The structure and properties of down feathers and their use in the outdoor industry

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The candidate confirms that the work submitted is his/her own, except where work which has formed part of jointly authored publications has been included. The contribution of the candidate and the other authors to this work has been explicitly indicated below. The candidate confirms that appropriate credit has been given within the thesis where reference has been made to the work of others.

Chapters 4 and 6 contain work also included in the publication: Fuller, M., Mao, N., & Taylor, M. (2013). The microstructure and tensile properties of goose and duck down fibres. In ATC12. Shanghai: The Asian Textile Council.

In the above work, M Fuller carried out all of the practical and analytical work and was lead author of the paper. N Mao and M Taylor carried out editing and proof-reading of the text.

"It doesn't have to be fun to be fun."

Mark Twight (Twight & Martin 1999)

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Abstract

Down feathers are remarkable insulating materials that reputedly have the greatest warmth-to-weight ratio of all natural fibres, and they possess excellent compressibility and compression recovery. Despite their outstanding performance as thermal insulators, down feathers have been relatively overlooked by the academic community and their structure and properties remain quite poorly understood. To provide insight into the fundamental properties of these feathers and to inspire the design and development of future synthetic insulation materials, a study into the structural, mechanical, and thermal properties of down feathers and their assemblies has been conducted.

The appearance, mass, size, and geometric shape of goose, duck, and eider down plumes and their barbs were assessed. While goose and duck down plumes were very similar in both appearance and size, the eider down feathers were found to be larger and their barbules had a greater number of prongs and nodes. In each type of down, barbules were adapted to occupy maximum space and utilised a planar cross-section as they divided from the barb to optimise compression recovery. The microstructure and morphology of goose and duck down barbs and barbules were analysed using SEM, TEM, and AFM and were found to be analogous to wool fibres. Melanin granules were found in goose down but not in duck down, and the examined goose down barbs had irregular, hollow cross sections, whereas duck down's were solid and more elliptical. With the help of novel down-based nonwovens developed to capture X-ray diffraction data from goose and duck down with excellent clarity, goose and duck down were found to share great similarities in their crystal structures.

The mechanical properties of goose, duck, and eider down feathers were studied, including a comparison of their barbs' tensile properties and the compression resistance and recovery of individual down plumes. Eider down barbs were found to have greater Young's moduli, ultimate strength, and strain at break than goose or duck down barbs, and individual eider down plumes were also more compression resistant than those from geese and ducks.

The compression resistance and recovery of goose and duck down assemblies were studied using a novel apparatus. Goose down proved to be more compression resistant than duck down, attributed to its cross sectional shape and hollow geometry. The thermal resistances of goose and duck down-filled test squares were extremely similar, but the densities of the down inside the face fabrics strongly influenced their thermal resistances.

Air-laid and thermal-bonded down-feather-based nonwovens were developed to alleviate the problem of constraining individual down feathers in insulated products. Their structural and thermal properties were evaluated and they possessed industry-leading warmth-to-weight ratios, which could be further improved by better engineering of these composite materials.

Great efforts have been made to provide a comprehensive investigation into the structure and properties of both individual and bulk down feathers in relation to their thermal insulation properties. It is hoped that this research will prove useful to the development of superior biomimetic synthetic insulations as well as the high performance products made from down, a world-leading natural material.

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Glossary and abbreviations

Down terminology

Plume – an individual down feather

Distal – end furthest from centre of plume

Proximal – end nearest centre of plume

Core – central point of feather

Barb – fibre emanating from core

Barbule – fibre emanating from barbule

Node – triangular/pyramidal structure on barbules

Crotch – hook on a barbule

Abbreviations

ADF – annular dark-field

AFM – atomic force microscopy

ANOVA – factorial analysis of variance

ATR – attenuated total reflectance

BMC – British mountaineering council

CT – computed tomography

CV – cumulative variance

DWR – durable water repellence

EDX – energy-dispersive X-ray

EOG – European Outdoor Group

FP – fill power

FT – Fourier transform

FWHM – full-width half-maximum

IDFL – International Down and Feather Laboratory

IR – infrared

MYOG – make your own gear

R_{CT} – resistance to conductive heat transfer

RMR – resting metabolic rate

SDD – silicon drift detector

SEM – scanning electron microscope

STEM – scanning transmission electron microscope

SD – standard deviation

TEM – transmission electron microscope

XRD – X-ray diffraction

Chapter 1. Literature review of down feathers and their use in relation to human thermophysiological comfort

Down feathers are used in many applications, including bedding, upholstery, and fashion clothing, though it is outdoor clothing and equipment that places the greatest demands on them. Outdoor wear must protect against the harshness of an environment hostile to the human body and down equipment is primarily used to protect against the cold. This chapter will establish the scope and context of this research project and provide a brief overview of how down feathers are used in garments and equipment. It will also review the existing literature regarding the properties of these remarkable insulating materials.

1.1 Human thermoregulation and clothing comfort

Humans are homeothermic and their 37 °C core temperature must be maintained to ensure wellbeing (Rossi 2009) and to make sure that the temperature-sensitive enzymes that drive the body's metabolic reactions can remain effective (Morrissey & Rossi 2013). The human body is well adapted to counteract overheating as it is equipped with very effective sweat glands (Edholm 1978b) but man is poorly evolved to deal with the cold found at high latitudes or altitudes (Edholm 1978a) and so man must wear clothing or increase heat production beyond that of resting metabolic rate to maintain core body temperature.

1.1.1 Cold weather

Cold weather can be distracting, uncomfortable, or dangerous (Gavhed 2003). However, it is an intrinsic part of many people's lives: an estimated 3.5 million people live year-round inside the Arctic circle (Hassi et al. 2002) and approximately half of the earth's landmass and one tenth of the oceans are covered with ice and snow year-round (Ashcroft 2001). Throughout history, major events have been influenced by cold weather, such as Hannibal's famous crossing of the Alps (Ashcroft 2001), the invasion of Russia by Napoleonic France in 1812, and Hitler's unsuccessful invasion of Russia (Winters et al. 2001). Protecting people from cold is extremely important to ensure their wellbeing and safety.

Cold injuries are still commonplace amongst people living or working in cold climates, and many cases of hypothermia and local cold injuries such as frostbite and trench foot, are attributed to inadequate clothing and equipment (Lloyd 1986; Reamy 1998). Hypothermia occurs when the heat lost from the body exceeds that gained through food, exercise, and external sources (Thompson & Hayward 1996). Even in developed countries, hypothermia is a significant danger, killing an average of 1300 people per year in the United States from 1999-2011 (Jiaquan 2013). Even mild hypothermia can be very serious, resulting in a clouding of consciousness, a blurring or vision, of a feeling of apathy (Parsons 2003a). These symptoms are dangerous in any situation, but more-so in remote places or in serious situations, where decisions must be made quickly and rationally. The polar explorer Berton (1988) described these manifestations as “the frost... seemed to extend to the brain”.

1.1.2 Thermal comfort and heat balance

The development of clothing (approximately 72,000 years ago) may coincide with the population of colder climes by humans (Hipler & Elsner 2006) and to fully appreciate the protection required of the human body, it is necessary to understand how the body regulates temperature. This section concerns how heat is produced by the body, how body temperature is regulated, and what thermal comfort is and how it might be maintained.

Physiologically, thermal comfort is achieved when the body is in a state of heat balance and where heat loss is approximately equal to heat production. Thermal comfort has been defined as “that condition of mind which expresses satisfaction with the thermal environment” (Parsons 2003c) and it is therefore both a psychological and physical phenomenon. Other academics, however, have argued that thermal comfort cannot be perceived, and only thermal discomfort can be detected (Li 2001).

The most important factors that influence thermal comfort are (Rossi 2009):

1) That the body is in heat balance
2) That the mean skin temperature is within comfortable limits
3) That there is no local thermal discomfort

As such, even when producing large amounts of heat in cold conditions, cold fingers or exposed skin may still detract from overall thermal comfort.

To survive cold conditions, reducing one’s heat loss or enhancing ones heat production is necessary (Vallerand 1995). The former can be achieved through the use of clothing or shelter and the latter by increasing thermogenesis, the heat produced through metabolic processes. The body produces heat by four main routes. These make up the body’s thermogenesis and are described in Figure 1-1 (Vallerand 1995):

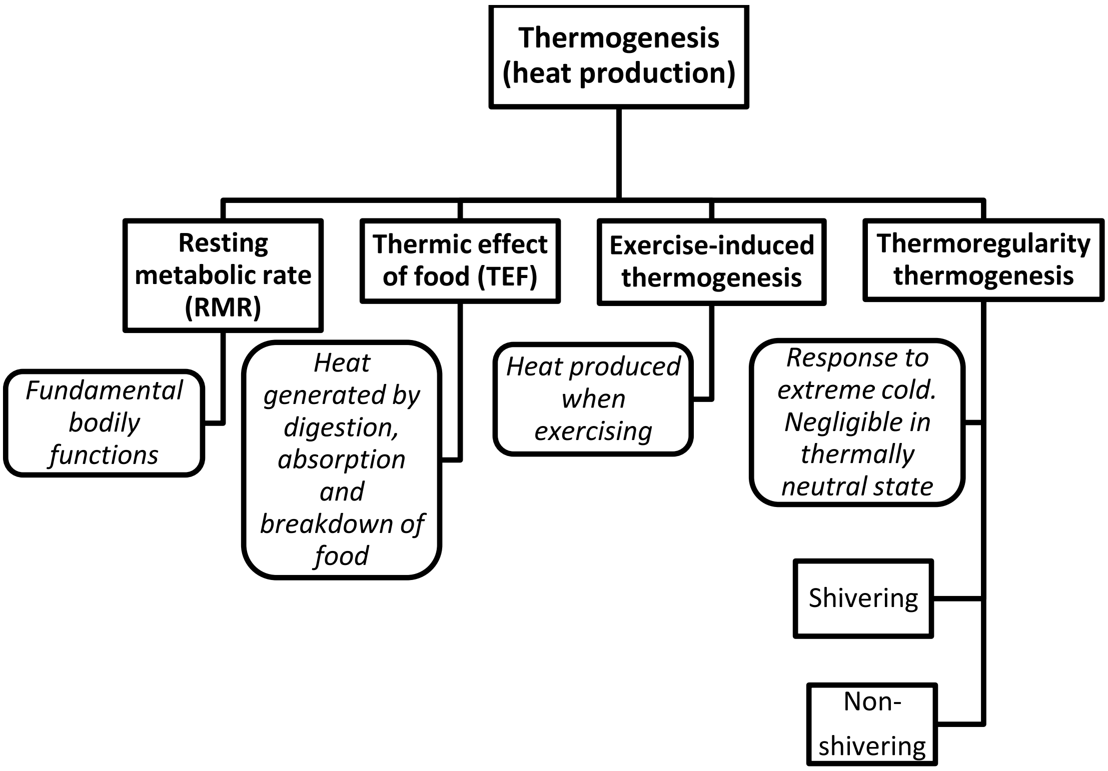


Figure 1-1 – the four main aspects of thermogenesis (Vallerand 1995)

As shown in Figure 1-1, the body's rate of heat production can be adjusted by consuming food or exercising. The body's thermoregulatory thermogenesis mechanisms such as shivering are used only when the body is very cold: though shivering can increase heat production by up to five times, it is partially hindered by the increased convective heat losses that result from trembling (Ashcroft 2001) and is extremely inefficient and exhausting. The duration of shivering that the body can undergo is limited by its glycogen store.

The body can be described by Fanger's heat balance equation (Fanger 1970; Edholm 1978b) shown in Equation 1-1 that lists major heat loss and heat gain pathways (Fanger 1970):

$$M = E + C + K + R + S \qquad (1\text{-}1)$$

where *M* = metabolic rate, *E* = evaporative heat loss, *C* = convective heat change, *K* = conductive heat change, *R* = radiative heat change, *S* = heat storage

Greater description of the terms described in Equation 1-1 is given in section 1.3.5.4.

1.2 Outdoor sports and mountaineering

As discussed in section 1.1.2, humans are poorly adapted to deal with cold conditions, requiring shelter or clothing to counter this environment. Many outdoor pursuits take place in cold and challenging places, and the number of participants in these activities continues to grow (Bowker et al. 1999). In these conditions, clothing that protects against low temperatures is required.

Winter sports that take place near to the safety of habitation and shelter require less protection in the form of clothing and equipment than sports taking place in remote areas. For example, a skier or snowboarder on-*piste* is unlikely to be more than thirty minutes from habitation, and relatively near a hospital in case of mishap. Cold is most threatening when one is in a remote place away from immediate shelter, and for this reason, many extreme-cold-weather garments have their origin in polar exploration, an activity that demands self-sufficiency and protection from very low ambient temperatures. However, despite the difficulties of maintaining core temperature in polar conditions, the constant heat output of polar explorers pulling sledges means that dressing in appropriate clothing is relatively simple, compared to if metabolic heat output varies greatly. Thus, the three aspects that most influence the demands of cold weather clothing are the temperature and conditions; a remote location; and frequently-changing activity levels. One sport that combines all of these factors is mountaineering. It is made yet more complex by the need to minimise the weight of any equipment, as any uphill movement is greatly hindered by additional mass; by the extreme changes in temperature encountered in the mountains; and by the effects of altitude. Therefore, though numerous outdoor sports take place in cold conditions, mountaineering could be considered the most demanding of its equipment and clothing, and will therefore be considered in more detail.

Mountaineering is a diverse sport that encompasses many different disciplines, but the ascent of mountains inevitably entails colder conditions – the Earth's dry lapse rate is 9.8 °K km^{-1} (McElroy 2002), meaning that if it is 10 °C at sea level it may be approximately 0 °C at 1000 m and -10 °C at 2000 m. Some routes or mountains cannot be climbed in summer time due to dangers of rock-fall (snow and ice can hold loose rocks in place), or the route may be a 'winter-only route', and so scaling mountains when it is cold is sometimes necessary. However, cold is not the only concern, as mountaineers encounter great extremes in temperature. A typical summer ascent in the European Alps may begin with an approach, carrying all one's equipment, in temperatures of 30 °C. The temperature at night may drop below -10 °C, demanding an extremely insulated sleeping bag and warm clothing for any climbing in the early morning. In the greater mountain ranges (Himalayas, Karakorum, Andes), similar temperature fluctuations may be encountered, though the minimum temperatures may fall as low as -50 °C. In maritime climates such as in the UK, the air temperature may not often fall as low as this, though the perceived cold can be severe because of the humid air, high wind speeds, and rapidly changing weather.

Of the major types of mountaineering, winter climbing, Alpinism and high altitude mountaineering present the greatest risk of cold conditions (at high altitude the risks of hypothermia and cold injuries are heightened because oxygen deficiency reduces the body's calorigenic response to cold (Blatteis & Lutherer 1976) and forces the climber to slow down, meaning that exercise-induced thermogenesis (see section 1.1.2) is also reduced. In remote mountain ranges the cold hazard is most dangerous; and fatigued and energy-starved mountaineers are most susceptible to cold.

1.2.1 The fundamental principles of mountaineering

To fully understand the problems associated with keeping warm in a mountaineering situation it is necessary to have an awareness of how a climb is usually undertaken. This section will describe some of the fundamental principles of mountaineering.

Climbs are often undertaken in pairs. The first climber (the lead climber) will ascend with one end of the rope attached to their harness. As they ascend they will attach the rope to pieces of protection that are held in the rock, snow or ice. These are intended to reduce the length and potential severity of a fall if one were to occur; they do not reduce the chance of a fall happening. Meanwhile, the second climber must belay, thus arresting a fall by using the rope that the lead climber might have. Once the lead climber has either used the full length of rope or reaches a suitable point they will secure themself and the second climber will ascend to join them, removing the protection that the leader placed. This process is repeated until the top of the climb is reached.

Climbing can be very physically and mentally demanding, leading to a great increase in thermogenesis. Belaying, however, generates very little heat, as the climber must remain almost stationary during this period. The great difference in heat output between these two states is a reason why mountaineering places such demands on its clothing, further compounded if clothes are wet from inclement weather or previous exertions. Additional demands of mountaineering are described in the following section.

1.2.2 Demands on mountaineering equipment

A mountaineer must carry their equipment, and so it must be as light and small as possible. It must also be durable to withstand abuse, and it must be adaptable to different environments and activity levels. Equipment may get wet from precipitation, spindrift (wind-blown snow), or perspiration, so ensuring that it can dry quickly is important. Additionally, clothing must fit well and enable ease of movement.

The primary role of insulated clothing is to keep the body warm, and as minimising the weight of a garment is vital in mountaineering, the first of three important criteria for an insulating material is its warmth to weight ratio. The insulating effect of clothing is largely governed by its ability to trap still air (Morrissey & Rossi 2013), an excellent insulator (see Table 1-2), and more air is trapped by thicker clothing (Rossi 2009; Keighley & Steele 1980). Indeed, there is a very strong relationship between the thickness of a textile and its thermal resistance (Fletcher 1945; Schiefer 1944; Pierce & Reese 1946; Goldman 2006; Morris 1955). It follows that any material that can maintain thickness with minimal weight will be an excellent insulating material for use in outdoor garments and equipment, but this high volume is a hindrance if trying to store and carry the material. This means that foams and similar high-volume materials are unsuitable for clothing that must be stored when not in use. Thus, a highly-compressible insulating material with excellent compression recovery is desirable. A final consideration is the performance of the material when wet: a material that loses its warmth when it is wetted is likely to be unsuitable for some conditions found in the mountains.

1.2.3 The layering system and the use of down in mountaineering equipment

To protect against cold, no single clothing layer is suitable. As a result, numerous garments are worn by mountaineers and this has become known as the layering system (Stevens & Fuller 2014). Because clothing hinders the cooling mechanisms of the body, particularly evaporative heat loss (Parsons 2003b), the layering systems allows the easy donning and removal of clothing to prevent overheating and subsequent sweating.

The layering system that most modern mountaineers and hikers are aware of describes the three layers that cover one's torso. These layers are: a baselayer, designed to wick moisture from the skin (Morrissey & Rossi 2013); a mid-layer, to provide insulation; and a waterproof jacket, designed to prevent water ingress and prevent wind displacing still air in and between clothing layers. For many users these three layers are adequate for summer use and this system is recommended for most users. However, it has some flaws (most vividly described by the mountaineer Twight (Twight & Martin 1999)) and in colder environments these three layers are often insufficient and more layers will be carried or worn. Extra layers are frequently insulated with down feathers, which were used in bedding centuries before they were used in outdoor clothing and equipment. The first down sleeping bags were developed at the end of the 19th Century, and the first known use of a down jacket was on Mount Everest in 1922, but the garment was dismissed over concerns regarding its durability. Eider down sleeping bags were a staple of the 1933 British Everest expedition (Ruttledge 1934), and breakthrough designs in the 1960s and, in particular, by British brands such as Mountain Equipment, led to the modern foundations of down clothing and equipment that have remained relatively unchanged since (Parsons & Rose 2003).

When assessed according to the three important criteria for an insulating material discussed in section 1.2.2 – warmth-to-weight ratio; compressibility and recovery from compression; and water-resistance – down fares very well. Indeed, down is regarded very highly by mountaineers (and is thought by some academics to be superior to all other insulations (Kasturiya et al. 1999)) because of its excellent compressibility (Gao et al. 2010) and compression recovery (Martin 1987), and virtually-unparalleled warmth-to-weight ratio (Gao, Yu & Pan 2007b). Eider down, in particular, has a near-legendary warmth and it has been suggested (Todd 1996) that eider down is the most thermally-insulating of all natural materials. Down also benefits from extremely high durability, excellent touch comfort, and a strong track record. It has remained the choice for many mountaineers in cold conditions, both as filling for their sleeping bags and in their warmest garments for 50 years, and is synonymous with ascents of very high mountains such as Mount Everest. When compared to traditional insulations such as wool or animal furs, down's greatest asset is its warmth-to-weight ratio (Havenith 2010) and when compared to modern synthetic insulations this property remains unsurpassed (Gao, Yu & Pan 2007b; Kaufman et al. 1982; Farnworth & Osczevski 1985). For example, a down jacket of equal warmth to a synthetic jacket would be approximately half of its mass (Morrissey & Rossi 2013).

The major disadvantage of using down in outdoor wear is its performance in wet conditions. Down can clump together when wet (Farnworth & Osczevski 1985) and this reduces its thermal resistance as air is forced from the insulation. This susceptibility to wet conditions is reflected in its use: down garments and sleeping bags are not normally very water resistant and so down sleeping bags are used inside waterproof shelters such as tents, bivouac ('bivvy') bags, or buildings, while down garments tend to be worn in cold and dry conditions, underneath waterproof clothing, or in conditions where it is too cold to rain and instead will snow. Examples of use are shown in Figure 1-2:

Figure 1-2 - example uses of down products
In each case the down is kept as dry as possible. 1 – Use of a down jacket on the summit of Mont Blanc, French Alps, the highest mountain in Western Europe. 2 – Use of down sleeping bags inside bivvy bags on an Alpine glacier. 3 – Use of sleeping bags inside bivvy bags in the Lake District, England. 4 – Use of a down jacket as a belay jacket while mountaineering in winter, Scotland

Frequently-used alternatives to down are nonwoven synthetic insulations, such as Primaloft (Donovan 1986), Thinsulate (3M 2012) and Polarguard (Harding 1979; Frankosky 1983). These fabrics are available in different basal weights and thicknesses and, when compared to down, perform very well when wet, losing little thermal resistance. However their dry warmth to weight ratio is inferior to down's and their performance diminishes with repeated compression (REI 2014). Synthetic insulations are usually cheaper than down, which is too expensive for some consumers (Farnworth & Osczevski 1985), though down's excellent lifespan offsets this over time.

Because of its excellent warmth-to-weight ratio and compressibility, down is invariably used as an insulating layer in outdoor clothing but because it is used in a diverse range of situations, from the high street to the high mountains, it is a component in many different types of garments and equipment. These will each be discussed in turn.

1.2.3.1 Down jackets

Down jackets vary greatly in design and use, from jackets weighing 200 g designed for use as emergency insulating garments or conventional midlayers, to jackets weighing 1000 g that are used in Polar exploration or on extremely high mountains. All down jackets share an excellent warmth-to-weight ratio that means they can be carried as 'just-in-case' clothing and they are extremely warm when worn.

Heavy-weight down jackets tend to be worn over the top of all other layers because of their extreme bulk. Because these jackets are so warm they are likely to be worn in conditions where rain is unlikely. Thinner down jackets, which might be worn in conditions above freezing, are often sized to fit underneath waterproof clothing. Mid-weight jackets of roughly 500-600 g total weight are usually sized to fit over or under a waterproof jacket, giving the greatest flexibility.

