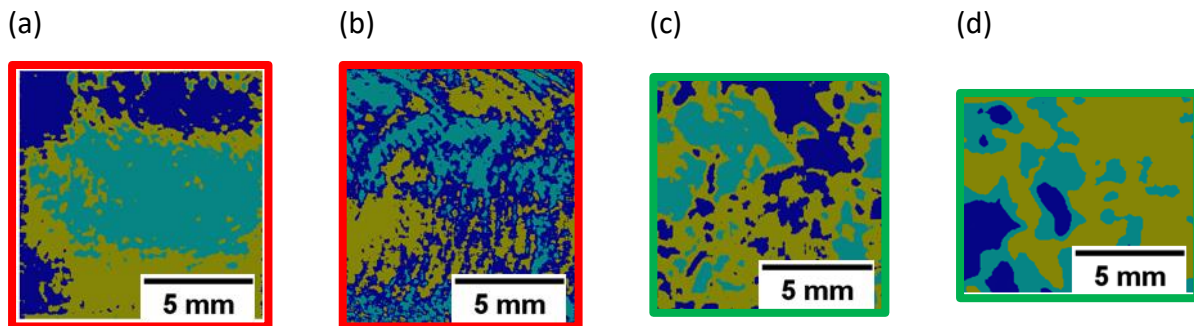


Figure 13.12 – Images of SRAS surfaces coloured by the cluster to which each pixel was assigned during k-means clustering with 3 clusters (a) poor SI, (b) poor SI (c) as-received and (d) good SI.