1.2.3.2 Down sleeping bags

Mountaineering routes lasting more than a day may require sleeping in a remote place, and a sleeping bag is the most common sleep system, being an efficient design that maximises insulation and minimises mass. Sleeping bags are usually protected from the weather by a tent or bivouac ('bivvy') bag, and kept from the ground by a compression-resistant mat, so their role is not to be weatherproof or compression-resistant but to be as warm and as light as possible. Two competing factors in choosing a sleeping bag's insulation are its maximum bulk and volume when in use, thus enabling the trapping of maximal air; and its minimum volume when not in use, therefore taking up minimal space in a rucksack (Weiner 1955). In this regard, down-filled sleeping bags have an enviable reputation.

The US military state that the highest pressure exerted by a body lying in a sleeping bag is 3 psi (Gibson 1990), which is almost identical to the 200 g cm^{-2} determined by Martin (Martin 1987). This pressure is sufficient to compress virtually any quantity of down until it is flat and thus has negligible insulating properties (Farnworth et al. 1985), making a compression-resistant sleeping mat required to reduce direct heat conduction to the ground. This arrangement is described in Figure 1-3:

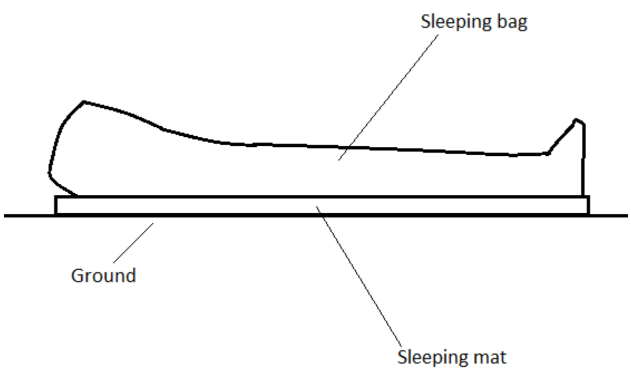


Figure 1-3 – use of a sleeping bag with a sleeping mat to protect the user from direct heat conduction through the ground

1.2.3.3 Other garments and equipment

Down can be used to insulate almost any item of clothing or equipment. In addition to jackets and sleeping bags, down vests are common in the UK. Down gloves, hats and suits are unusual in the UK but are common in polar exploration or in use at very high altitude. Down suits are a staple of extreme-cold-weather use, but their exceptionally-high thermal resistance makes them unsuitable for most conditions. Down has also been used in insulated sleeping mats which inflate to protect the down from compression (Exped 2014), and down quilts are popular amongst some mountaineers.

1.2.3.4 Construction of down garments and sleeping bags

Down equipment's construction is deceptively complex and makes a significant contribution to the overall performance of the final product. Face fabrics that are easily abraded or torn or possess a high resistance to evaporative transfer could ruin an otherwise excellent product. Similarly, down that migrates in the garment or sleeping bag and therefore insulates unevenly is undesirable. Down is an excellent insulating material but it must be allied to sensible designs and construction methods to maximise its performance.

Unlike synthetic insulations that can be sewn into place, down must be contained in baffles to keep it in place. Baffles are usually sewn shut on three sides and the down is then either blown or hand-stuffed into place. The final side of the baffle is then sewn shut to entrap the down. There are numerous baffle designs that are used in different applications, the simplest of which is stitched-through and is shown in Figure 1-4:

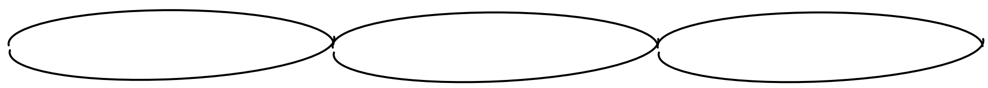


Figure 1-4 – cross-sectional view of stitched-through baffle construction

Stitched-through construction is low-cost and lightweight making it ideal in sleeping bags designed to be used in warm climates, or in lightweight down sweaters or jackets. Using this construction technique the baffles are limited in their thickness, however, which means this technique is not suitable for making very warm equipment. It also creates 'cold spots', where the stitching joins the inner and outer fabric together.

Box-wall baffle construction, as shown in Figure 1-5, is more costly than stitched-through designs but offers advantages in some applications. Box-wall construction separates down compartments with a thin mesh fabric orthogonal to the face fabric to prevent down migration (Rab & Equip 2010). Cold spots can still occur at the edges of each baffle and compression resistance is thought to be poor if the baffle is pushed from the side (the baffle can 'topple').

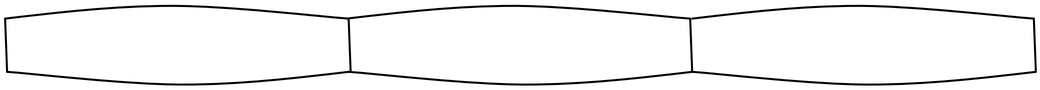


Figure 1-5 - cross-sectional view of box-wall baffle construction

The most advanced baffle construction regularly used in commercial products is the trapezoid baffle (Figure 1-6) which tends to be used only in very warm sleeping bags. By using slanted baffles the structure is resilient to compression and down is distributed evenly, preventing cold spots (Rab & Equip 2010). However, trapezoid baffles are difficult and costly to manufacture and are marginally heavier than box-wall baffles.

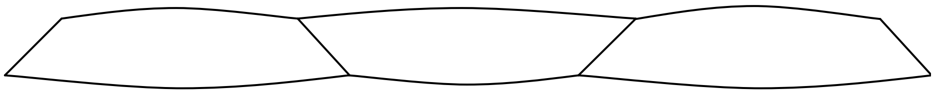


Figure 1-6 - cross-sectional view of trapezoidal baffle construction

Down must be enclosed by face fabrics that do not allow down to penetrate them: if down can escape the fabric then it can no longer insulate. The British Standard BS 12132 for down-proof materials demands that very little down escapes the fabric when shaken, impact-tested, and abraded (British Standards Institute 1999a; British Standards Institute 1999b). Low air permeability in a fabric can also be an indicator of down-proofness but should not be regarded as definitive (IDFL 2013), as not all fabrics with low air permeability are down-proof. During use of a product, down very rarely escapes a fabric, but flight feathers with their more penetrating quills may occasionally, particularly at seam points.

The weight of a face fabric is also important: lightweight fabrics with good drape allow down to loft as much as possible and minimise the overall mass of the equipment. However, lightweight fabrics often lack the durability of heavier fabrics and a balance must be reached. Fabrics should also be water resistant and allow moisture vapour to escape them as these help ensure that the down remains dry and thus can insulate with maximum efficacy. Waterproof fabrics that have a relatively high resistance to water vapour transport can offer advantages in very wet weather but they hinder the drying of a sleeping bag which means these fabrics are rarely used (Twight & Martin 1999).

1.3 Structure and properties of down feathers

This section will provide a review of the research that has previously been carried out on down feathers. Considering the exceptional insulating qualities of down, relatively little research has been carried out into its structure and so, on occasions when existing information is scant, the literature regarding analogous materials such as wool and hair will be reviewed as this will aid subsequent discussions. The hierarchical nature of down lends itself to a hierarchical approach to a literature review and as such, the down feather will be discussed considering its origins; its microscopic and macroscopic features; and its individual and bulk properties.

1.3.1 Down production

Down feathers are unique to waterfowl (geese, ducks and swans (Todd 1996)) and only develop in species in which the female incubates their eggs alone (Kear 2005a). Man has interacted with waterfowl for thousands of years (Kear 2005a): ducks and geese have been domesticated for more than 4000 years (RSPCA Research Animals Department 2011) and have been subject of a long-lasting fascination for humans; even Tutankhamen's tomb depicts the young Egyptian Pharaoh hunting them (Todd 1996).

Down is largely a by-product of the meat industry and holds little economic sway. As dictated by our diet, geese and ducks are the major sources of down, not swans. Geese are exclusively herbivorous and are adapted more to land-based feeding than ducks, which tend to lead a more aquatic life than geese (Kear 2005c). Down has excellent properties to help keep a clutch of eggs warm and, according to some academics, moist (Kear 2005a).

1.3.1.1 Domestication of geese and ducks

The Greylag Goose and Swan Goose are thought to be the first goose species to be domesticated and provided humans with meat, oil, down, feathers, and quills for use in arrows and pens (Kear 2005a). These two species are the wild ancestors of most domestic geese (Todd 1996; Guy & Buckland 2002) and are found worldwide from the Tropic of Cancer to the Arctic circle (Kear 2005c). However, researchers stress that most modern farmed geese tend to be "barn door" types and as such are not of any particular breed, though they tend to share common ancestors (Wyeld & Wyeld 1980).

The Mallard and Muscovy were almost certainly the first ducks to be domesticated (Kear 2005a). The Mallard is ancestor to many of the farmyard breeds in Europe and Asia and is probably the world's most common duck (Young 2005). The Muscovy duck tends to be farmed in warmer climes (RSPCA Research Animals Department 2011), such as in South America (Kear 2005a).

1.3.1.2 Goose and duck down sourcing and processing

Very equal quantities of goose and duck down are used in the outdoor industry: figures from the European Outdoor Group (EOG) show that 51 % of down used is from ducks and 49 % is from geese (European Outdoor Group 2013). The vast majority (90 %) of duck down is sourced from China, the remainder mostly obtained from Europe. Goose down is more mixed: 49 % of goose down is sourced from China but Hungary (24 %), the Ukraine (9 %) and other Eastern European countries are also major suppliers.

Most duck down is obtained from the birds 7-8 weeks after hatching, coinciding with slaughter, whereas goose down is obtained after 8-9 months (Bedard et al. 2008). Feathers make up a relatively small percentage of a bird's overall weight: 4-12 %, depending on the type of bird, its age, and other factors (Benedict 1937), so a domestic goose yields approximately 200 g of feathers and down of commercial quality. Down covers all parts of both males and female geese and ducks (Hardy & Hardy 1949) but is gathered in greatest quantity from the breast (Guy & Buckland 2002).

Hand-plucking of feathers from carcasses is time-consuming and inappropriate for large-scale operations, so dry-plucking machines may be used to remove feathers. Alternatively, the carcass may be plunged into a scald tank (Guy & Buckland 2002) and then plucked by a machine. Waxing may also be used to aid feather removal (Wyeld & Wyeld 1980). Once removed from the birds, feathers and down are washed in specialist soap and dried in industrial tumble driers (see Figure 1-7) and sorted in air currents (see Figure 1-8) (Guy & Buckland 2002). This is discussed in further detail in section 1.3.4.2.1.

Figure 1-7 – drying of washed down and feathers
(Taken at Peter Kohl Industries, Germany, 13th July 2012)

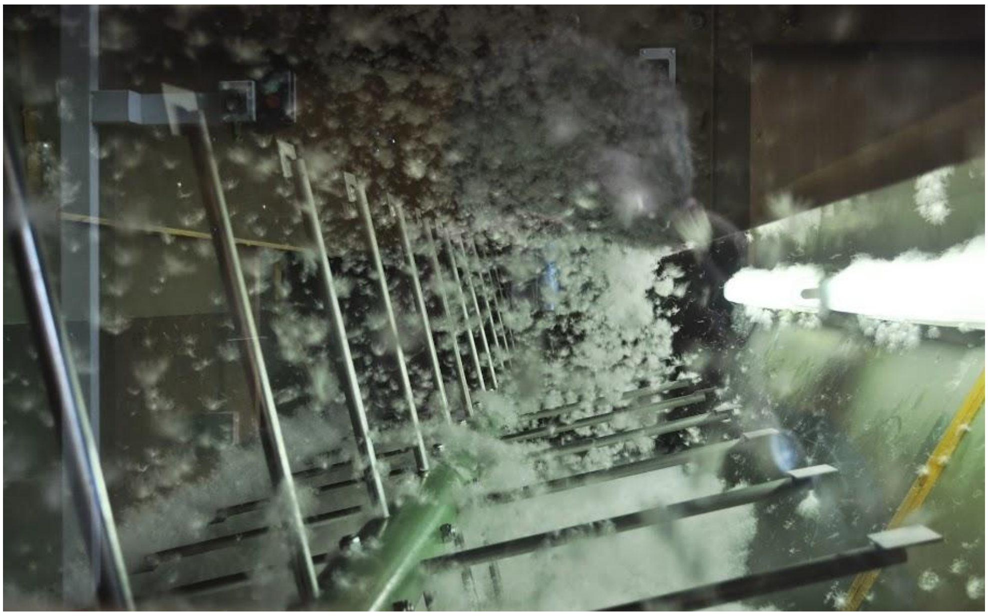


Figure 1-8 – sorting of down and feathers in air currents
(Taken at Peter Kohl Industries, Germany, 13th July 2012)

In down sorting machines, the down of greatest desirability and volume-to-weight ratio flies furthest in the air currents and thus is separated into different chambers to inferior down or feathers. The process may be considered analogous to fractional distillation. Because down is sorted alongside flight feathers, a portion of flight feathers inevitably mixes with the down and as a result '100 % down' is not commercially available: the highest quality down is usually 93:7, down: feather. Down of lower quality contains more feathers (IDFL 2010f), discussed in greater detail in section 1.3.5.3.

It is generally regarded that down quality tends to increase with the age of the source bird (Bedard et al. 2008; Yuwanta 2002) and, age being equal, goose down is regarded as superior to duck down, and fetches a higher price (Bedard et al. 2008; Jacob et al. 2011; IDFL 2010a). In a military study, European geese were found to produce higher quality down than Asiatic geese, though the results were not given extensive analysis (Cohen 1968), and the origin of down is not generally regarded as important in its final properties, though it may impact on desirability to a consumer (European goose down being highly regarded).

Goose and duck downs are processed separately to avoid mixing. They are also kept completely separate from other birds' feathers, which have far lower market values (for example, fowl feathers). In fact, following American concerns in the 1950s that the supply of down in wartime was not secure, extensive trialling of chicken feathers for use in sleeping bags took place. However, the chicken feathers (Hardy & Hardy 1949), a material in great abundance and extremely cheap (Loconti 1955) were considered unsuitable due to their low bulk density when compared to goose or duck down (Cohen 1968).

There are numerous factors that affect the overall quality of down, from the husbandry and age of the animal to the sorting and processing techniques. This makes the material highly variable and this influences all analyses of down.

1.3.1.3 Ethics of goose and duck down production

Down production is a major business and growing rapidly (figures from 1994 state that 67,000 tonnes of feathers and down were traded internationally, at a total value of 650 million US $ (Guy & Buckland 2002); estimations from 2014 indicate global production is now at least 270,000 tonnes (Bible 2014)) and like in many trades, ethical dilemmas exist. Over recent years, duck and particularly goose husbandry has come under widespread scrutiny (Boggan 2012) from the media, public, and animal rights groups, and has contributed to the starting of a large investigation and subsequent report (EFSA Panel on Animal Health and Welfare 2010) that contained many recommendations for animal welfare. The main problem reported was the live plucking of birds.

A factor that confuses 'live plucking' is that the collection of feathers from live birds should be divided into two types: 'gathering', which is plucking of ripe feathers from a bird and is not harmful if carried out correctly; and live plucking, which is the cruel and harmful plucking of unripe feathers (EFSA Panel on Animal Health and Welfare 2010; Wang 2010). Many academics agree that harvesting feathers at the time of a bird's natural moult (9-10 weeks after hatching and at subsequent 6 week intervals in the case of geese) is harmless (Labatut 2002; Rosinski 2002) and assert that harvesting of feathers from live birds can form a significant and important income for farmers breeding geese for foie gras or meat production (Guy & Buckland 2002). If plucking leaves blood marks then this is regarded as the wrong time to pluck (Labatut 2002), though even this may be tolerated on some farms, as it enables the collection of a greater quantity of feathers (Bedard et al. 2008). Whether birds bred primarily for the production of foie gras should be used in down production is another ethical issue that manufacturers of down products must address. Certainly, ethical down production remains a difficult issue and the greater awareness of the public now force manufacturers to better track their supply chains. Mountain Equipment (UK) are one such manufacturer who have started a traceability system for their down products (Mountain Equipment 2012), enabling the consumer to determine the source of their down. The North Face (USA) and Patagonia (USA), two of the world's largest outdoor equipment manufacturers, have recently introduced their own ethical sourcing procedures (Gunther 2014). Though the outdoor industry is a very small part (less than 1 %) of global down production and therefore lacks strong leverage of the supply chain, it is keen to promote best practice in down production and animal husbandry (Baseley 2014).

Regardless of the traceability and transparency of a down supply chain, down comes from birds that are bred for meat: either the birds are plucked live, or killed and plucked afterwards. For this reason, some vegans or other consumers may be averse to buying down products and may source synthetic alternatives instead.

1.3.1.4 Eider down

One type of down is not obtained from domestic birds: eider down comes from eider ducks, seabirds that inhabit temperate and arctic zones (Kear 2005b) and winter on subarctic or arctic shores (Jenssen et al. 1989). There are four species of eider (Ogilvie 2005), and of these the common eider is the most widespread and is the species from which down is harvested most frequently (Bedard et al. 2008).

Most eider down is sourced from Iceland. Eider farming, initiated by the Vikings (Todd 1996), has gone on there for 1000 years (Bedard et al. 2008), and the birds have been partially-protected since 1702 and completely-protected since 1900 (Todd 1996). Domestication of these seabirds is not practical and because of their protected status, eider birds' down cannot be harvested in the same way as goose or duck down. Eiders nest on land, and the female birds remove some of their feathers to expose a brood patch to warm their eggs (Bedard et al. 2008). When nests are unoccupied, skilled collectors remove some of the down from these nests. This is painstaking work, as only 7-15 g of down is obtained from each nest (Todd 1996; Bedard et al. 2008) and this contributes to the very high cost of eider down (£512 per kg in 2002 (Kear 2005a), now almost certainly much higher). Iceland's total annual production of eider down is 3000 kg (Kear 2005a) and yearly worldwide production is approximately 5000 kg.

Because of the method of collection, eider down tends to be very clean and contains little of the body-attachment part of the feather (Loconti 1955) that may result from plucking of carcasses. It is also claimed to have a total absence of flight feathers (Bedard et al. 2008), which is quite distinct to down from geese or ducks. Eider ducks have evolved to live on water, and it has been suggested that the highly-disruptive forces of water on a bird's feathers may lead to thicker and shorter feathers in seabirds (de Vries & van Eerden 1995), though this has not been verified and it is unclear whether these adaptations would occur in down feathers, as they are protected by the flight feathers from most mechanical disruption.

The extreme cost and rarity of eider down means that it is very unusual for garments or equipment in the outdoor industry to utilise it. Indeed, just one garment (Black Diamond 2014) from all leading outdoor manufacturers could be found that was filled with eider down. Most eider down is used in luxury bedding.

1.3.2 The structure of keratin fibres

The structures of keratin fibres, including feathers, have been studied for hundreds of years. The first detailed investigations were by Hook in 1665 when he used a microscope to study the structure of wool (Phan 1991).

All feathers, including down feathers, are made primarily of keratin (Bradbury 1973), the second-most-abundant biopolymer found in animals, after collagen (McKittrick et al. 2012). Keratins are a family of proteins that occur in higher vertebrates such as birds, mammals and reptiles. They vary widely in their appearance - down, wool, hair, horn and scales are all keratinous - but each act as barriers between the animal and its environment (Bradbury 1973; Briki et al. 2000). They are not vascularised and as such are regarded as 'dead' tissues (McKittrick et al. 2012).

Keratins cannot be grouped together by amino acid content, morphology or molecular structure because they are too disparate (Mercer 1961) and there have been numerous attempts to define them. Characteristically, keratins contain cysteinyl residues (Fraser & MacRae 1979) (shown in Figure 1-9) and these oxidise in the final stages of biosynthesis to create a network of disulfide bonds, as shown in Figure 1-10. As a result, in this thesis, keratins are to be defined as "protein[s] stabilised by disulfide cross-linkages" (Mercer 1961).

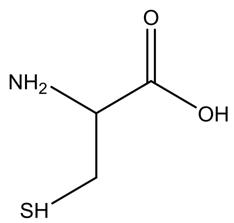


Figure 1-9 - cysteine molecule

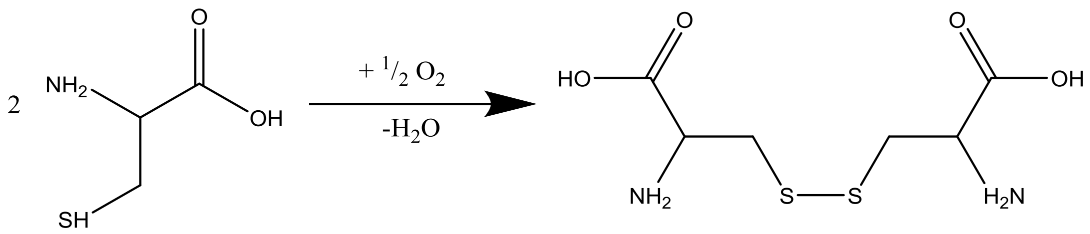


Figure 1-10 - oxidation of two cysteine molecules to form a disulfide bridge

The ground-breaking work of Astbury and co-workers (Astbury & Woods 1930; Astbury & Street 1932; Astbury & Woods 1934; Astbury & Woods 1932) at the University of Leeds divided keratins into three groups (four groups if amorphous is included (Briki et al. 2000; Fraser et al. 1972)) according to their high-angle X-ray diffraction patterns. Astbury cautiously labelled the diffraction patterns from native wool and hair as α (Astbury & Street 1932); those from stretched wool and hair as β (Astbury & Street 1932); and feather was found to be a "rather bewildering elaboration" of the β form (Astbury & Marwick 1932). The α, β, and feather labels have remained to the present day.

All mammalian keratin is α-keratin (MacLaren & Milligan 1981), which can be further subdivided into 'hard' keratins that include nails, hair, claws, beaks and hooves and horns; and 'soft' epidermal keratins that form the outermost layer of skin (Parry 1996). The terms 'soft' and 'hard' for keratin subsets have previously been criticised (Fraser & MacRae 1979) for their presumptions about mechanical properties but the nomenclature has remained. The properties and composition of α-keratin structures have been studied extensively (Kitchener & Vincent 1987), with wool being perhaps the most-thoroughly investigated (Wortmann et al. 2007) owing to its commercial importance in the early 20th century. Also, recent research in the cosmetics and hair-care industries has led to a detailed understanding of the structure of hair.

β-keratins are not naturally-occurring and are produced when α-keratins, which consist of unstretched protein chains in their native forms, are stretched under specific conditions (Cao 2002). For example, in wool, the β-pattern emerges at 25 % extension, and by 70 % extension almost completely replaces the α-pattern; there are no intermediate values (Onions 1962). β-keratins might be thought of as a halfway-point between the structure of α and feather keratins (Parry 1996) but unfortunately (Fraser et al. 1972) many authors refer to feather keratins as being β-keratinous despite the differences in their diffraction patterns.

Feather keratin describes feathers, scales and also parts of the beaks and claws of birds (Parry 1996) and is also remarkably similar to reptilian keratin. Far fewer studies exist into feather keratin than α-keratin and there is little information available on the internal structure of feathers; only one paper has been found that describes the internal structure of down. Gao et al. (Gao, Yu & Pan 2007b) found that the cross section of goose down was ellipse-shaped and they identified an epicuticle film, a cuticle, a "skin layer" and a cortex, analogous to the structures found in wool (Marshall et al. 1991). However, there was some information missing from the paper, such as detailed sample preparation methods, scale bars, and individual structures in the images. The images were small and not easy to interpret. In addition, only goose down was studied; duck down was not reported. Certainly, there is great need for these materials to be studied in greater detail and because of the lack of information surrounding the structure of down, and to aid comparisons in later chapters, a review of the structure of wool and hair will be presented here. As both fibres are keratinous there is expected to be significant overlap in their internal morphology and properties.

1.3.2.1 The structure of wool and hair (α-keratinous fibres)

Wool's internal structure is complicated. It is a semi-crystalline polymer consisting of crystalline microfibrils embedded in an amorphous matrix (Huson 1991) and has three main components (Hock & McMurdie 1943): the cuticle, cortex, and medulla. The cortex and cuticle are shown alongside some of their subcomponents in Figure 1-11:

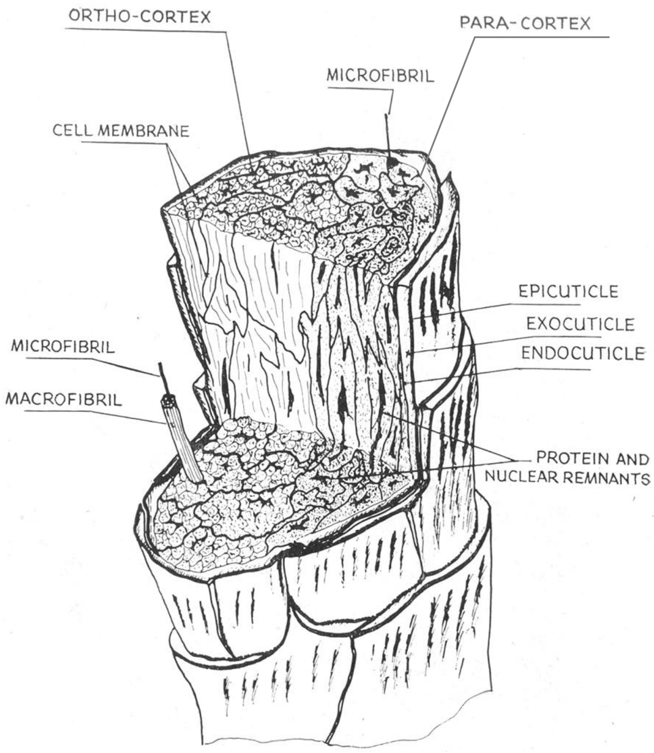


Figure 1-11 - the main features of wool's cuticle and cortex (adapted from Onions (1962))

The differences in chemical structure between these sub-components are small, so the differences in their properties are derived mainly from their morphological differences (Hock & McMurdie 1943). The ground-breaking 1959 paper by Feughelman (1959) was the first to assert the idea of keratin structures being analogous to non-absorbent cylinders embedded in a water-absorbing matrix, and much of the work in this area has stemmed from this premise. It is now well established that wool materials can be considered a two-phase composite of a viscoelastic and amorphous matrix with α-helical filaments - microfibrils that are unaffected by water (Postle et al. 1988) - embedded in it (Wortmann et al. 2007). Research into hair has yielded similar conclusions (Wortmann et al. 2006). The α-helices form filaments in a coiled-coil rope structure (Crick 1953) (this discovery was quite controversial between Crick and Pauling, two of the 20th Century's greatest scientists, but was vital in the 1953 determination (Watson & Crick 1953) of the structure of DNA).

Wool's microfibrils are approximately 7 nm in diameter (Filshie & Rogers 1961) and this is thought to be consistent across all mammals (Jones et al. 2006). They group together in units of approximately 0.5 µm diameter (Feughelman 1997) which are termed macrofibrils. Macrofibrils do not seem to vary considerably from one keratin to another, though the matrix surrounding them varies in composition (Feughelman 1997).

The structures found in wool are summarised on the next page in Figure 1-12:

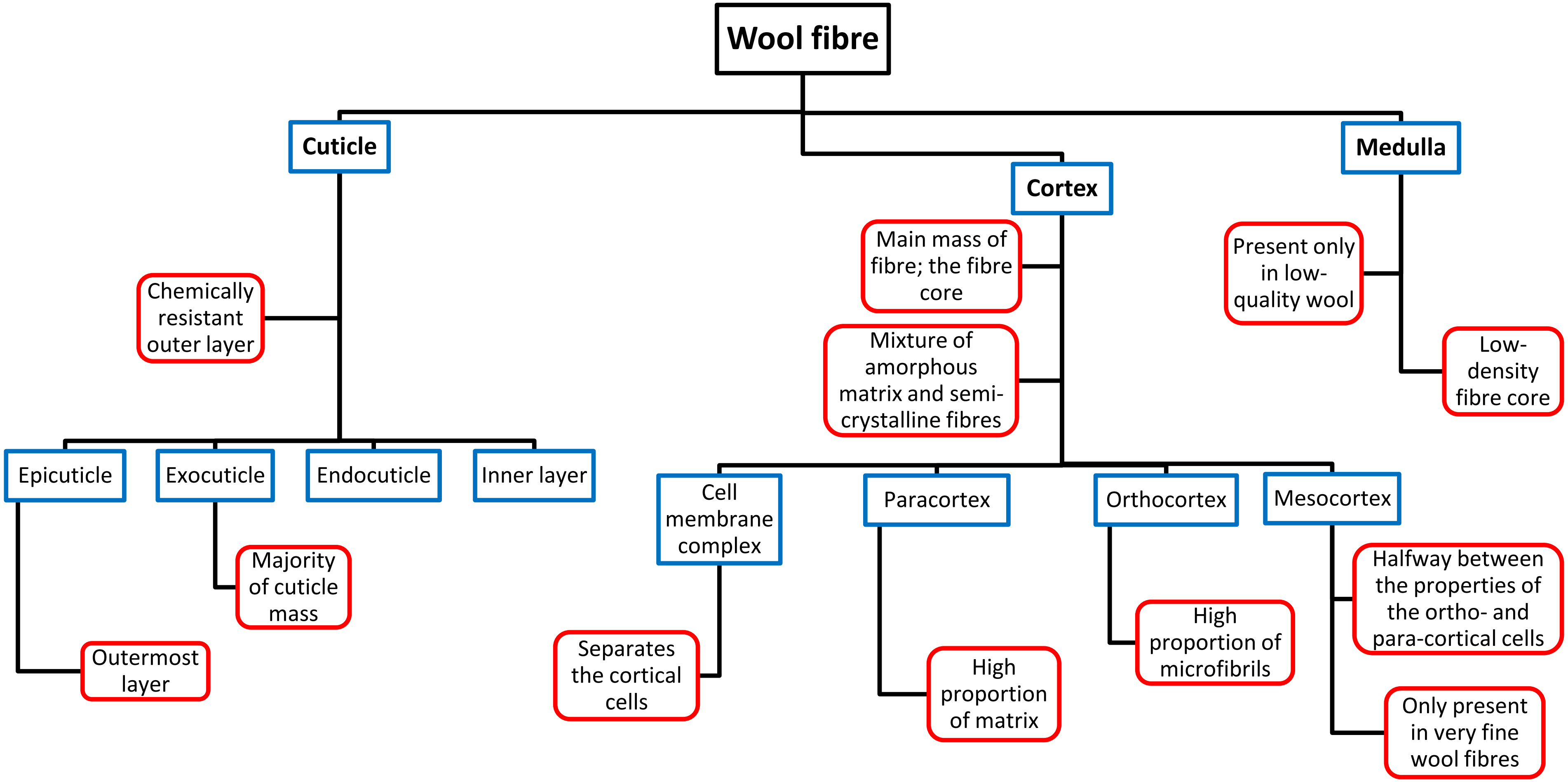


Figure 1-12 – diagram describing the major structures in a wool fibre
Blue boxes indicate individual structures and sub-structures in the wool fibre; **red** boxes describe these structures

1.3.2.1.1 The cuticle

The cuticle is the chemically-resistant layer of sheet-like cells that protects the fibre from external influences (Phan 1991). It is usually one cell thick and surrounds the wool fibre (Church et al. 1997). Cuticle cells are thought to be amorphous (Hock & McMurdie 1943) and non-fibrous (Woods 1938), and this theory has been reinforced by the small quantities of helix-forming amino acids in their structure (Church et al. 1997). The cuticle has numerous subcomponents, listed in Figure 1-12, which are chemically distinct: the epicuticle and exocuticle (the two outermost layers) are both sulfur-rich while the endocuticle is heterogeneous and non-keratinous (Phan 1991). The endocuticle is made of once-living cuticle cells and is the most well-defined layer in the cuticle (Bradbury 1973). Cuticle cells are thought to be “restricted to wool and hairs” (Marshall et al. 1991).

The cuticle is thought to play little roll in the bulk longitudinal properties of a fibre, but its role in bulk torsional properties is large (Jachowicz 1987). The ratchet-like structure that the cuticle imparts to the outer surface of hair and wool anchors the hair in the follicle and help expel debris and dead cells from the coat (Moncrieff 1954). To reinforce the theory of the cuticle helping keep the fibre in place, the cuticle of a porcupine’s quills are arranged the opposite way to wool’s so that the quills anchor in a predator’s skin (Moncrieff 1954).

Anionic detergents barely affect the mechanical properties of keratinous hair, and this is thought to be because of the protective role of the cuticle (Jachowicz 1987) and it has even been suggested that the cuticle’s chemical properties may adapt to its environment (Maxwell & Huson 2005).

1.3.2.1.2 The cortex

The cortex is the main mass of the wool fibre (approximately 86.5 % (Bradbury 1973)). Like the cuticle, it is composed of different component parts (Phan 1991): the paracortex and the orthocortex; and a mesocortex may also be present in very fine wool (Bradbury 1973). The cortex is roughly cigar-shaped (Onions 1962) or spindle-shaped (Hock & McMurdie 1943; Marshall et al. 1991).

Wool’s cortical cells are usually 80-110 µm long, far greater than their width (4.5-6.0 µm (Marshall et al. 1991)) and are oriented parallel to the fibre axis (Andrews 1957; Ross 1955; Feughelman 1997), which contributes to wool’s anisotropic physical properties (Hock & McMurdie 1943). The cortical cells of hair are similarly anisotropic (approximate length 100 µm, with a largest diameter of 5 µm (Wortmann et al. 2007)).

The paracortex usually comprises 30-50 % of the total area of the cortex and is either surrounded by the orthocortex (Bradbury 1973) or present alongside it. It contains proportionally more matrix than the orthocortex (Marshall et al. 1991) and contains numerous nuclear remnants that are usually observed in a dendritic form and are the remains of the once-living cell (Bradbury 1973).

The orthocortex usually comprises more than 50 % of the cortical mass (Church et al. 1997) and in general, as the diameter of the wool fibre increases, so does the proportion of orthocortex (Marshall et al. 1991). This same tendency has been observed in hair, though the relationship is quite variable (Hynd 1989). The orthocortex is differentiated into macrofibrils which are very inconsistent in appearance: some are hexagonally-arranged and regular, some much more irregular (Onions 1962). Large defined areas of nuclear remnants are unusual in the orthocortex of wool fibres; instead the nuclear remnants tend to accumulate in intermacrofibrillar material (Rogers 1959) that forms thin lines between orthocortical macrofibrils. The orthocortex in both wool and hair (Kassenbeck 1981) has a high microfibril to matrix ratio.

The way in which the paracortex and orthocortex interact influences wool's properties: ortho- and para-cortical cells that twist around one another helically give the fibre crimp, whereas ones aligned side-by-side along the fibre axis result in a straight fibre. The clear divide between orthocortex and paracortex in a wool fibre can be seen in Figure 1-13:

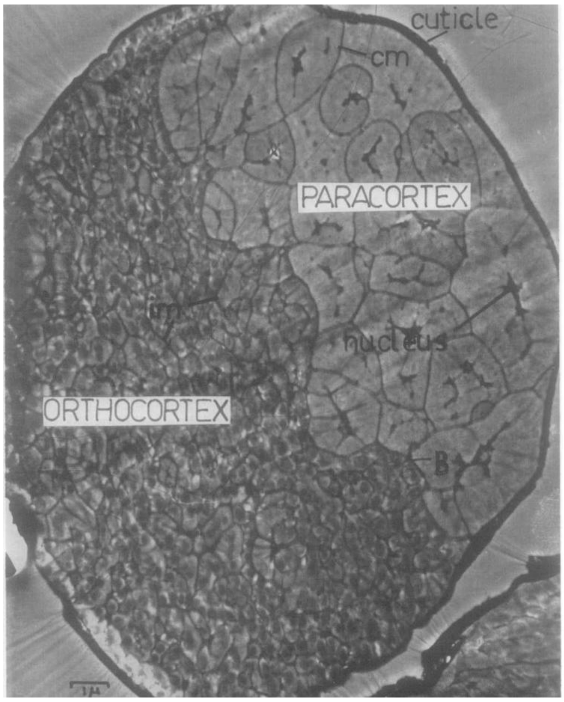


Figure 1-13 - merino wool fibre cross section
Some of the wool proteins have been extracted with thioglycolic acid. Amorphous protein between the macrofibrils is more abundant in the orthocortex than in the paracortex. Cell boundaries can be distinguished in the paracortex. Stained with osmium and embedded in Araldite. From Rogers (1959)

The cell membrane complex (CMC) is the ‘glue’ that helps hold the keratinous cells together by weaving itself around the paracortical and orthocortical cells (Bradbury 1973). Little is known about this hard-to-analyse structure (Marshall et al. 1991) though it appears to be composed of two separate materials, termed β and δ. The cell membrane complex can be seen in Figure 1-14. In hair, the cell membrane complex provides a penetration route for moisture to reach the inner part of the hair (Naito et al. 1992).

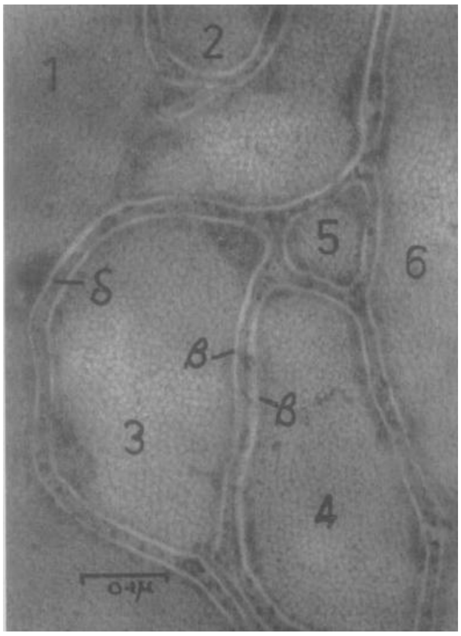


Figure 1-14 - cross section of a Lincoln wool fibre showing the cell membrane complex
The numbered areas are each part of different cortical cells. The cells are separated by layers labelled β and δ. Reduced with thioglycolic acid and stained with osmium. Araldite embedded. From Rogers (1959)

The medulla tends to be present in coarse wool fibres (Hock & McMurdie 1943) and as fibre diameter increases, so does the tendency of the fibre to be medullated (Ross 1955). It is found in the centre of these fibres (Andrews 1957) as an air-filled network of membranes and interstices (Onions 1962). It is less dense than keratin, making the resultant wool fibre open and light, but also stiff (Bradbury 1973). Its formation in sheep wool is thought to be partly hereditary as well as environmentally-influenced (Ross 1955).

In animal fur the medulla makes up a significant portion of the fibre (Franbourg & Leroy 2005) and is thought by some academics (Franbourg & Leroy 2005) to play a role in thermal insulation. For some time the hollow nature of polar bear fur led one group (Grojean et al. 1981) to believe that the bear's fur acted like optic fibres but this has now been disproven (Koon 1998).

1.3.2.2 The structure of down feathers (β-keratinous fibres)

The terminology used to describe down feathers varies considerably and can lead to confusion, so will be discussed here. For example, barbs and barbules have been described as primary and secondary structures (Wilde 2004); branch fibres and fibrils (Skelton et al. 1985); filaments and fibrillae (Loconti 1955); and as sub-branches and filaments (Yildiz et al. 2009). The terms used by the British Standards Organisation (British Standards Institute 1998b) will be used in this project. They are: the core, the central down growth; down barbs, emanating from the core; and barbules, emanating from barbs (British Standards Institute 1998b) (see Figure 1-15 and Figure 1-16). Nodes, are defined as "protuberance[s] or swelling[s] appearing on barbules" whereas prongs are “short spiny outgrowths” (British Standards Institute 1998b). Despite nodes being the standard terminology (British Standards Institute 1998b) for these structures, researchers have used numerous terms, including knars (Gao, Yu & Pan 2007b), triangular nodes (Gao, Yu & Pan 2007b), trows (Loconti 1955), and solid tertiary structures (Wilde 2004), to describe these features. Prongs have also been called crotches (Gao, Yu & Pan 2007b), hooklets (Yildiz et al. 2009), and split tertiary structures (Wilde 2004). The term ‘plume’ will be used to define individual down feathers.

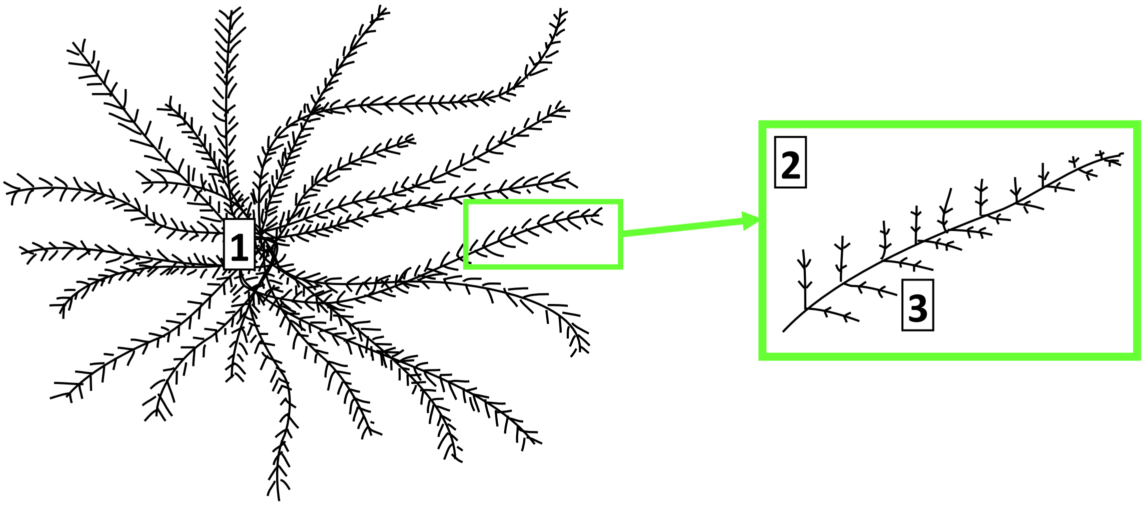


Figure 1-15 – pictorial representations of a down plume defining the major features
1 – down core; 2 – barb; 3 – barbules with prongs

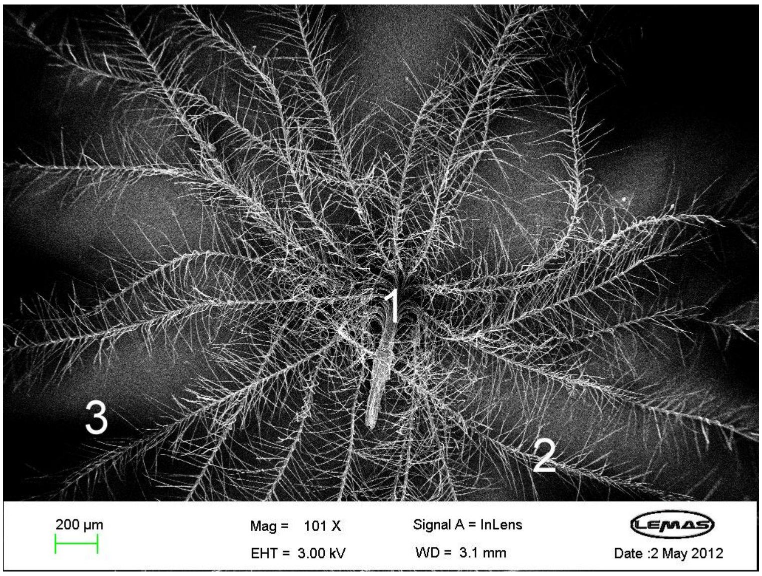


Figure 1-16 - scanning electron micrograph defining the main features of a goose down plume
1 – down core; 2 – barb; 3 – barbule

Feathers are regarded as the most specialised form of “epidermal appendage” (Fraser & MacRae 1979) or attachment to the skin (Bodde et al. 2011) and exemplify a hierarchical structure (Bodde et al. 2011). As shown in Figure 1-16, however, down feathers are quite distinct from flight feathers and have a tree-like (Yan & Wang 2009) structure with radial symmetry (Xu et al. 2007). Their symmetry is exceptional, in fact, as their overall structure has a fractal dimension very close to the 'golden mean' (Gao & Pan 2009), indicating a highly self-similar and organised structure. The arrangement of fibres in a material is crucial in determining thermal resistance (Gao et al. 2009) and by possessing fractal characteristics, down is shown to be well optimised as an insulating material. The exceptional fineness of down's barbs and barbules were well-demonstrated by Loconti (1955), who reported a number of impressive but perhaps frivolous figures gained from measuring European goose down feathers, such as there being 845 plumes per gram of down, 77,000 barbs per gram, 91 million barbules per gram, and the total fibre length in a single down cluster as 65 yards (59 metres)), which equates to 50 km in a gram of down. The unique morphology of down almost certainly impacts on its exceptional thermal properties, and as such this will be investigated in further detail in this project.

1.3.3 Mechanical properties of individual mammalian keratin fibres and down feathers

Insulating materials used in outdoor equipment must possess exceptional mechanical properties, being subject to compressive, confining (compression from all sides), tensional, and shear stresses during their use. Their mechanical properties affect their ability to insulate and withstand damage and repeated use and also affect handle and many other properties. The mechanical properties of down will be compared to those of the better-known fibres of wool and hair.

As discussed in section 1.3.2, keratins are filament-matrix composites. The matrix tends to distribute any applied stress evenly over the filaments and this prevents the propagation of cracks from local imperfections (Fraser & MacRae 1979). The fibre's sum mechanical properties are influenced by the filaments' and the matrix's properties, as well as the adhesion between them and because keratin's filaments are stiff, aligned along the fibre axis, and possess a high aspect ratio, they contribute to a very strong material. Strong adhesion of the matrix to the filaments is ensured by a network of disulfide bonds (McKittrick et al. 2012).

1.3.3.1 Tensile testing of α-keratin fibres

Tensile testing is often used to determine the strength of a material and numerous studies of the tensile properties of wool and hair have been reported. The tensile properties of wool fibres follow a distinct pattern, as shown in Figure 1-17 (McKittrick et al. 2012):

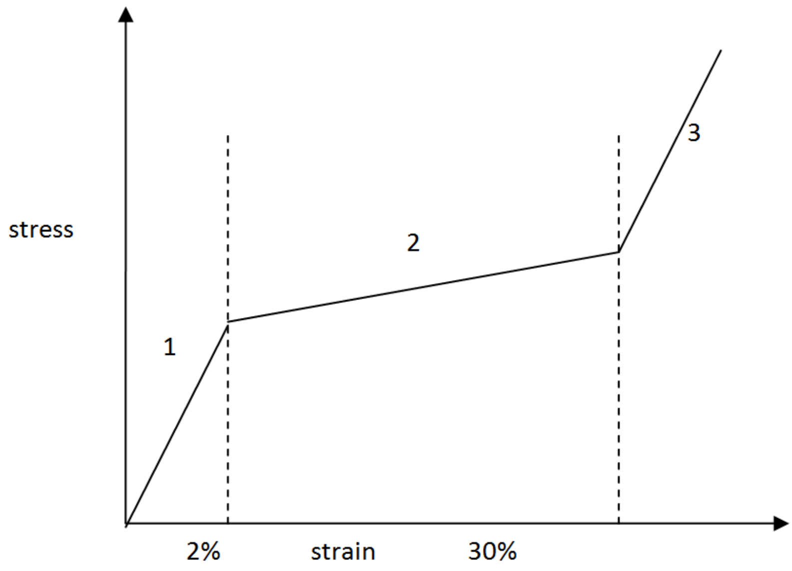


Figure 1-17 - approximated stress-strain character of a wool fibre undergoing extension
1 = uncrimping region and linear elastic region (viscoelastic (Feughelman & Robinson 1971)); 2 = yield region – α to β transformation occurs; 3 = strain hardening of the matrix or opening of remaining α-helices. Fracture occurs between 50 - 60 % strain (McKittrick et al. 2012)

In Figure 1-17, region 1 includes the uncrimping and elastic extension of the fibre and this portion is Hookean, meaning that the material can return to its original unstressed state once the load is removed. In region 2 the stress is concentrated on the filaments in the fibre and as the α-helices begin to stretch (Astbury & Street 1932) the modulus decreases. Region 3 occurs when the α-helices have recrystallised as β-sheets and can extend no further. Thus, loading begins to occur through the matrix and as this hardens the modulus increases.

Wool and other keratinous composites may have physical properties not necessarily possessed by either of the individual components (Fraser & MacRae 1979), though some of their properties result from a combination of those of their subcomponents. For example, the modulus, E, of a fibre reinforced composite material comprising a filament, *f,* and matrix, m, can be described by Equation 1-2 (Harris 1980; Fraser & MacRae 1979):

$$E = \eta E_f V_f + E_m(1 - V_f) \qquad (1\text{-}2)$$

where E is the Young's modulus in GPa, η is the efficiency of reinforcement (value of 1 if the filaments are perfectly aligned, value of 0 if filaments are arranged randomly), and V is volume fraction. The subscript *f* and *m* represent contributions from the filament and matrix respectively

Hard α-keratins generally have an elastic modulus of 1-1.52 GPa at 60-65 % relative humidity (Fraser & MacRae 1979), though the elastic modulus of human hair has been measured as high as 3.89 GPa (Robbins 1994). Such variability is commonplace in these natural materials.

1.3.3.2 Tensile properties of down and feathers

The tensile properties of various feathers have been reported by Bonser (Bonser 1996) and he and Dawson have also carried out tensile testing on down barbs, finding that goose down had a Young's modulus of 1.31 GPa and duck down 2.21 GPa, from a study of 29 goose down barbs and 24 duck down barbs (Bonser & Dawson 1999). Following statistical analyses (factorial analysis of variance, ANOVA) these feathers were determined to be significantly different in terms of Young's modulus. Another study found duck down barbs to have a Young's modulus of 2.07 GPa (Bonser & Farrent 2001). As in other keratins, environmental conditions can affect the mechanical properties of down, and in the tensile testing of duck down barbs, tensile strength and modulus decreased as humidity increased (Bonser & Farrent 2001).

Feathers are not as extensible as wool or hair. They break at approximately 12 % extension (Fuller et al. 2013) and this is due to their filaments being composed of β-sheets that cannot extend in the same manner as α-helices.

1.3.3.3 Compression and bending resistance of down feathers

The compression and bending resistance of down feathers is extremely important in determining their thermal properties: bulkier materials trap more air and are therefore more insulating. If a fibrous material cannot resist compression nor recover from compression then it will be a poor insulator for use in applications such as sleeping bags or cold-weather clothing.

The compression resistance and recovery of a fibrous material is related to its bending and buckling properties (Dunlop 2008) but carrying out bending testing on very fine fibres (Scott & Robbins 1978) such as down barbs is challenging. Scott and Robbins presented a method of determining the bending resistance of human hair, whereby masses were attached to two ends of a length of hair and the hair then draped over a pin; the distance between the two fibre ends was taken as a measure of stiffness (Scott & Robbins 1978). However, this method used a 5 cm gauge length that would be unsuitable for down (the barbs are too short) and the handling of down feathers is significantly more challenging than the handling of hair. Indeed, previous researchers (Butler & Johnson 2004) have tended to carry out tensile testing on feathers rather than bending tests because of tensile testing's relative ease of operation. However, the compression resistance of individual down fibres has been tested by Gao et al. (2010) who used a 2 mm sample length, thus removing the effect of buckling and so the results represented compressive yield instead. This sample length is ineffective for demonstrating the compression resistance of a full-length down barb that may be ten times longer than the analysed length. Gao's (2010) results showed that the individual down fibres had a compression rigidity of one tenth that of wool, and the bending work was also much smaller.

The compression hysteresis of individual down barbs was very low, implying an excellent recovery from compression. Gao (2010) suggested that this may be because of down's compact crystal structure, though this compact crystal structure was neither referenced nor supported by any evidence given in the paper. Wilde (Wilde 2004) has also carried out work on the characterisation of down's bending properties and suggested that the crystal structures of barbs and barbules might be different, thus accounting for the discrepancies in the way they bend but the work did not state the quantitative differences in their properties. There is still significant room for further research into the relationship between down's bending properties, its crystal structure and morphology, compression resistance of down feathers, and the relationship between the compression resistance of individual down plumes and that of larger masses of feathers, particularly considering the important bearing these factors have on down's thermal performance.

1.3.4 Sorption and wetting properties of down and keratins

For many years, mountaineers expecting sustained wet weather have avoided using down equipment because of its relative ineffectiveness when wet. However, down in its source-state (on birds, underneath the flight feathers), remains insulating in all weathers and when the bird is either on or under-water. Clearly, the way down interacts with water is complex.

1.3.4.1 Effect of wetting on down's performance

Many researchers have asserted that the thermal properties of down feathers are much reduced when wet. Skelton et al. (1985) stated that a "dramatic deterioration" is seen in a feather's thermal resistance when wet and that this is because of their lower modulus, and interstitial water exacerbating structural collapse. Bonser and Farrent (2001) also state that down feathers retain water when wet and clump together, making them less effective insulators, though in their paper they reference work carried out by McCafferty et al. (1997) on live owls, rather than down taken from geese or ducks, which may react quite differently. Two further publications reference down's decrease in thermal performance when wetted (Kasturiya et al. 1999; Farnworth & Osczevski 1985) but do not give quantitative information regarding this change in performance.

Gibson (1990) has carried out work regarding down's wet thermal conductivity. He submerged down and synthetic samples in water, compressed them, and then tested their thermal conductivity on a hotplate. When comparing the dry and the wet insulating materials, wetted down increased in thermal conductivity more than the synthetic materials did but down's thermal conductivity remained relatively low even when wet (0.38 Btu in. hr^{-1} ft^{-2} $^{o}F^{-1}$ (Gibson 1990), equivalent to 0.055 W m^{-1} K^{-1}). This was an increase in thermal conductivity of

16.8 % and the thermal conductivity of the wet down actually remained less than that of the two dry synthetic battings (Gibson 1990). The large decline in thermal resistance that some people (Eastern Mountain Sports n.d.) might expect from wet down may not have been observed in Gibson's test because the down may have failed to be sufficiently wetted. Indeed, it is relatively difficult to test completely saturated down on a hotplate because the down will inevitably dry during the test, making experimental design difficult.

Farnworth and Osczevski (1985) of the Canadian military have also carried out work on the wetting of down samples. They found that samples "in typical sleeping bag construction" filled with down absorbed less weight of water than the synthetic insulations they tested (one of polypropylene, one of polyester) but the down lost 80 % of its thickness; the synthetics retained 80 % of their thickness. The drying on the hotplate, which simulated body temperature, indicated that the down dried quickly but with a subsequent high rate of heat loss due to evaporation (Farnworth & Osczevski 1985).

Van Rhijn (1977) carried out tests on various feathers by soaking them in water for different durations and at different temperatures. It was found that the surface tension of the water greatly affected water absorption. This might have relevance in outdoor equipment when oils from the body or surfactants from external sources such as detergent or petrol, come into contact with down. If this were to occur, the down would wet more easily.

Down feathers are fibrous and comparisons with the sorption properties of fabrics are useful in gaining further understanding of the manner in which they wet. In a fabric, if capillary water is present in the interstices between fibres then these fibres become attracted to one another by the surface tension of the water (Morton & Hearle 2008c). In addition, when saturated fabrics are dried the individual fibres remain drawn together (Morton & Hearle 2008c) unless external force is applied. These effects are assumed to also be present in wet down and contribute to its willingness to collapse when wet and its tendency to remain clumped even when dry: a drying procedure that provides mechanical agitation is thus assumed to aid the restoration of wet down's bulk.

The moisture regain of down has been studied and compared to wool, cotton and polyethyleneterephthalate (PET) in five different temperatures and relative humidities (Gao, Yu & Pan 2007b). In the study, down gained less water than cotton or wool, but more than PET fibres. At low temperatures, down absorbed the least moisture when normalised against the other results (Gao, Yu & Pan 2007b).

1.3.4.2 Surface properties of down

The surface properties of down are crucial to the way it interacts with water and other chemicals: all of the contact that down has with its environment occurs through its surface. As such, an appraisal of down's surface chemistry is beneficial.

1.3.4.2.1 Effect of processing on down's surface chemistry

To remove undesirable smells, commercially-available goose and duck down is cleaned of its fats and oils before it is sold (Bonser & Dawson 1999), and eider down is sterilised (Bedard et al. 2008). However, complete removal of these fats and oils is difficult (Rijke 1970), and some may remain in the commercial product, which might contribute towards down's hydrophobicity (commercial down does not sink in water (Fuller 2012; Farnworth & P. Dolhan 1983)).

Unwashed goose and duck feathers are coated in different fats (Odham 1967). Goose feathers have two different fatty coatings, whereas duck feathers have numerous types. These fats are shown in Figure 1-18 and Figure 1-19 (Odham 1967):

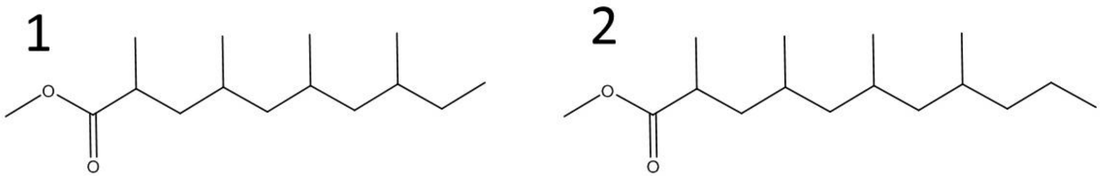


Figure 1-18 – waxes present in goose feathers
1: methyl 2,4,6,8-tetramethyldecanoate, 93.6 % abundance; 2: methyl 2,4,6,8-tetramethylundecanoate, 6.4 % abundance. IUPAC molecular names generated by Chemspider (Royal Society of Chemistry 2014)

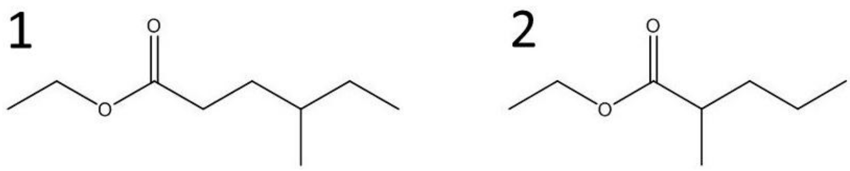


Figure 1-19 – the two most abundant waxes present in duck feathers
1: ethyl 4-methylhexanoate, 33.7 % abundance; 2: ethyl 2-methylpentanoate, 40.5 % abundance. IUPAC molecular names generated by Chemspider (Royal Society of Chemistry 2014)

1.3.4.2.2 The hydrophobicity of down feathers

Down feathers are hydrophobic in their natural environment and birds rely on this coating to maintain sufficient insulation. Even eider ducks, which possess extremely effective thermoregulation (Steen & Gabrielsen 1986), become hypothermic very quickly if their coats are covered in crude oil (Kear 2005a), which negates the water repellence of their feathers (Jenssen & Ekker 1991).

Some academics (papers listed in Dyck (1985)) previously thought that the preen oils from birds' urological glands were the source of flight feathers' water repellence, though many now claim that the oil barely contributes to either buoyancy or repellence (Dyck 1985; van Rhijn 1977). The actual function of the oil might be to retain feather elasticity (van Rhijn 1977) and it is now thought that it is the constant distances between the feathers' barbules that leads to their water repellence (Dyck 1985). Indeed, flight feathers are much more repellent than a flat keratin surface owing to the air gaps between the barbs (Dyck 1985). However, washing of down has been shown to decrease its water repellence (Farnworth & P. Dolhan 1983) and this reinforces the theory that preen oils do contribute to hydrophobicity. In summary, though there is doubt over the role of the urological gland and preen oils, it is certain that the structure of feathers makes a difference to their repellence (Dyck 1985). Surface roughness is vital in determining the interaction of a surface with water (Zhang et al. 2008; Ma & Hill 2006) and is thought to act as an 'amplifier' of either hydrophobic or hydrophilic character. This is demonstrated in Figure 1-20 and Figure 1-21:

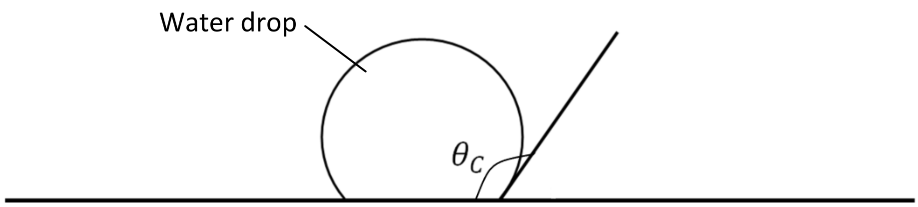


Figure 1-20 – a water droplet on a flat hydrophobic surface
The contact angle, θ_C is greater than 90 °, indicating hydrophobicity

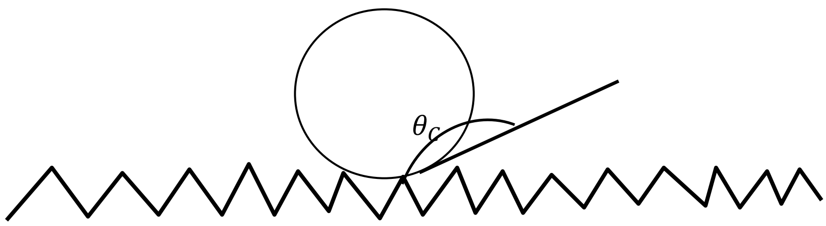


Figure 1-21 – a water droplet on a rough hydrophobic surface
The contact angle θ_C is correspondingly greater than that shown in Figure 1-20, indicating a greater degree of hydrophobicity

The even-spacing between flight feather barbs that contributes to hydrophobicity is shown in Figure 1-22. The same consistency is not observed in down feathers and their geometry's ineffectiveness in shedding water may be the reason why they seem to be particularly reliant on their oils to maintain hydrophobicity.

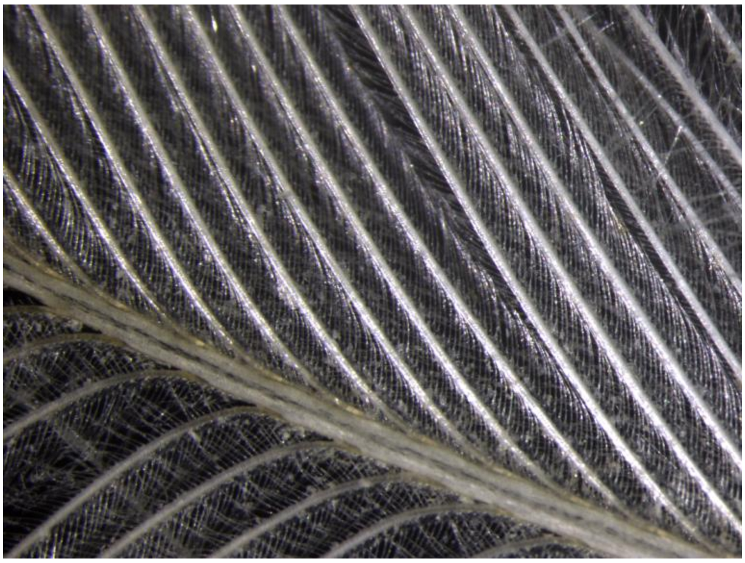


Figure 1-22 – a flight feather from a duck showing the even spacing between barbs and barbules that contribute to hydrophobicity
Image taken using a calibrated Leica M205 C microscope and processed using Leica Application Suite v4.1

Wool's surface also possesses some hydrophobic properties (Lo Nostro et al. 2002), but wool can still absorb a significant quantity of water (Hind 1948) in a manner similar to down. This is thought to be as a result of its absorbent internal structure (Alexander et al. 1963). Human hair, similarly, has both hydrophobic and hydrophilic components (Kitano et al. 2009), and just like down, despite being denser than water, hair floats on water (Franbourg & Leroy 2005).

1.3.4.2.3 Commercially-available water-repellent or 'hydrophobic' down

In attempts to increase the water repellence of down while maintaining the low odour of washed down, some commercial down is treated with hydrophobic coatings. This modified down has become known as water repellent or hydrophobic down, and the term 'hydrophobic down' is used from this point.

The Tan-O-Quil-QM treatment (Cohen 1968) is probably the first man-made hydrophobic treatment for down. It was reported by the US Army Research Laboratories in 1968 and was still being used in 1990. It has been described as the "most outstanding development in the last hundred years in the field of down and feathers" (Cohen 1968) and was adopted commercially. The treatment uses a chrome tanning agent and chrome complex to increase water repellence (Cohen 1968). It was originally developed in an attempt to modify chicken feathers, enabling them to be used in military sleeping bags instead of down feathers, but it was unsuccessful because of the difference in the geometry of the feathers and the small size of the feathers from young commercial chickens (Cohen 1968). It was also noted (Cohen 1968) that the fill power, which reflects how much volume down occupies and is discussed further in section 1.3.5.3, of goose and duck down increased following the treatment, though this was not a permanent improvement. The equilibrium moisture content of the treated feathers was also lower than in untreated down (Cohen 1968). However, the Tan-O-Quil-QM treatment has lost favour owing to the stiffness of the treated feathers, the concerns regarding the disposal of harmful effluents, and the chemical attack on the processing machinery by the aggressive chrome salts (Fleet & Hewlinson 1985). There are numerous other heavy-metal-based treatments for down that have been patented, including the Aversion process (Fleet & Hewlinson 1985) developed by Henkel GmbH (Germany), which uses a chromium compound and a mordant; and the Nocar process (Fleet & Hewlinson 1985) (Peter Kohl, Germany) which uses an aluminium salt of a fatty acid to achieve hygienic properties.

In the 1980s a patent detailing a fluorocarbon (fluoroalkyl acrylate) finish for down feathers was filed (Fleet & Hewlinson 1985) whereby the treatment was applied then dried and activated in temperatures exceeding 60 °C. Following a washing cycle, this treated down dried faster than untreated down and faster than down treated with chrome tanning agents (Fleet & Hewlinson 1985). Silica-coated down (Farnworth & Dolhan 1983) has also been developed and was found to decrease the water uptake of down, both when loose and when enclosed in nylon face fabrics.

Recently, the outdoor industry has realised the potential benefits of hydrophobic down for use in insulated clothing and sleeping bags and it is now very common in the marketplace. Berghaus (UK) was the first company in recent years to release such products commercially (Berghaus 2015). Since then, specialist suppliers of hydrophobic down such as Downtek (USA) (Sierra Designs 2012; Downtek 2012) have begun trading. Other manufacturers, for example Rab (Equip, UK), have collaborated with companies such as Nikwax (UK) (Nikwax 2015) that specialise in wax-based repellent treatments, to develop a product claimed to be more environmentally friendly than those based on fluorochemical technologies. More recently, Patagonia (USA) (The Gear Caster 2012) have released a limited-edition jacket filled with down treated by plasma and a patent (Pavlos et al. 2011) on the treatment of down using plasma has been filed. However, at present, plasma treatment is considered by many as too expensive for chemically-treating down.

1.3.5 Bulk properties of down assemblies

Down is never used in commercial products as individual feathers. Thus, to understand how down behaves as a bulk material and how the interactions between individual down feathers influence performance, down assemblies must be analysed.

1.3.5.1 Assembly composition

There are four components in a commercial down assembly: air, water, down feathers and flight feathers. Each of these components has vastly different thermal and mechanical properties and the ratios of these four materials have large implications for the overall properties of the bulk material. The structure of these assemblies are difficult to investigate as many observational techniques disturb the system during study (Gao et al. 2009) but they have been studied (Gao et al. 2009) by using micro-CT (micro-computed tomography (Ho & Hutmacher 2006)) which disclosed the pore size distribution in the structure of the down assembly. This was not as evenly-distributed as initially suspected, instead being influenced by local factors such as the diameter, length, and positions of barbs.

1.3.5.2 Compression resistance and recovery

The compression properties of a down insulating material are particularly important in outdoor products, where the conflicting interests of maximum volume while in use, and minimum volume when packed away, must be balanced: during use, clothing and sleeping bags must resist compression so any air remains trapped inside the assembly, but when storing the product it must compress as small as possible so as to require minimal volume in the rucksack. Compression recovery is thus important in ensuring that a stored insulated material can insulate fully once it is needed.

The resistance of a down assembly to compression, and its ability to recover, has been previously tested (Gao et al. 2010). Though the bending recovery of individual down barbs had been found to be greater than in wool fibres, down assemblies had a greater hysteresis in compression than wool, indicating inferior compression recovery (Gao et al. 2010). This was attributed to the formation of a frame of intertwined and embedded fibres. In other work (Gao, Yu & Pan 2007b), researchers determined the densities of down, polyethyleneterephthalate (PET), wool, and cotton assemblies under different compressive loads and the compression recovery was also measured. Of the four materials, down was found to recover most effectively from compression (Gao, Yu & Pan 2007b). The difference to the previous result (Gao et al. 2010) can be rationalised by the difference in experimental design: in this experiment, the samples were agitated as the load was removed, and this ‘unlocked’ the entwined barbs, barbules, and nodes. This had not occurred in the other experiment (Gao et al. 2010).

Gibson (1990) compared the compression resistance of down and synthetic battings. He subjected the down to far greater pressures than the other studies did as he sought to exceed the maximum pressure that a supine person would impart on a sleeping bag (approximately 200 g cm^{-2}). Gibson (1990) stated that a lower work to compression in a sleeping bag filling material was desirable as it aided storage of the sleeping bag, but this is not necessarily true, as it implies that compression of the insulation would occur very readily when in use which is not desirable, as a reduction in thickness would reduce the ability of the material to trap air, thus increasing heat loss. When comparing the down and synthetic insulations, Gibson (1990) noted that down compressed isotropically, whereas synthetic battings are much more compressible in x and y directions than in the z direction (through the thickness of the batting). This is relevant in sleeping bags or jackets as fillings are compressed in one dimension (the z direction) during use but when they are put into a 'stuff-sack' in which they may be carried, they are compressed in all directions so as to minimise their volume.

The International Down and Feather Laboratory (IDFL), the world's biggest down and feather testing laboratory, have stated (IDFL 2011) that down and feathers are supremely resistant to compression and that they will return to their uncompressed state even after the application of extreme pressure and that only the use of chemical or thermal treatments can permanently affect bulk density (IDFL 2011). However, in further testing carried out by the IDFL (2010c) there was an average decrease of 13 % in fill power when down products were compressed then shipped from China to the IDFL testing facilities in America. This seems contrary to their aforementioned conclusions, though it is thought that the fill power value would recover to its pre-conditioned value, given sufficient agitation and the correct environmental conditions.

1.3.5.3 Fill power testing

In the outdoor industry, down quality is usually assessed and sold using two main measurements: the down to feather ratio, and the fill power. Typical down to feather ratios are 93:7, 90:10, or 80:20 for high quality downs used in outdoor equipment; the greater the ratio of down to feather, the greater the quality.

Fill power is defined by the IDFL (2010e) as "the volumetric measurement of a specific amount of down and feathers subject to a standard compression weight" and it is advertised on almost all down products in the outdoor industry and is regarded by many people as the most important factor in determining the quality of down. There are different international standards for testing fill power (IDFL 2011), but each rely on the same fundamental methodology, whereby a down sample is conditioned, weighed, and put in a large cylinder. It is then aerated and a plunger applied until it rests on the down. The height of the down under the plunger is then measured and the volume of down occupied is calculated and divided by the sample mass to produce the value of fill power. The only known exception to this methodology and reporting of results is in the European Norm BS EN 12130: "Feather and down - test methods -determination of the filling power (massic volume)" (British Standards Institute 1998a). This test describes the "filling power" as the "height of the volume occupied by filling material… expressed in millimetres" and the massic volume ("volume occupied by a given mass of filling material… expressed in cubic centimetres per gram") is instead used to define volume per unit mass (British Standards Institute 1998a). The standard does not use the phrase "fill power" at any point. The discrepancy between the IDFL's and the British Standard's wordings can cause confusion, but because of the near-universal acceptance of the term "fill power" in the outdoor industry, this phrase will be used in this work to describe the volume occupied by a mass of down.

Typical fill powers of down used in commercial products are listed in Table 1-1:

Table 1-1 - description of down fill powers

(adapted from a table from PHDesigns (2012))

Fill power (inches^3 30 g^{-1})	Description
400	Grade of down used in bedding or poor-quality sleeping bags. Often contains >15 % feather
500	Reasonable quality down used in cheaper sleeping bags and jackets
600	Good down used in high quality products
700	A rare and expensive down and the upper-limit for duck down. Only goose down of very high quality will exceed this
800	Achieved by very few down feathers. Used in premium outdoor wear and sleeping bags

Flight feathers contribute little towards fill power so a small down to feather ratio yields a low fill power, and fill power increases markedly as the down to feather ratio increases (IDFL 2010f; IDFL 2010d) because flight feathers are two dimensional and so contribute little to bulk (Wilde 2004). It has also been stated that under compression, permanent entanglement can result from the presence of feathers (Wilde 2004), though the IDFL refute this (IDFL 2011).

1.3.5.3.1 Conditioning

There are many different methods used to condition down before it is fill power tested. Box conditioning is the longest-serving method of conditioning, is still recommended in EN 12130 (British Standards Institute 1998a), and was until quite recently the method recommended by the IDFL. In this method, down is placed in a mesh-sided box of specific dimensions and kept in a controlled atmosphere. Box conditioning is cheap, easy to carry out, and intends to impart the down with a specific water regain.

More modern methods of conditioning include the use of steam, tumble-drying or water-rinsing. All of these methods of conditioning produce greater fill power values than those from box conditioning (IDFL 2010g) and some critics might argue that these methods are now favoured by industry as they yield greater fill power values, but they also prevent direct comparisons between modern results and those of a decade ago (Alpkit 2013). Steam conditioning produces the greatest fill powers of all the aforementioned methods but the IDFL asserts (IDFL 2011) that steam conditioning does not produce falsely-inflated results, and in fact most closely replicates the original fill power of the feathers fresh from processing and also replicates the increase in fill power that occurs when a wearer heats and moistens down in their clothing or sleeping bag. The IDFL also state (IDFL 2011) that steam conditioning is faster and more convenient than other conditioning methods and that it gives more reliable results.

1.3.5.3.2 Criticism of fill power testing

To the author's knowledge, fill power testing was first used by the US military in the 1950s (Weiner 1955). Despite its origin, Loconti (1955), working for the US military, criticised fill power testing in the 1960s and claimed that it did not reflect the bulking qualities of down when used as a sleeping bag filler, as down's compression resistance was not linear, so the single-mass analysis of fill power testing was not representative (Loconti 1955).

Because fill power is tested under very low pressures, there is also potential that the pressures exerted in a garment may compress downs of different fill powers to similar degrees, thus negating any difference between them. Down samples of exceptional fill power (for example, 900 or 1000 inches^3 30 g^{-1}) may be unable to maintain this extreme loft under their own weight when held in high columns.

The relationship between fill power and compression resistance at higher pressures is extremely important, has not been reviewed previously, and needs further exploration, as the outdoor industry's down supply is based almost entirely on the use of fill power as a quality control.

1.3.5.4 Heat transfer pathways in down

As discussed in section 1.2.3, the thermal properties of down feathers are extremely impressive, supposedly superior to all other fibrous insulating materials (Kasturiya et al. 1999) and considered unsurpassed when the feathers can be kept dry (Gibson 1990). In order to understand the heat transfer in a down assembly, heat transfer pathways must be explained. There are two types of heat transfer: heat transfer that occurs with no change of state, and heat transfer that occurs with a change of state (Houdas & Ring 1982). The former describes conduction, convection, and radiation; the latter describes phase-changing processes such as evaporation (Kerslake 1972). It has been stated (Skelton et al. 1985) that it is down's complex structure that helps impart its excellent thermal properties: the fine fibres blocking radiative heat transfer and the thicker fibres maintain its loft, thus trapping maximum air.

1.3.5.4.1 Conduction

Heat conduction is an energy transfer process driven by a temperature gradient between materials that share physical contact. It occurs due to vibrating molecules exchanging energy through delocalised electrons. Conduction is a dominant heat transfer mechanism in many solids and also contributes to heat transfer in still gases and liquids, though convection is dominant in these fluids (Jessen 2001). It is relevant to human thermal comfort when the body comes into contact with solids, such as when holding cold objects or standing on cold ground, and it is also an important heat transfer mechanism between adjacent clothing layers. Conduction is thought by some academics to be the most important heat transfer mechanism in clothing assuming that the fibre-volume fraction is greater than 9 %, meaning radiation and natural convection can effectively be ignored (Woo et al. 1994).

Fourier's expression describing conduction, Q_k is shown in Equation 1-3 (Houdas & Ring 1982):

$$Q_k = -\frac{k}{x}(T_1 - T_2)A_k \qquad (1\text{-}3)$$

where Q_k is the heat flow in Watts (W), k is thermal conductivity in W m^{-1} K^{-1} , x is the distance in the direction of heat flux between regions 1 and 2 in metres, T is the temperature of regions 1 and 2 in Kelvin, and A_k is the area of the regions 1 and 2 in square metres through which heat flows

Just as electrical resistance is inversely related to electrical current, thermal resistance (in m^2 K W^{-1}) is inversely related to thermal conductivity. Thermal resistance is defined as "[the] ratio of the temperature difference between the two faces of a test specimen to the rate of flow of heat per unit area normal to the faces "(British Standards Institute 2005). The relationship between thermal resistance and thermal conductivity is shown in Equation 1-4:

$$k = \frac{t}{R_k} \qquad (1\text{-}4)$$

where k is thermal conductivity in W m^{-1} K^{-1}, t is the thickness of the material in metres, and R_k is thermal resistance in m^2 K W^{-1}

The thermal conductivity values of selected materials relevant to the discussion are shown in Table 1-2. As can be seen in Table 1-2, gases tend to conduct heat poorly, liquids are fair conductors, and solids (particularly metals) conduct heat effectively.

Table 1-2 - thermal conductivity values of various materials

(Data from Jessen (2001); Lienhard IV and Lienhard V (2008); and Baxter (1946))

Material	**Thermal conductivity, k (W m^{-1} K^{-1})**
Still air	0.025
Animal fur	0.038
Sheep wool assembly	0.050
Keratin	0.192
Nylon	0.250
Water	0.600
Ice	2.215
Copper	401

Conduction in a down assembly is composed of two elements: conduction through the barbs and barbules of the feathers and conduction through the air. The contribution of the barbs and barbules is very small (Farnworth 1983) and is only significant when the fibre:volume fraction reaches approximately 1:10 (Farnworth & Osczevski 1985) (i.e. under significant compression). Thus, the resistance to conductive transfer of a material is largely due to its ability to trap air, and this increases with thickness (Farnworth & Osczevski 1985), and only if fibres are very tightly-packed might they force air from an assembly and thus increase thermal conductivity (Wang 2010). This also explains why the keratinous sheep wool assembly (i.e. sheepskin) shown in Table 1-2 has a low thermal conductivity despite keratin's thermal conductivity of 0.192 W m^{-1} °K^{-1}. It follows that the thermal properties of down's individual barbs are unimportant compared to their air-trapping efficacy, as the conductivity of individual feathers contribute very little to the overall conductivity of the assembly (Farnworth 1983; Farnworth et al. 1985). Thus, it is prudent to focus work on the structure of down assemblies rather than the inherent thermal conductivity of individual feathers.

1.3.5.4.2 Convection

Convection occurs in a fluid or between a fluid and a solid. There are two types of convection: free (Morrissey & Rossi 2013) convection and forced convection. Free convection occurs when diffusion causes circulating currents that dissipate heat; forced convection is the motion of fluids caused by an external force such as wind or waves. Convective heat transfer occurs by a displacement of matter as well as energy, making it distinct from conduction. Convectional heat flux, Q_c, is described by Equation 1-5:

$$Q_c = -h_c(T_1 - T_2)A_c \quad (1\text{-}5)$$

where Q_c is heat flux by convection in W, h_c is the convective heat transfer coefficient in W K^{-1} m^{-2}, A_c is the surface area through which heat is transferred in m^2

Convection is most apparent in a clothing system in windy conditions (forced convection), or when the wearer is moving (the 'bellows' effect, which forces air around or out of a clothing system) (Birnbaum & Crockford 1978).

It is thought that negligible to zero natural convection occurs inside a down assembly (Farnworth 1983) because of the very small air gaps that exist between individual down barbs and barbules (Rossi 2009). This is common not just to duck and goose feathers, but also to other avian coats such as penguin fur (Dawson et al. 1999). Despite the absense of free convection, forced convection caused by movement of the user of the down garment or sleeping bag, or caused by the wind, may cause convection to be a relevent heat transfer pathway through down. It is for this reason that garment and equipment designs should allow freedom of movement so the down is not unduly disturbed by the movement of the user, and that face fabrics are sufficiently impermeable to air so as to prevent unnecessary ingress of external air currents.

1.3.5.4.3 Radiation

Thermal radiation is carried by electromagnetic light of wavelengths 10^{-7}-10^{-4} m (100 nm to 100 µm), corresponding to visible light, infrared light, and some microwave radiation (Houdas & Ring 1982). It is emitted by all objects with temperatures exceeding zero Kelvin (Morrissey & Rossi 2013). Heat transfer by radiation does not require molecular contact between objects and as such can happen through a vacuum, for example between the sun and the earth. It is perhaps the most complex heat transfer mechanism but the heat flux between two surfaces can be described by Equation 1-6 (Houdas & Ring 1982):

$$Q_r = \sigma F_2'(T_1^4 - T_2^4) \quad (1\text{-}6)$$

where Q_r is heat flux through radiation in W m^{-2}, σ is the Stefan-Boltzmann constant (5.670373 × 10^{-8} W m^{-2} K^{-4}), and F_2' is a geometric factor describing surfaces 1 and 2

Radiative heat loss from a clothed person accounts for approximately 5 % of total heat loss (Torvi & Dale 1999), and can be reduced to a minimum by wearing thick clothing or by incorporating a reflective layer into the clothing, such as aluminium (Jordan 2004). This has been exemplified by Blizzard's metallised emergency blankets (Blizzard Survival 2012).

Though sometimes ignored (Farnworth 1983), radiative heat transfer through down assemblies is quite important. A study (Farnworth & Osczevski 1985) has stated that the main difference between dry insulating materials of the same thickness is their radiative properties, and that radiative heat transfer in clothing may be as significant as conduction (Farnworth 1983). The most important controllable factors in a material's radiative conductivity are fibre diameter and fibre: volume fraction. Radiative heat transfer decreases to a minimum as fibre diameter approaches 2 µm but below this value the fibres are smaller than the wavelength of thermal radiation, limiting the efficacy of radiative scattering and thus increasing radiative heat transfer (Farnworth & Osczevski 1985). The diameter of duck down barbules is very close to the theoretical optimum fibre diameter to block maximal radiative heat transfer (Wan et al. 2009) and goose down's are similarly sized, making for a potent block against radiative heat transfer. The greater the fibre: volume fraction the greater the number of radiation-blocking surfaces (Farnworth 1989), so the thousands of barbs and barbules in down feathers are beneficial in reducing radiative heat transfer.

1.3.5.4.4 Evaporation

Evaporation of perspiration is the body's primary defence mechanism against overheating but evaporation can also be a hindrance in cold weather, as wet clothing dries due to the heat produced by the body. The evaporation of water requires significant energy (the latent heat of water evaporation, $\Delta H_{vap\ (water)}$, is approximately 2500 kJ kg^{-1} at skin temperature (Katzir 1993)) and this may cause unwanted cooling of the skin if the body is producing insufficient heat to maintain core temperature.

Evaporative heat transfer is perhaps the most complex heat transfer mechanism that occurs in a down assembly. Wet down, as has been discussed previously, possesses significantly-reduced thermal resistance versus dry down because of its reduced thickness and increased thermal conductivity, so keeping down dry is important to maintain its insulating performance. However, this proves difficult in many situations, even if precipitation is not occurring, because the body continuously produces water vapour by sensible perspiration and as this vapour evaporates the body cools. If this water vapour condenses in the down assembly then this may eventually reduce the thermal resistance of the down as it becomes wet and loses loft. The likelihood of condensation occurring is influenced by factors such as air pressure (at high altitudes, evaporation will be reduced (Fukazawa et al. 2003)), temperature, and water vapour pressure. So, though heat transfer through a down assembly should normally be minimised, heat transfer by evaporation may be beneficial in maintaining the thermal performance of the down if it were to get wet. It is for this reason that keeping the down dry in the first instance is so important and why down, which retains water when wetted, is often regarded as an inferior insulator in wet conditions compared to synthetic insulation (Farnworth & P. A. Dolhan 1983).

1.3.5.5 Principles affecting the warmth of a down product

Two major factors influence the perceived warmth of a down-filled product: the materials used, and the design. The former will be discussed here; the latter is outside of the scope of this work.

With regards to down, the primary factors that influences the warmth of a garment are the amount of down it contains and the quality of that down. A simple equation sometimes used by 'make your own gear' (MYOG) hobbyists to estimate the relative thermal resistance of a filling is shown in Equation 1-7:

$$fill\ volume = mass\ of\ down \times fill\ power\ of\ down \quad (1\text{-}7)$$

Equation 1-7 is used to estimate the total volume of down that a product contains and can be related to thermal resistance (in the UK this is often measured in togs, 0.1 $m^2\ K\ W^{-1}$) or a more subjective measurement, such as a comfort rating, as might be used in a sleeping bag test (British Standards Institute 2002; McCullough 2009).

A more subtle factor influencing the thermal resistance of a down product is the influence of the size of the product's baffles and the subsequent density of the down inside them. For example, 200 g of down compressed into a volume of 100 cm^3 will differ in thermal resistance to the same mass of down in a volume of 1000 cm^3. Very few outdoor equipment manufacturers have carried out detailed testing on the influences of down density on thermal resistance but previous work (Fuller 2012) has indicated that it may be influential, and its effect will be studied in detail in subsequent chapters.

1.4 Down and feathers in composites and nonwovens

Down feathers are almost invariably used on their own as insulating materials, but combining them with other materials may offer ways to reduce their propensity to dispersion, making them easier to work with. Using feathers to make composite materials is relatively common, using chicken feathers in particular: chicken feathers are a by-product of the meat industry and millions or possibly thousands of millions of tons are produced each year (Molins et al. 2013; Wrześniewska-tosik et al. 2011). They are currently disposed of by largely environmentally-unsound methods (Molins et al. 2013; Wool 2005) and as a result, research has attempted to use chicken feathers in numerous areas (George et al. 2006), including mixing chicken feathers with polycaprolactone to make hard plastic composites (Molins et al. 2013); with soybean-oil-derived polymers to make materials suitable for use in electronics (Wool 2005); with cellulose to make paper-like materials (Wrześniewska-tosik et al. 2011); with vinyl ester and polyester to make fibre-reinforced engineering composites (Uzun et al. 2011); and with polyester to make printed-circuit board materials (Kiew et al. 2013). Duck feathers have been used in analogous ways, such as in the manufacture of nonwovens designed to absorb dye effluent (Jin et al. 2013).

Use of down feathers in composites has been much less widely reported. This is likely due to the far greater cost of down versus chicken feathers, their widespread use in clothing and bedding, and crucially, unlike chicken feathers, there is no global surplus of down. One use of down in composites was reported by Liu et al. (Liu et al. 2013) who used duck down to make fibre-reinforced polypropylene composites. However, in the project they significantly modified the down before it was mixed with the polypropylene by shredding it into 'down feather whisker', which they described as needles of diameter 1-15 μm and with an aspect ratio of 20-50 (Liu et al. 2013). The resulting composites were stronger than non-reinforced polypropylene but the use of down in the form of whiskers negated its unique and perhaps most impressive attributes – its shape and geometry.

Making down-based composite nonwovens for insulation has been previously carried out by two groups. A patent filed in the 1990s that has since lapsed concerns using down feathers combined with meltblown polymers to make bulky battings of low density (Li et al. 1998). In the method, polymers are meltblown while down is simultaneously mixed with them to produce composite wadding, though no details are given regarding the performance of the final product. More recently, goose down feathers have been mixed with 20 and 40 weight percentages of Cellbond 254 bicomponent fibre and then heated in an oven to produce nonwoven fabrics (Ye & Broughton 1999). These were found to be lower density and thus more insulating than equivalent nonwovens made from chicken feathers or polyester fibres. They were also found to recover from compression to a greater degree than down feathers alone; this was thought to be due to the reduced slippage of feathers. Despite the promising results of this study, no further information could be found regarding the manufacture of nonwovens using down feathers to make composite insulating materials.

Another product that combines down and synthetic fibres is Primaloft Down Blend (Primaloft 2014) which has recently won outdoor industry awards (McHale 2014). It combines goose down with Primaloft fibres (Primaloft is a polyester microfibre (Donovan 1986)). Down Blend claims to offer superior water repellence and warmth-when-wet when compared to down feathers. The manufacturing process is more akin to down filling techniques than nonwoven production methods: nonwoven batts are constructed then shredded and blown alongside down feathers (Groh & Laskorski 2001) into baffled jackets and sleeping bags. As such, this product does not negate the complex patterns and designs required of down clothing and equipment, instead it enhances down's water repellence. There remains significant scope for a down-based nonwoven material intended to be used as an insulating material in the outdoor industry.

1.4.1 Techniques used to form nonwovens

The major rival to down insulation in the outdoor industry is synthetic nonwoven insulation. There are numerous definitions, sometimes contradictory and often complex, to describe nonwovens. For the purposes of this thesis, nonwovens are described as sheet or web structures of textile origin bonded together by any means (British Standards Institute 2011). Nonwovens have uses in areas as diverse as disposable cleaning wipes, automotive interlinings, and geotextiles. In this thesis, a nonwoven will be made from down feathers to attempt to make the manufacture of down-filled clothing simpler, and so an appreciation of the techniques used in the manufacture of nonwovens is beneficial.

Nonwovens are generally divided into three categories: wetlaid, polymer-laid, and drylaid. Wetlaying manufacturing processes are somewhat similar to those used in the making of paper; polymer-laid processes vary widely but usually incorporate polymer extrusion processes; and drylaid nonwovens tend to utilise methods derived from the traditional textile industries of knitting and weaving (Wilson 2007). Of these processes, drylaying methods are most suitable for manufacturing insulating materials from feathers, as wetlaying tends to produce webs with insufficient thickness, and feather cannot be used in polymer extrusion processes without significant chemical modification.

1.4.1.1 Airlaying

The two main methods used for dry-laying are carding and air-laying. Carding is generally regarded as an unsuitable preparation method for feathers as they are too short, stiff, and complex in shape to combine in the card (Fan 2008; George et al. 2003). Air-laying does not possess the same limitation, as it works for almost any material of correct size (Nelson 1993). Air-laid webs are theoretically isotropic and tend to be high-lofting (Brydon 2007), and therefore ideal as insulation materials. Air-laying also uses the vast majority of the raw material (100 % has the potential to form part of the final product), it is relatively environmentally-friendly as it uses neither harsh chemicals or conditions and generates little waste, and it is a cost-effective and simple method (Pourmahammadi 1998).

The first patent for an airlaying machine was filed as early as 1892 (Kellner 1892) and various methods since then have been filed. The Kroyer system, patented in 1971 (Rasmussen & Tousvej 1971), is in common use (Wilson 2010) and is the system most used at The University of Leeds so will be discussed in further detail. A diagram representing the Kroyer airlaying system is shown in Figure 1-23:

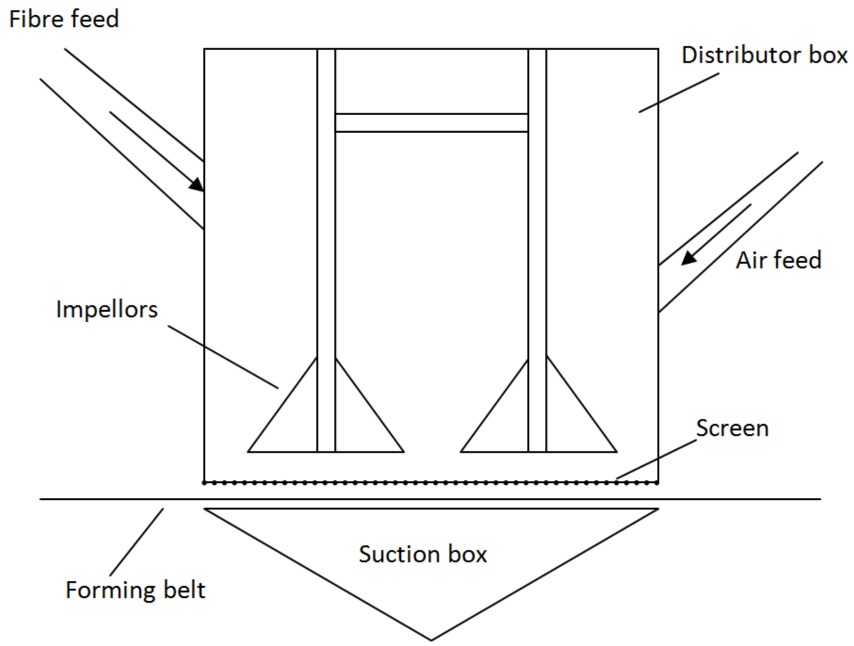


Figure 1-23 - diagrammatic representation of a Kroyer airlaying machine
Redrawn from Rasmussen and Tousvej (1971)

The distributor box houses large impellors that create strong air currents. Fibres are fed into the distributor box where they circulate before being drawn by the vacuum of the suction box towards the screen. They pass through the screen and collect on the forming belt. Fibres introduced into the airlaying line may be pre-opened to ensure sufficient mixing and dilution in the air (Pourmahammadi 1998). The individualisation of fibres is also preferable so as to minimise friction between them (Brydon 2007) and prevent the premature agglomeration of fibres which can lead to fibres unable to pass through the screen, or uneven distributions.

1.4.1.2 Web bonding and thermal bonding

Airlaying mixes and loosely tangles fibres, but they must be formally bonded for the finished product to have sufficient dimensional stability. There are numerous bonding methods used in the manufacture of nonwovens, including chemical, thermal, and mechanical bonding (Brydon 2007). Of these, mechanical bonding methods can be disregarded for the purpose of forming insulation materials from feathers, as their stiffness and complex shapes makes them insufficiently compliant. Chemical bonding tends to lead to relatively poor internal bonding and strength (Malkan 1993), making it potentially unsuitable for use with loose down plumes. Thermal bonding, however, will be discussed in further detail.

Thermal bonding fuses thermoplastic fibres at their crossover points (Malkan 1993) or utilises thermoplastics as adhesives. To achieve this, thermoplastics are incorporated directly into the web where they are subsequently melted. Low-melting polymers such as polypropylene are commonly used. Bicomponent fibres that are composed to two different polymers are often used, as half of the fibre can melt and form bonds in the web while the remainder stays solid and thus mechanically stable. Use of thermoplastic powders to bind down feathers is of limited use because of the difficulty in maintaining an even distribution and avoiding gravitational effects.

The melting of the thermoplastic or bicomponent fibres in a nonwoven may be achieved by various means including: through-air (use of an oven), radiative heat, ultrasonic heating, or calendaring (heated rollers). Most applicable to the manufacture of insulating materials is through-air bonding as when compared to calendaring the lack of direct pressure means that the unmelted fibres remain relatively unaffected, thus able to loft fully (Rogers 1993). It also requires simpler machinery than the other methods, and products are made quickly. Through-air bonding generally yields products of medium-to-high thickness, with high tensile strength.

1.5 Conclusions

Humans are poorly adapted to deal with cold conditions and using clothing is essential to counteracting adverse weather and climate. Many people live in cold conditions and many seek out cold places to take part in activities such as skiing, hiking, and mountaineering. The lack of immediate shelter, changing activity levels, and cold conditions make mountaineering, in particular, a very difficult demanding environment, and clothing for this activity must be very warm, lightweight, compressible, and weatherproof. Down clothing and equipment has been used by mountaineers for almost a hundred years and its superior warmth-to-weight ratio and compressibility mean it remains the choice for many mountaineers' sleeping bags and insulated clothing. Though down feathers are used in numerous applications including in bedding and upholstery, their greatest performance is demanded by outdoor clothing and equipment, and so this work will address down's properties with this end-use in mind.

Down is regarded as having an exceptional warmth-to-weight ratio, compressibility, and compression recovery and is considered by some (Todd 1996) to be the greatest natural insulating material. Despite these remarkable properties and the propensity of information on wool and hair – two other keratinous materials – down has been studied relatively little by academics, leaving great potential for further work into its structure and properties.

1.6 Aims and objectives of this study

A review of the previous research into down feathers has been presented and highlights the importance of down's morphology and mechanical properties in its exceptional performance as an insulating material. As such, structure-property relationships form a major part of this thesis.

A detailed investigation will be carried out into some of the fundamental properties of down feathers, such as their true density, the masses of individual feathers, and the diameters of their barbs. The morphology and features of down feathers will be assessed using both optical and scanning electron microscopes, paying particular attention to cross-sectional shape and tertiary structures (nodes and prongs).

The internal structures of down feathers will be studied, as they are crucial to both mechanical and chemical properties and have barely been examined previously. The structure of wool and hair, analogous keratinous materials, are well-known, and a study into down feathers provided an excellent opportunity for comparisons. Transmission electron microscopy will be used alongside differential scanning calorimetry and X-ray diffraction, which will be used to assess crystal structure. The chemical properties of down feathers will be determined, in particular their sulfur concentration and surface chemistry. A thorough investigation into the cross section and internal structure of down will greatly benefit the interpretation of mechanical testing results.

The mechanical properties of an insulating material are crucial in determining performance, and as the mechanical characteristics of each individual down feather impacts on the attributes of the bulk material, testing of individual down feathers and individual barbs will be carried out in this thesis, alongside the testing of down feather assemblies. Particular emphasis will be given to compression resistance and compression recovery properties, as very little previous research has been carried out in these areas. The mechanical properties of down inside fabric squares designed to mimic real products will also be studied. The thermal properties of down-filled squares will also be determined and the possible influence of down's density on thermal performance investigated. The relationship between fill power and compression resistance is also poorly understood, and will be studied in this work.

The limitations of the baffled constructions of down garments and equipment mean that a continuous down-feather-based insulation, able to be sewn directly into products, would be of great potential use as an insulating material, combining the thermal resistance of down with the ease-of-use of nonwovens. Attempts will be made to manufacture this material.

Chapter 2. Materials and techniques

Many of the materials and techniques used in this research project are described here. When the techniques described are complex, some background theory will be given. When novel experimental procedures were developed, these will be described in their relevant chapters.

2.1 Materials and their conditioning

Goose and duck down were supplied by Mountain Equipment, Outdoor & Sports Company Limited, United Kingdom. The goose down was brown in colour and had a fill power, measured by the International Down and Feather Laboratory (IDFL), of 865 in^3 oz^{-1} (IDFB cylinder and IDFB steam conditioning). The duck down was white and had a fill power of 775 in^3 oz^{-1} (IDFB cylinder and IDFB steam conditioning). Eider down was supplied by Peter Kohl Industries, Germany, and any small pieces of plant matter and detritus encountered in the feathers were removed before testing. Unless stated otherwise, the goose, duck, and eider downs used throughout this study were from the same respective batches and were used as-supplied.

Samples were conditioned for at least 72 hours in an air-conditioned environment of 20 ± 1 °C and 65 ± 5 % relative humidity prior to testing, unless otherwise stated. In cases where experiments were to be conducted in a non-environmentally-controlled atmosphere, the temperature and relative humidity were measured using a miniature environmental sensor and kept within acceptable limits.

2.2 Microscopy and imaging techniques

Optical microscopy, FEG-SEM, SEM, TEM, EDX-TEM, and AFM were used for the examination of the morphologies and microstructure of down feathers. Each of the techniques will be described in turn.

2.2.1 Optical microscopy

A calibrated Leica M205 C microscope (Leica Microsystems 2015) was used for optical imaging and to analyse large features of the down feathers. Leica Application Suite v4.1 was used for capturing images. Samples were held between microscope cover slips to reduce errors encountered when imaging subjects with significant thickness. Z-Plane Image-Builder was used to capture images with a large depth-of-field.

2.2.2 Scanning electron microscopy (SEM)

Different scanning electron microscopes were used depending on what was desired from the images.

2.2.2.1 FEG-SEM

The microstructure of down feathers were examined using an LEO 1530 Gemini field emission gun SEM (FEG-SEM) (Institute for Materials Research 2014). The advantage of this machine was that a whole down feather could be mounted in the SEM in a way that did not affect overall appearance, so both the whole down feather and its barbs and barbules could be analysed.

2.2.2.2 High resolution SEM

The microstructure of down feather barbs and barbules including their cross sections were examined under high magnification and high resolution in a JEOL JSM-6610LV scanning electron microscope (SEM). Individual feather samples were attached to SEM stubs using carbon conducting tape and gold coated using a Quorum Q150RS sputter coater. Attempts were made to minimise voltage (≤5 kV) and subsequent charging effects in the SEM examination.

In order to analyse numerous fibre-cross sections simultaneously using this machine, down feathers were packed tightly into Beem capsules and then transferred to the slot of a Hardy microtome, as described by Greaves and Saville (Greaves & Saville 1995). The tongued part of the device was then used to secure the fibres and nitrocellulose applied to the barbs and barbules. Once the resin had cured, a scalpel was used to cut the fibres and then the fibres moved by use of a threaded plunger. Nitrocellulose was then applied to the fibre ends and once cured, the fibres cut and mounted on SEM stubs.

2.2.3 Transmission electron microscopy (TEM) and energy-dispersive X-ray spectroscopy (EDX) measurements

While different transition electron microscopes were used for TEM depending on the desired result, the sample preparation was the same regardless of the instrument used.

2.2.3.1 Sample preparation

Goose and duck down plumes were prepared for TEM analysis by fixing them in a 2.5 % glutaraldehyde in 0.1 mol dm^{-3} phosphate buffer solution for 2.5 hours (Glauert 1974). They were then washed twice for 30 minutes in 0.1 mol dm^{-3} phosphate buffer solution. In order to increase the contrast between similar chemical structures, selective staining was performed: a 1 % osmium tetroxide in phosphate buffer was used as a secondary fixative and as a contrast agent and the samples were then washed in 0.1 mol dm^{-3} phosphate buffer. The samples were then dehydrated in an ascending ethanol series (20 %, 40 %, 60 %, 80 % and 2 x 100 %, each changed every 30 minutes) (Glauert 1974).

The samples were left in propylene oxide for 20 minutes and then added to new propylene oxide for a further 20 minutes. A combination of 1:1 propylene oxide: Araldite C212 was used to embed the sample for 16 hours then a 1:3 propylene oxide: Araldite C212 mixture was left for 4 hours and an Araldite C212 mixture was then used to embed for 4 hours. The samples were then transferred to embedding moulds with fresh Araldite and left to cure overnight at 60 °C (Luft 1961). Sections of thickness 80-100 nm were cut from the resins using a Reichert-Jung UltracutE Microtome. They were picked up on thin bar, Formvar/carbon-coated copper grids of 3.05 mm diameter and 200 mesh, and stained with saturated uranyl acetate solution for 2 hours followed by Reynolds' lead citrate solution (Reynolds 1963) for 30 minutes.

2.2.3.2 Conventional TEM

For cross-sectional analysis of the down barbs and barbules, most TEM images were taken using a Tecnai Spirit FEI transmission electron microscope equipped with a Gatam digital camera and DigitalMicrograph software. On average, 18-25 images were taken of each of the goose and duck down samples.

2.2.3.3 Energy-dispersive X-ray spectroscopy (EDX)-TEM

EDX measurements were carried out on goose down samples prepared for TEM analysis using a Philips CM200 FEG-TEM/STEM (scanning transmission electron microscope) fitted with a Gatam 7941F MultiScan camera and Oxford Instruments XMax 80 mm^2 silicon drift detector (SDD) EDX. The silicon drift-detection enabled the machine to compensate for sample or beam drift that occurred during analysis (Crewe & Nellist 2009). A quantitative elemental analysis was carried out using AZtec software from Oxford Instruments.

The mechanical, enzymatic and chemical solvation techniques carried out by previous academics to analyse wool and other keratins are more time-consuming and involved tasks than the use of EDX: EDX is an excellent technique to determine the chemical composition of parts of a complex natural material.

2.2.4 Atomic force microscopy (AFM)

Atomic force microscopy (AFM) is a valuable technique for examining surfaces and interfaces (Crossley et al. 2000). It possesses the greatest resolution of all analytical techniques, at approximately 1 nm, and gross lateral resolution is approximately 100 µm, but it requires very careful sample preparation (Haugstad 2012). A diagram describing the basics of AFM is shown in Figure 2-1 (Haugstad 2012):

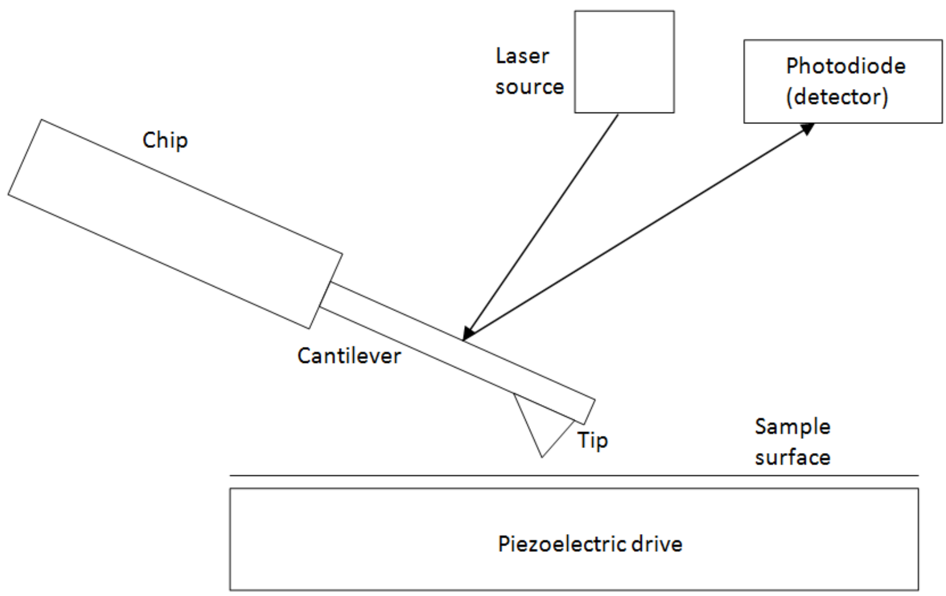


Figure 2-1 – the fundamental features of an atomic force microscope
Redrawn from Haugstad (2012)

In AFM, the sample is mounted on a piezoelectric drive which can move it in the x, y, and z directions (Binnig et al. 1986). AFM measures the forces between the tip and the samples: the forces cause the cantilever to move (Lang & Gerber 2009), and by measuring the deflection using the position of a laser on the cantilever surface it is possible to create an image of the surface with resolution equal to that of the tip's width. AFM relies on the spring constant of the cantilever being smaller than that of the equivalent spring between atoms (Rugar & Hansma 1990), enabling the cantilever to move without displacing the sample's atoms. The path of a tip over a sample is shown in Figure 2-2:

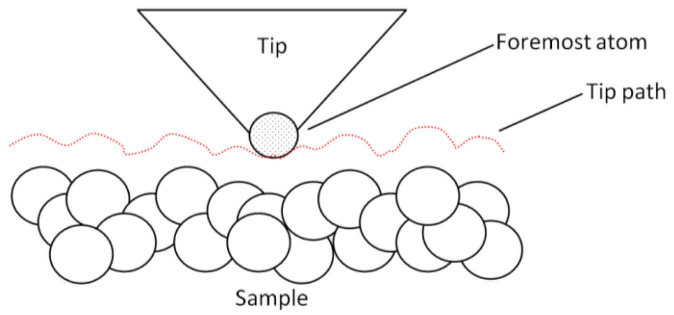


Figure 2-2 – the principle of AFM's tip moving over a sample, whereby the tip's foremost atom follows the path above the sample to maintain constant force between sample and tip

Correct distance must be maintained between the tip and the sample – too far between them and the force is insufficiently strong; too close and the tip risks crashing into the sample surface. AFM also requires meticulous operation: obtaining images is time-consuming and the intuitive interface and operation of modern SEM or TEM systems has not yet reached AFM. For example, recording images at low magnification is not possible using AFM, making identification of the gross features in which you are studying, difficult.

AFM was used for analysis of down's ultra-fine internal structure. Goose down samples that had been embedded in resin for use in TEM (see section 2.2.3.1) were cut from the resin blocks using a Reichert-Jung UltracutE Microtome and slid onto muscovite mica which had been cleaved using adhesive tape to make an atomically-flat surface. The mica was then glued to AFM sample stubs using epoxy resin and mounted on a Bruker Multimode 8 AFM hanging on an anti-vibration table. Samples were analysed and images recorded in colours relating to height.

2.2.5 Digital imaging and post-processing

Three different cameras were used for photography of bulk quantities of down: a Nikon D5000 with AF-S DX VR Zoom-Nikkor 18-55 mm f/3.5-5.6 G lens and a Panasonic DMC-FX30 compact camera were used for most photographs. A Canon EOS 5D Mark II with EF 100 mm f/2.8 Macro USM lens was used for macro photography.

Where appropriate, images taken using the techniques listed were post-processed using Picasa 3.0 freeware to enhance the quality of the image: the contrast of some images was adjusted and some of the images were cropped. Measurements of distance, length, and area, such as when identifying the diameter of barbs, or the area of barb cross sections, were made using Image Pro 7 and Image J software.

2.3 Structural and chemical characterisation

Techniques used to study chemical composition, density, and the mass of the down feathers are described in the following sections.

2.3.1 The mass of down feathers

To determine the mass of down feathers, individual goose, duck, and eider down plumes were weighed using a Sartorius CP225D 5-decimal-place balance (Sartorius n.d.). Over 60 samples from each down type were analysed and efforts made to minimise sizing bias.

2.3.2 The true density of down feathers (pycnometry)

Measurement of the down feathers' true density was made by using pycnometry. In contrast to bulk density, this would determine the density of the down feathers excluding the influence of pores and air existing in the down feather assemblies. The dry and extremely pure helium used in pycnometry means that pores as small as 0.1 nm are filled with helium, guaranteeing that only the mass of the solid material is measured (Rude et al. 2000). Pycnometry is a relatively fast procedure with high repeatability, does not require use of harmful chemicals (Truong et al. 2009), and is free of the disadvantages of other techniques: for example, the

Archimedes method relies on complete wetting of the test material (Rude et al. 2000), and down's inherent hydrophobicity prevents its wetting during the test.

True density (pycnometry) measurements were made on down feathers either conditioned in a laboratory atmosphere (labelled 'un-dried') or having been dried in an oven ('dried'). Dried samples were prepared by transferring 1.5 g of down into 4 permeable nylon mesh sacks and drying them in an LTE Scientific Raven 2 oven at 150 °C. The samples were reweighed after 30 minutes of drying then reweighed after further 10 minute intervals of drying until constant mass (to 10 mg) was achieved. Un-dried samples were tested following laboratory conditioning.

Samples were tested using a Micromimetics Accupyc 1330 Density Analyser (pycnometer) which recorded 20 results per type of down. Samples were weighed into a steel sample tube, compacting the down as much as possible to minimise the moisture present and to maximise sample mass. Un-dried samples weighed 0.90 ± 0.10 g (measured accurately to 0.01 g); dried samples weighed 0.80 ± 0.10 g (measured accurately to 0.01 g). The dried samples were re-dried in the samples tube in the oven for 30 minutes and reweighed to ensure constant mass to 10 mg. As soon as constant mass was achieved, true density measurements took place by measuring the change in pressure of the helium entering the sample tube.

2.3.3 X-ray diffraction

X-rays are of a similar wavelength to interatomic distance, making X-ray diffraction (XRD) a powerful technique for studying internal structure (Margaritondo 1988). X-ray diffraction is used to characterise the internal crystal structure of solids, in which the periodic array of atoms can be considered a recurring distribution of electron density. The diffraction patterns from the interaction of X-rays with these crystals relate to the size and shape of the crystal's unit cells (Low 1955). Keratins, and wool in particular, have played an important part in the development of X-ray diffraction, crystallography techniques, and their use in molecular biology. This work was pioneered by Astbury and his co-workers in the Department of Textile Industries (now School of Design) at The University of Leeds (Andrews 1957).

Analysis of down feathers using X-rays presents considerable challenges. The first is the non-uniformity and three-dimensional shape of the samples preventing the formation of a flat plane for X-ray diffraction. Bragg's Law (Equation 2-1) relies on the gap between planes, d (see Figure 2-3), being consistent to ensure sharp diffraction patterns. In a homogeneous and isotropic material such as some metals, the orientation of the crystals is unimportant but because down plumes are anisotropic along their barbs and their geometry is highly complex, meaning that even if *d* is consistent, the alignment of the crystal planes may not be, which affects the clarity of diffraction patterns (Wilson 1966). Similarly, the non-uniform surfaces of down feathers means that X-rays cannot always meet the surface in the idealised way shown in Figure 2-3 and therefore the value of θ and *d* are not consistent, leading to significant peak broadening.

$$n\lambda = 2d\sin\theta \quad (2\text{-}1)$$

Bragg's Law, where n is an integer, λ is the wavelength of the X-ray radiation in nanometres, d is the lattice spacing nanometres as defined in Figure 2-3, θ is angle of diffraction in degrees

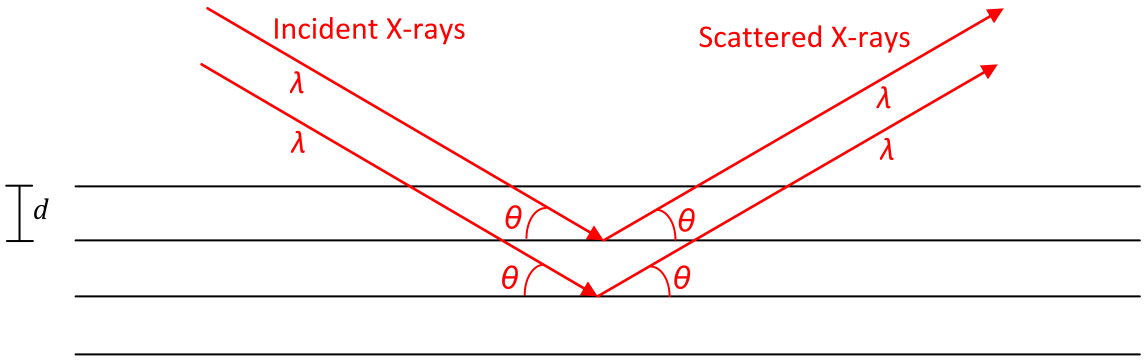


Figure 2-3 – diagram describing X-ray diffraction and the terms used in Bragg's Law

The second difficulty associated with the X-ray analysis of down feathers is the low density of the material (the large amount of air present in and between plumes) reducing the intensity of any diffraction patterns. It means that scans must be conducted for long periods to ensure sufficient data is recorded that will enable separation of meaningful information from the noise. Alternatively, samples must be compressed to make a sample of approximate thickness 2.4-3.0 mm (Kasai & Kakudo 2005) and to maximise sample density. Some linear fibres may be aligned in a sample holder to try to increase signal strength (Kasai & Kakudo 2005; Bertrand et al. 2003; Cao & Billows 1999) but this is not a suitable technique for down feathers, owing to their complex shape and morphology.

X-ray analysis of fibres can take place with two different sample orientations, shown in Figure 2-4 and Figure 2-5:

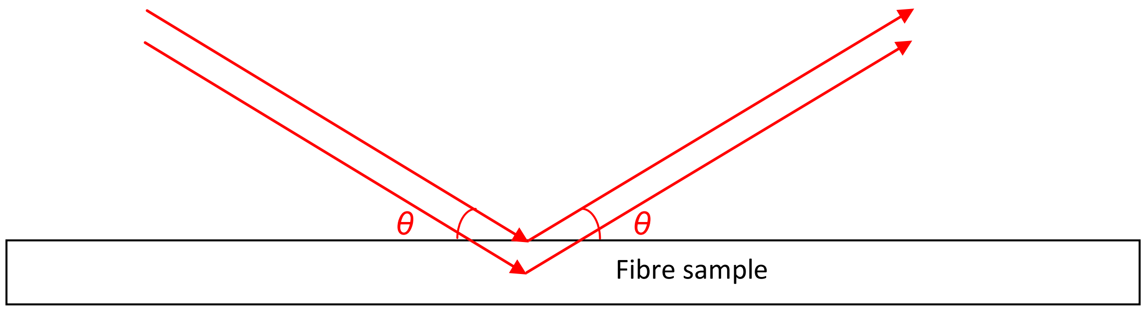


Figure 2-4 - axial (equatorial) orientation for reflection measurements of fibres

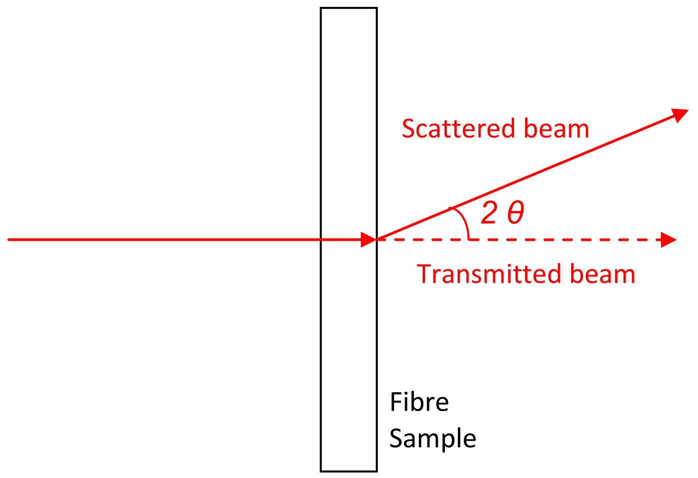


Figure 2-5 - non-equatorial (meridional) orientation for transmission measurements of fibres

For single fibres aligned exactly as shown in Figure 2-4 and Figure 2-5, these two techniques provide data about the different axes of the fibres. This can be seen in the plate diffraction patterns of early work (Bear & Rugo 1951) where the meridional reflections align in the vertical plane and the equatorial reflections lie in the horizontal plane. In down feathers, the practical alignment of multiple fibres is difficult and so in the experiments in Chapter 4, measurements are assumed to be an average of equatorial and meridional measurements.

All X-ray diffraction experiments were carried out using a Philips X'Pert MPD X-ray diffractometer with a tungsten X-ray source (providing a wavelength of 1.54 Å). The software used for data capture was P'Analytical Data Collector version 4.1. A scan angle of 5 to 50 ° was used with a scan size of 0.20 and 224 steps in each experiment. Diffraction patterns were modelled using WinPLOTR from FullProf Suite (FullProf Team 2006) version 2.05. Attempts to model the data were made using 3, 4, 5, and 6 individual peaks.

Four different methods were used for sample preparation, which were developed to improve the quality of X-ray diffraction measurement. Each are described in turn.

2.3.3.1 Method 1 – rolled samples

Three to four down plumes were rolled by hand to form thin dense cylinders of 2-4 mm diameter. Five of these cylinders were then mounted on a single silica crystal to form samples of 15 mm x 15 mm. A 15 x 15 mm beam mask was used. These measurements were made quickly to ensure that goose and duck down produced a diffraction pattern that could be detected.

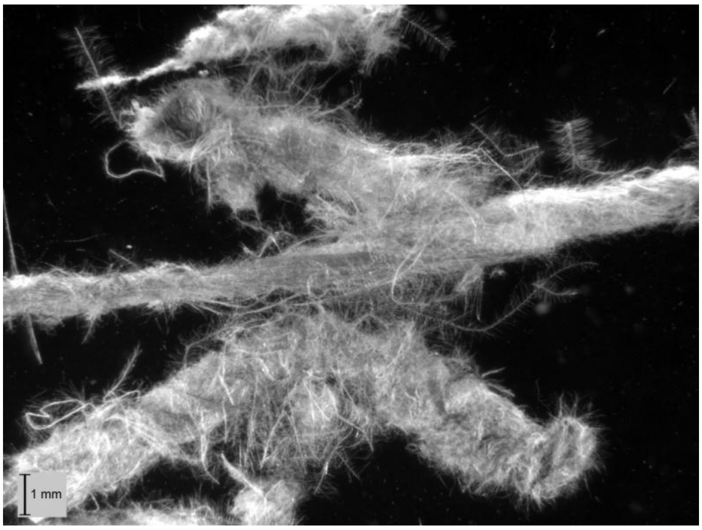


Figure 2-6 – microscope image of rolled down plume samples used in X-ray diffraction experiments

2.3.3.2 Method 2 – powdered samples

Down plumes were cut as finely as possible using a razor blade to make a powder. The powder was then mounted on pressure-sensitive kapton tape (kapton is non-leaching under vacuum) and attached to a single crystal of silica and mounted on the X-ray diffractometer using a spinner mount. A 15 x 15 mm beam mask was used.

2.3.3.3 Method 3 – rolled samples on a bracket

A 3 mm thick brass sample mount (shown in Figure 2-7) was constructed and a 20 x 20 mm beam mask used to increase X-ray flux and subsequent signal strength. A lead mask was mounted on the detector to stop any direct X-ray beam flooding the detector. Down plumes were twisted as described in section 2.3.3.1 and these samples mounted over the brass sample holder’s 25 x 40 mm window using kapton tape. They were placed in a bracket (kinematic mount) and analysed.

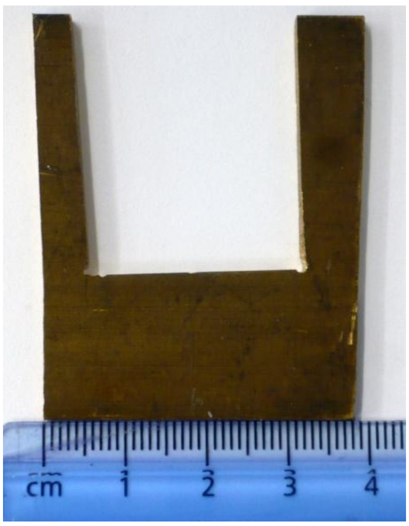


Figure 2-7 - brass sample mount used in some X-ray diffraction experiments

2.3.3.4 Method 4 – nonwoven samples

Nonwoven fabrics were made from both goose and duck down, individually. The down feathers were fed into a Kroyer-type airlaying machine and a mask put onto a Loomstate open-weave linen scrim to produce a web of dimensions 150 x 100 mm and even thickness. The airlaid feathers were transferred to a plastic sheet and a polyester scrim placed on top of each web. The webs were hydroentangled using a 0.5 m wide Hydrolace system (120 µm jet strips, 3 m min^{-1} roller speed, 60 bar pressure, 1 pass) and then dried. SEM images were taken of these nonwoven samples using the procedure described in section 2.2.2.2 to ensure the airlaying and hydroentanglement had not damaged the feathers. The nonwovens were cut to fit the brass sample holder's window and mounted using kapton tape. The samples were placed in a bracket and analysed in three different ways:

a. When dry and in an atmosphere of air.
b. Samples were weighted down and held under water for 15 minutes until completely saturated. They were then analysed while wet.
c. Samples were subjected to vacuum to dry them: the 'Chiller' (Leybold Vakuum Ionivac) was attached over the bracket and a vacuum of $6 \pm 1 \times 10^{-2}$ Pa was achieved by using of an Oerlikon Leybold Combivac IT23 and Turbotronik NT10. They were then analysed while in this evacuated and dried state.

2.3.4 Differential scanning calorimetry (DSC)

Differential scanning calorimetry (DSC) is a technique used to study the physical and chemical transitions that a material undergoes when they change in temperature. Peak fit software was used to determine the peak onset, peak tip, and enthalpy change of a transition.

DSC analyses were carried out using a calibrated Perkin Elmer Jade DSC (Perkin Elmer 2015a) and Intracooler to determine thermal properties in the down. 6-12 mg samples of goose and duck down were sealed in aluminium sample containers, weighed to 0.01 mg, and a pinhole made in the top of each to stop build-ups of pressure (Cao 1999). An empty sample container was used as a reference. Measurements took place from 30 to 350 °C at a heating rate of 10 °C min^{-1} under a flow of nitrogen of 13 cm^3 min^{-1}.

2.3.5 Attenuated total reflectance Fourier-transform infrared spectroscopy (ATR-FTIR)

A Perkin-Elmer Spectrum BX Fourier transform infrared (FTIR) spectrophotometer (Perkin Elmer 2015b) fitted with an attenuated total reflectance (ATR) crystal was used. ATR made for easier sample preparation than use of a nujol mull and KBr discs. ATR-FTIR spectra were measured as an average of 64 scans at a resolution of 1 cm^{-1} over a range of 4000-600 cm^{-1}. A blank background measurement was run before analysis of each sample.

2.4 Characterisation of the mechanical properties of down feathers

Two machines were used for measuring the tensile properties of individual down feather barbs: an Agilent T150 and a Zwick Roell universal testing device. All of the compression and recovery testing on down plumes and barbs was carried out using the Zwick Roell machine.

2.4.1 Testing of tensile properties of individual down barbs using Agilent T150

Cardboard testing templates were cut using a CAD-CAM Technology (UK) 50 W FB Series laser cutter. These templates (Figure 2-8) were modified from an existing design used for the testing of spider silk (Holland 2012) and prepared to specify a specimen length of 5 mm across the middlemost aspect of the fibre (modulus is thought to increase towards the tip of some feathers (Bonser & Purslow 1995)). Individual goose and duck down barbs were taken from plumes and mounted on the cardboard templates using cyanoacrylate glue.

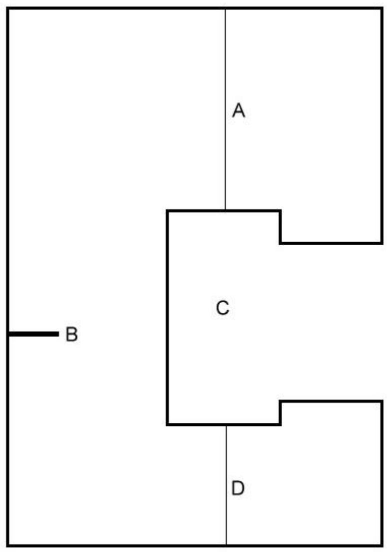


Figure 2-8 – diagrammatic representation of a tensile testing template
A – upper fibre-mounting guide; B – cutting mark; C – sample area; D – lower fibre-mounting guide

The barbs' diameters were measured using the Leica M205 C microscope (at 160 times magnification) described in section 2.2.1, and Image Pro 7 software. Tensile testing of the barbs was carried out in monitored ambient conditions (24 ± 2 °C, 60 ± 5 % RH), using an Agilent UTM T150 tensile testing machine. The UTM T150 was mounted on a T150 Vibration Isolation Table and inside a T150 Acoustic Isolation Cabinet. The card templates were mounted between the instrument's jaws and then the side cut to leave only the specimen held by the instrument. Samples were pulled at a strain rate of 2.7 x 10^{-3} s^{-1} until failure, utilising a load resolution of 50 mN and a displacement resolution of 35 nm. Any tests that resulting in sample slippage were discounted. Breaking of the samples tended not to occur at the grips, indicating that the grips did not concentrate stress at the ends of the specimen length (Butler & Johnson 2004). Aspects of the test procedure are shown in Figure 2-9:

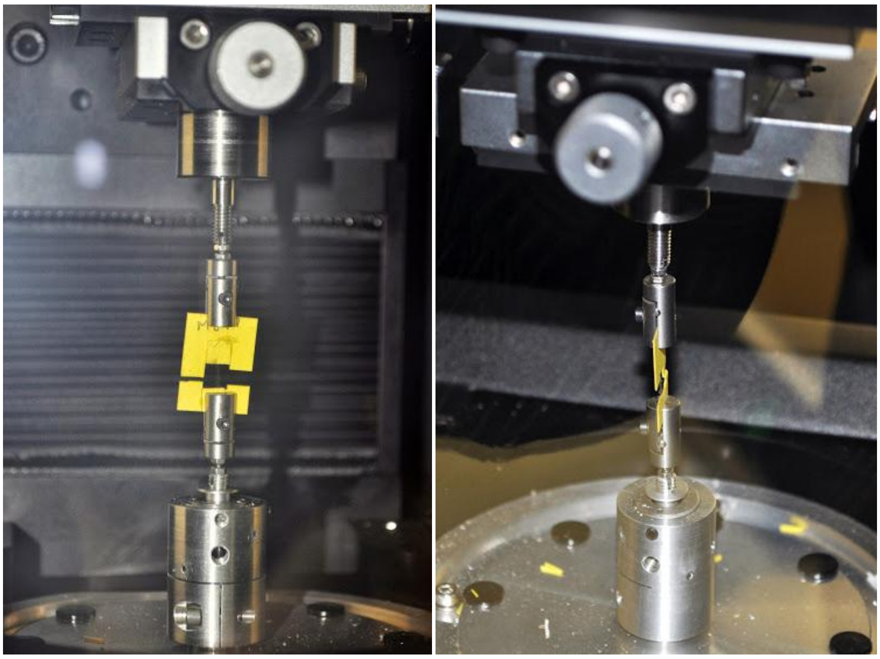


Figure 2-9 – tensile testing of a down feather barb, shown front-on and from the side

2.4.2 Zwick Roell universal testing device

Compression and recovery testing of down plumes, and tensile testing of individual down barbs was carried out using a Zwick Roell Z010 universal testing device. Several different load cells and jaws were used depending on the test method.

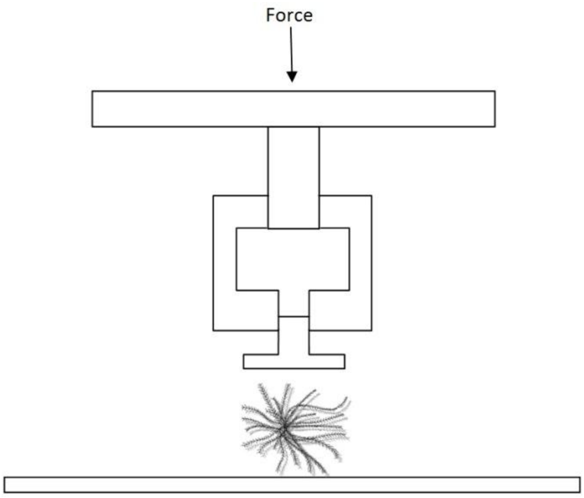
2.4.2.1 Tensile testing of individual down barbs

A 10 N load cell (AST KAP-S) was used with 20 N jaws. As a machine not designed specifically for the testing of very small samples, the 10 N local cell was tested to ensure it offered sufficient resolution: <1 g masses (exerting <10 mN force) were weighed using a Mettler-Toledo New Classic balance accurate to 0.0001 g before being suspended from the 10 N load cell. The agreement between results from both the digital balance and the Zwick Roell Z010 universal testing device was extremely good (within 1 % for each sample) so the 10 N load cell was deemed sufficiently accurate for the intended testing.

Templates were cut and barbs mounted as described in section 2.4.1. Samples were pulled at a rate of 0.27 mm s^{-1} from a preload of 0.3 mN until failure. Testing was carried out in a monitored ambient environment (23 ± 2 °C, 40 ± 5 % RH). The data from 29 eider, 30 goose and 28 duck down samples was analysed.

2.4.2.2 Compression and recovery of individual down feather plumes

The Zwick Roell machine's 10 N load cell was equipped with 200 N jaws that clamped a 25 x 25 mm uniform and flat-bottomed aluminium plate as shown in Figure 2-10. The down feathers were positioned one-at-a-time under the aluminium plate and compressed at a speed of 20 mm min^{-1} to a minimum sample height of 0.1 mm. A preload of 1 mN was reached before data was recorded. Testing was carried out in an uncontrolled but monitored atmosphere of 22 °C ± 1 °C and 50 ± 5 % relative humidity.



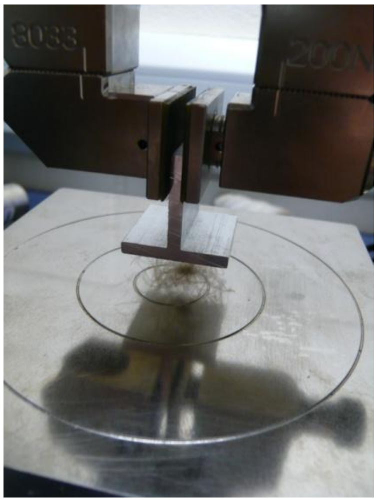


Figure 2-10 – schematic representation and photograph of the compression of a down plume under the aluminium plate

2.4.2.3 Uniaxial compression of individual down barbs

The 10 N load cell and 20 N jaws were used. Individual barbs were mounted on cardboard templates with an effective gauge length of 15 mm, prepared as described in section 2.4.1. Samples were mounted in the jaws and the long-side cut; 1 mm of travel then took place to reduce the gauge length to 14 mm to minimise the effect of any tension that had resulted from the attachment of the down barbs to the testing templates. Testing was carried out to a gauge length of 3 mm length, a displacement of 11 mm. Displacement in excess of this was impractical as the sample mount had the potential to make contact with the opposite jaws.

2.5 Manufacture of down-based nonwovens

Bicomponent fibres (Fibre Vision PP bico, AL Adhesion, 1.7 dtex, 6 mm staple length) were fed through an M&J 'Kroyer' airlaying line to open the fibres. They were collected on a Loomstate open-weave linen scrim. The bicomponent fibres were then mixed with duck down feathers in different ratios. These blends were incrementally released into the distribution chamber over the course of 10-15 minutes to ensure that low fibres concentrations were maintained in the chamber. Custom-sized 31 x 31 cm webs were made by masking the collection belt with a template. A scrim was placed over this and the down and bicomponent blend collected on top to make samples known as ‘blend samples’ from this point.

In order to investigate different construction methods and to aid the bonding of down feathers, sandwich webs were also made. In this method, a portion of bicomponent fibre was put into the distribution chamber, then a blend of down and bicomponent fibre, then a portion of bicomponent fibre.

The airlaid webs were bonded by being covered on all sides by the scrim and then fed into a Spooner Through Flow Dryer at 2 m min^{-1}. They were heated for 5 minutes at 150 °C and with a fan output of 50 %.

2.5.1 Determination of thickness

The thicknesses of the down-based nonwovens were measured using a Shirley Thickness Measurer and a method based on BS: EN 5084 (British Standards Institute 1997). The samples were placed under the foot of the tester and a 20 g mass applied (exerting a pressure of 39 Pa) and the foot slowly lowered until it touched the sample. After 30 seconds of dwell time the measurement was recorded to the nearest 0.1 mm. 4 measurements were made in different areas of each sample.

2.6 Measurement of the thermal resistance properties of down-filled test squares and down nonwovens

The thermal resistance of down-feather-filled test squares and down nonwovens were tested according to ISO 11092 (British Standards Institute 1993) using a Measurement Technology Northwest sweating guarded hotplate (SGHP) 8.2 with a heating area of 31 x 31 cm. Using a guarded hotplate ensured faster testing and highly reproducible results when compared to tog testing. The test plate, guard ring and lower guard were all set to 35.00 ± 0.01 °C. The testing was carried out inside a Thermal Products Solutions Lunaire CEO910-4 environment chamber which controlled temperature and relative humidity. The relative humidity was maintained at 65 ± 1 % and the airspeed at 1 ± 0.05 m s^{-1}, as measured by an air velocity sensor positioned 15 mm above the sample. The environmental temperature, as measured by the sweating guarded hotplate, was maintained at 20.0 ± 0.1 °C.

Samples were mounted on the test plate and guard ring before being held in place by four additional guards. The samples were kept as flat as possible to ensure good contact to the test plate. The plenum table on which the sample rested was adjusted to maintain the specified air gap between the sample top and air velocity sensor.

During testing, the heat flux from each of the three heated areas (test plate, guard ring, lower guard) was recorded and the thermal resistance to conductive transfer (R_{CT}) calculated. The thermal resistance to conductive transfer (R_{CT}) of the bare test plate had a thermal resistance of 0.07 °C m^2 W^{-1} and this was deducted from any measurements. Testing was carried out until equilibrium had been maintained for at least 30 minutes and each test lasted 40-90 minutes. The mean value of R_{CT} and the percentage of cumulative variance (CV) were obtained from the last 20 minutes of data collection. Three repeats were carried out on each down-filled test square. For each repeat, the sample was removed completely from the sweating guarded hotplate before being retested to negate any differences in measurement due to sample positioning or the distribution of down inside the test squares.

Chapter 3. The morphology of duck, goose, and eider down feathers

The unique shape and morphology of down feathers have a large impact on their performance as insulating materials: the most obvious difference between down (an excellent insulator) and flight feathers (poor insulators) is their geometry, because while flight feathers are fundamentally flat, down plumes are approximately spherical (Loconti 1955).

In this chapter, the structures observed in the three major types of down feathers used in commercial applications (goose, duck, and eider), are examined and the diameters of the down feathers' barbs compared. The influence of the down feathers' morphology on their mechanical properties is discussed, and how this might influence their use in outdoor equipment. Eider down has barely been studied save for imaging by Loconti in the 1950s (Loconti 1955) and a brief mention in a paper from 2002 (Dove & Peurach 2002). Here, it will be compared to both goose and duck down.

3.1 Qualities of the received feathers

A large mass of goose or duck down could be poured and flowed slowly, somewhat like a viscous liquid or a liquid aerosol such as fog. The down feather assemblies moved gently in moderate air flows and could move unpredictably in the same way as some non-Newtonian fluids. Single down feathers were much more affected by air flow than a larger mass of down, and they would migrate readily even in very small air flows. The barbs of individual down feathers moved constantly unless air flow was near-zero. Static electricity could affect the feathers, as reported previously (Wilde et al. 2006), and they tended to cling to plastic bags and other static surfaces. As a result of this attraction to static, liability to move, and their small size, handling of the down feathers was challenging.

3.1.1 Duck and goose down

The supplied duck down was uniformly off-white (Figure 3-1) and had a faint smell. Goose down was more varied in its colour, containing grey, off-white and dark brown feathers (Figure 3-2). Individual duck and goose down feathers can be seen in Figure 3-3 and Figure 3-4, respectively. With the exception of colour, the supplied goose and duck down feathers were very difficult to differentiate between and were of approximately similar size. However, in both materials, there were considerable differences in the dimensions of each individual down feather.

Figure 3-1 - duck down

Figure 3-2 - goose down

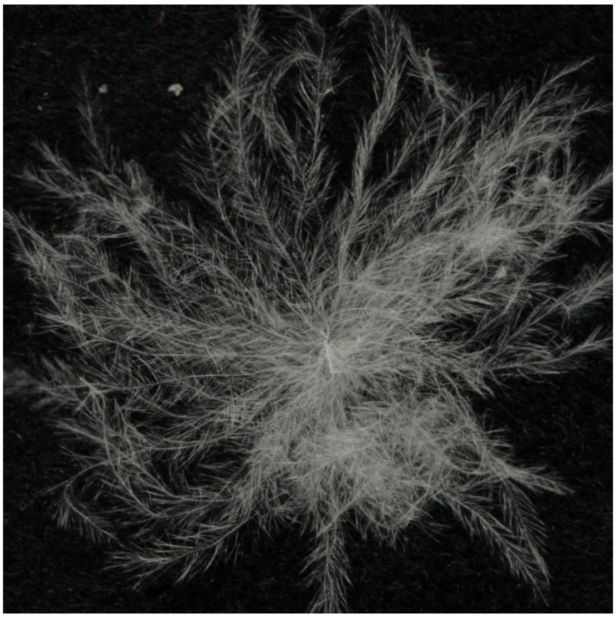


Figure 3-3 – a duck down plume

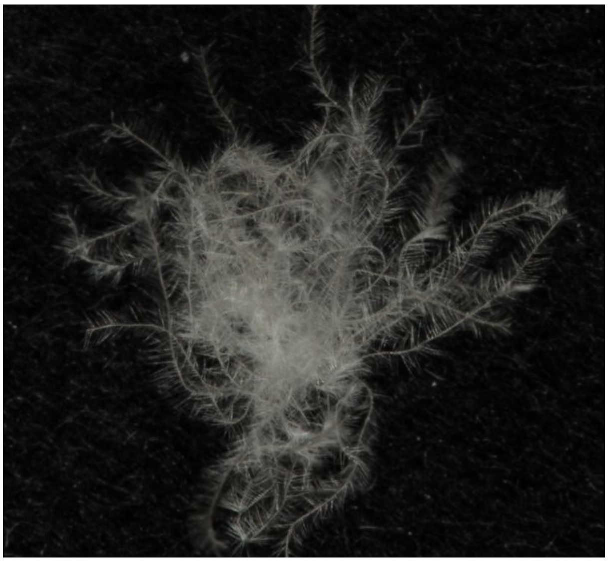


Figure 3-4 – a goose down plume

Few flight feathers were present in either goose or duck down. Flight feathers (Figure 3-5) are flat and contribute little to insulation, so minimising the fraction of these is advantageous in providing an optimal thermal barrier. Intermediate feathers (see Figure 3-6) that share properties of both down and flight feathers were more common than flight feathers, accounting for approximately 5 % of the total number of feathers. Intermediate feathers are expected to contribute more to insulation than flight feathers do, but less than pure down feathers due to their large feather quills that add significant mass to the feather but little to its bulk and subsequent insulation performance (Loconti 1955).

Figure 3-5 – a flight feather

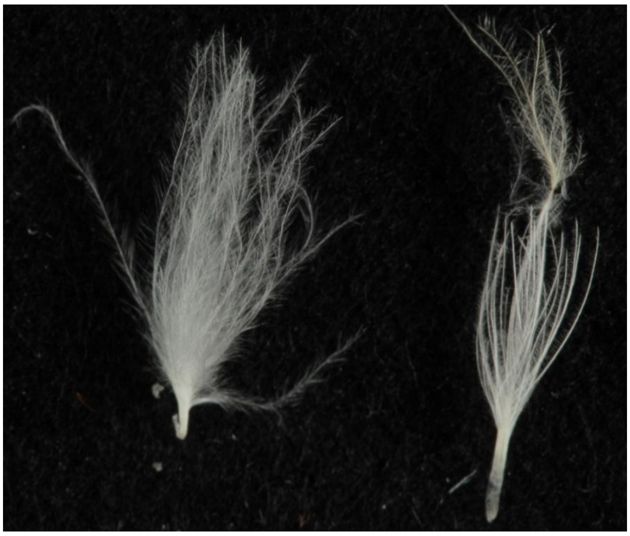


Figure 3-6 – intermediate duck down feathers with both down-feather-like and flight-feather-like qualities

Some stray barbs were present in both the goose and duck down assemblies. These are presumed to have broken from plumes during processing or in transit. These individual barbs contribute very little to insulation, having little ability to trap air.

3.1.2 Eider duck down

Eider duck down (eider down from this point) was quite different to either goose or duck down. The plumes tended to be larger, more resistant to air flow, and their colour was different: a mixture of light brown, grey and dark grey. Figure 3-7 shows a bulk quantity of eider down, and Figure 3-8 shows an individual eider down feather:

Figure 3-7 - a bulk quantity of eider down

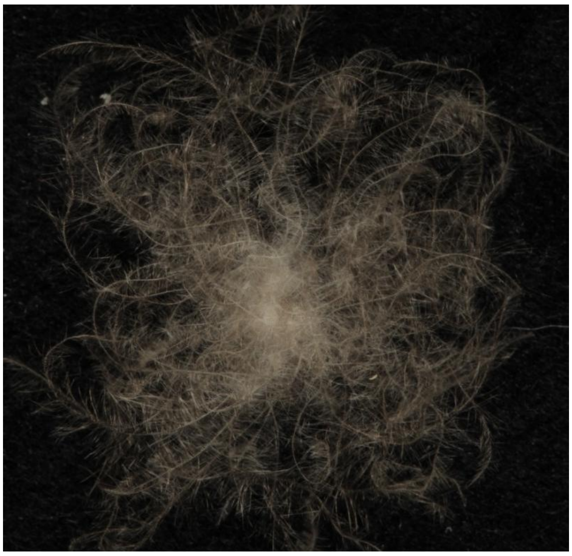


Figure 3-8 – eider down plume

Eider down plumes clung (Bedard et al. 2008) to one another far more readily than goose or duck down plumes did. As a result, they were more resistant to movement in air flows. In nature, this feature may have benefit in eider nests, where plucked feathers must not be blown away by the wind.

The supplied eider down contained approximately 1 % flight feathers. It has been previously claimed that eider down is completely free from flight feathers (Bedard et al. 2008), and while this was not true, their low count would maximise any insulation properties. Some other contaminants were found in the eider down, however, and these were usually plant-derived, such as very small twigs or leaves.

3.2 Characterisation of down plumes

In this section, the plumes of goose, duck, and eider down are characterised in company with optical and SEM images. The masses of the plumes are determined using the method described in section 2.3.1 and the true density of the down feathers measured using the method described in section 2.3.2.

3.2.1 Morphology

Optical microscopy was used to examine a great number of down plumes, negating some of the sampling bias that may be associated with the study of naturally-derived materials. The observations in this chapter were recorded having studied over 50 of each type of down plume using an optical microscope. The procedure used is described in section 2.2.1.

The shape of down feathers is vital to their performance and as the primary purpose of each down feather – to provide insulation – is the same, their shapes were very similar when viewed under a microscope, as shown in Figure 3-9 to Figure 3-11. Barbs left the core in all directions and along the length of the barbs, barbules were observed. The quill, which acts as the anchor for the down feather while it is attached to the bird, varied significantly in size. Sometimes it was barely observed at all, such as in Figure 3-9, though in other specimens it was clearly visible, such as in Figure 3-10.

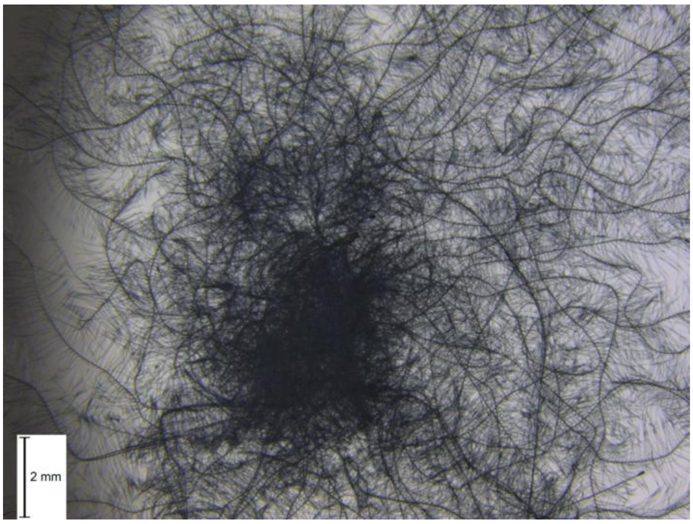


Figure 3-9 – a duck down plume viewed under an optical microscope

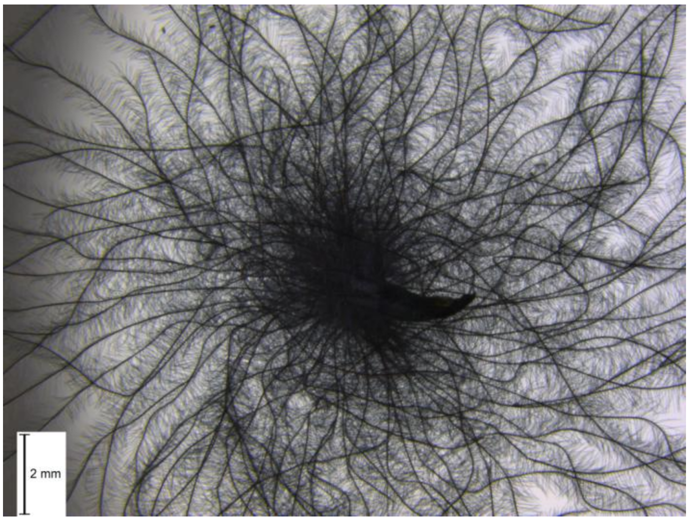


Figure 3-10 – a goose down plume viewed under an optical microscope

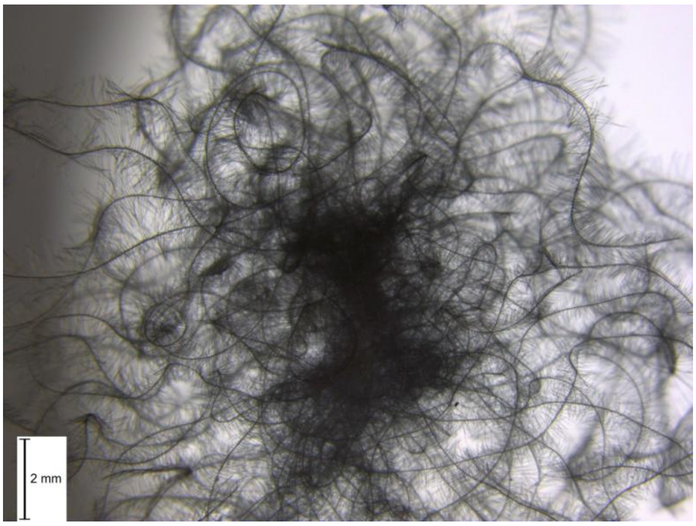


Figure 3-11 – an eider down plume viewed under an optical microscope

In all types of down the plumes' overall shapes were approximately spherical, despite the variety of feather sizes. Some feathers measured less than 10 mm from the distal end (the end furthest from the centre of the feathers, as opposed to the proximal end which is the end nearest the centre of the feather (see Figure 3-12)) of one barb to the distal end of an opposite barb. Others were more than 40 mm over the same measurement. Measuring plumes, however, was very difficult because the 'span' (distance between the distal ends of opposite sides of a single plume) changed readily in turbulent air and could vary during measuring. Also, because down feathers are approximately spherical, it was not easy to measure their diameter at their widest point with instruments such as rulers or callipers. As such, feather masses were to be measured (see section 3.2.3).

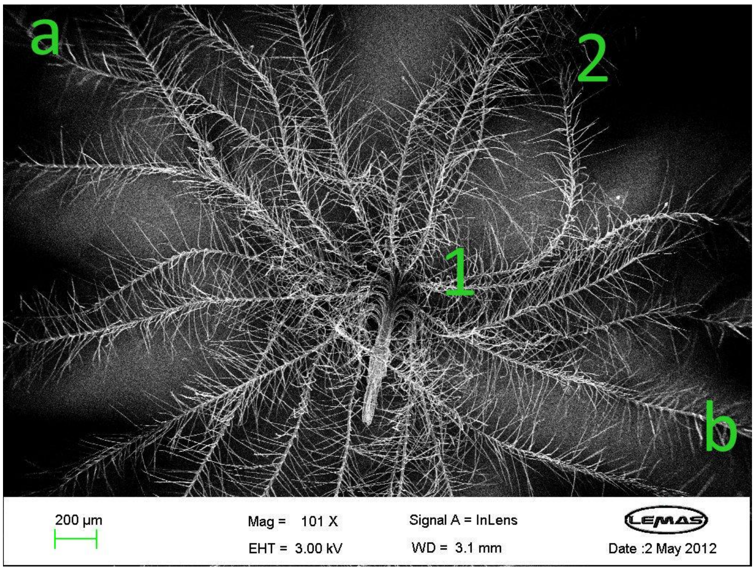


Figure 3-12 - SEM micrograph of a goose down plume
1 - Proximal end of barb; 2 - distal end of barb; distance a-b is the span of the feather

The approximately-spherical symmetry of the down feathers was very different to that of flat flight feathers. This can be described by group theory: a point group defines how many symmetry elements (the ability of a shape to undergo reflection or rotation through an axis or plane and appear to be the same following this operation (Wiberg & Wiberg 2001)) a shape has. For example, an equilateral triangle might be turned by 120 °, 240 °, or 360 ° (see Figure 3-13) and remain unchanged in appearance. These transformations are described by symmetry elements:

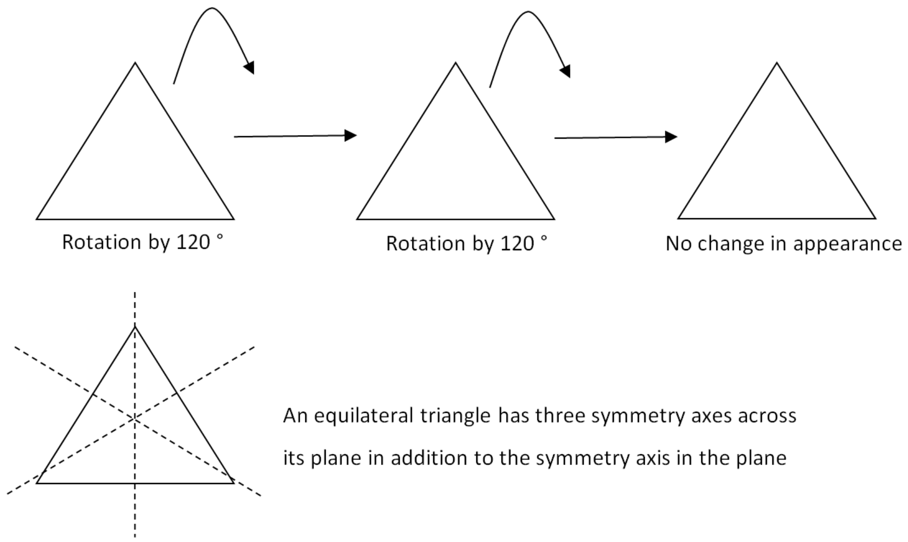


Figure 3-13 – diagrammatic representations of some of the symmetry operations that an equilateral triangle might undergo with no discernible change in appearance, thus defining the location of its symmetry elements (e.g. a plane of symmetry)

Flight feather are members of the point group C_{2V} because they have two planes of symmetry (one in the flat plane and one through the plane) and two inversion centres, in addition to the 'identity' operation that all shapes possess. A down feather, however, can undergo any number of symmetry operations and remain essentially unchanged as it possesses an approximately spherical shape. Thus, it is part of the point group K_h.

The exceptional symmetry of down feathers aids packing and maximises their insulating efficiency: unlike flat feathers, down insulates equally along all axes, ensuring it can block heat transfer in all directions. This exceptional symmetry was also reported by Gao et al. (J. Gao, Pan & W. D. Yu 2007).

3.2.2 True density of goose and duck down plumes

To the author's knowledge, the true density of down has not been determined, while the density of keratins in wool, hair, hoof, and other feathers have been reported previously (Filshie & Rogers 1957; Filshie & Rogers 1961). Because true density is crucial to the 'warmth to weight ratio' (thermal resistance per unit mass) of an insulating material, it was deemed necessary to determine whether there was a difference in the densities of duck and goose down. The moisture regain of the down samples was also examined.

The method described in section 2.3.1 was used and both dried and un-dried down feathers analysed. The mean and standard deviation values for the down feather densities are shown in Table 3-1 and Table 3-2:

Table 3-1 – true density of un-dried down feathers

Un-dried Down	Mean density (g cm^{-3})	Density SD (g cm^{-3})
Duck	1.444	0.003
Goose	1.416	0.002

Table 3-2 – true density of down dried in an oven until constant mass was achieved

Dried Down	Mean density (g cm^{-3})	Density SD (g cm^{-3})
Duck	1.266	0.002
Goose	1.251	0.007

There was no significant difference (p-value (*p* from this point) > 0.05) between the true densities of goose and duck down, either before or after drying. The density decreased following drying, indicating that a significant volume of water was present in the micropores of the down feathers. The results from the dried down feathers correlated with literature values for the dry density of feather keratin, measured as 1.27 g cm^{-3}, and other values collected from keratins of 1.27-1.32 g cm^{-3} (Filshie & Rogers 1957; Filshie & Rogers 1961; R Schor & Krimm 1961). The low standard deviation values confirmed the high repeatability of the pycnometry technique.

As no significant difference between the densities of duck and goose down could be found, this testing proved that any differences in their properties was not simply due to dissimilarities in density.

3.2.3 Masses of goose, duck and eider down plumes

While accurately determining the size of down plume was not practical, weighing plumes is possible with a sufficiently-accurate balance. With consideration that there was no measurable difference in the true density of goose and duck down, the difference in the mass of the plumes could represent differences in plume size. The method used is described in section 2.3.1.

A summary of results from the mass analyses are shown in Table 3-3:

Table 3-3 – masses of down feather plumes

Down	Mean mass (mg)	Mass SD (mg)	Sum of masses (mg)
Eider	2.02	1.17	127.32
Goose	0.77	0.62	48.54
Duck	0.70	0.60	44.09

As shown in Table 3-3, the mean mass values of goose and duck down were very similar, and both approximately 1/3 that of eider down. ANOVA analyses confirmed that the masses of goose and duck down feathers were not significantly different to one another (p = 0.51) but the mass of eider duck down feathers was significantly different to both duck and goose down ($p = 1.11 \times 10^{-11}$ when comparing goose and eider; $p = 8.70 \times 10^{-13}$ when comparing duck and eider).

Box and whisker plots of the data are shown in Figure 3-14:

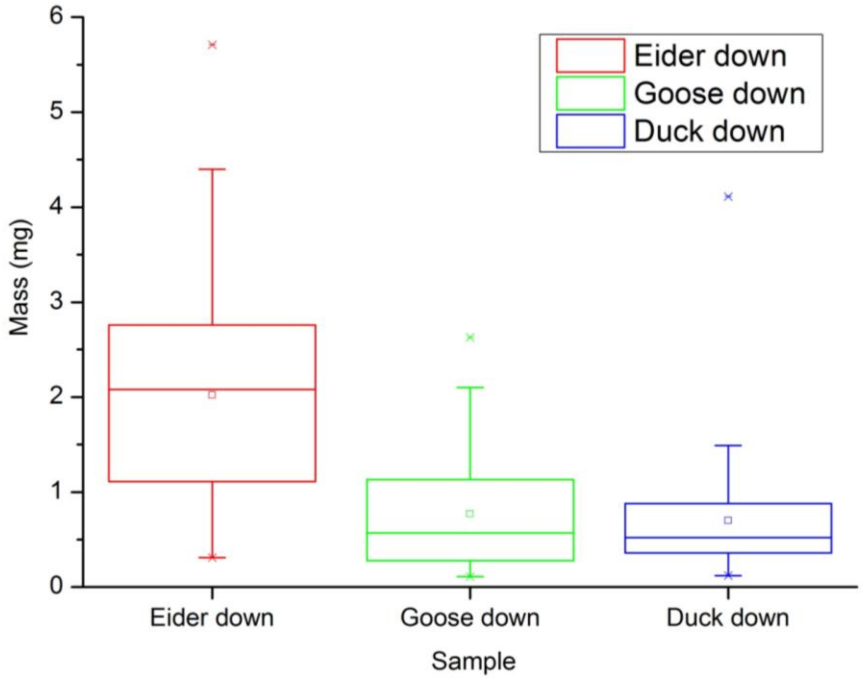


Figure 3-14 - box and whisker plots of eider, goose and duck down masses
x refers to the highest and lowest data values, the top whisker represents the 90th percentile, the top of the box represents the 75th percentile, the line inside the box the median value, the □ the mean value, the bottom of the box the 25th percentile, and the lower whisker represents the 10th percentile

The data is also displayed in histograms in Figure 3-15. These histograms' bin-widths were calculated according to the Freedman and Diaconis (Freedman & Diaconis 1981) model (shown in Equation 3-1), which is more suitable than arbitrary bin-width sizing for complex or wide-ranging distributions (Freedman & Diaconis 1981). Correct bin-widths are crucial in presenting data that has sufficient but not overwhelming detail (Wand 1997).

$$bin\ width = \frac{2\ (interquartile\ range)}{\sqrt[3]{number\ of\ measurements}} \quad (3\text{-}1)$$

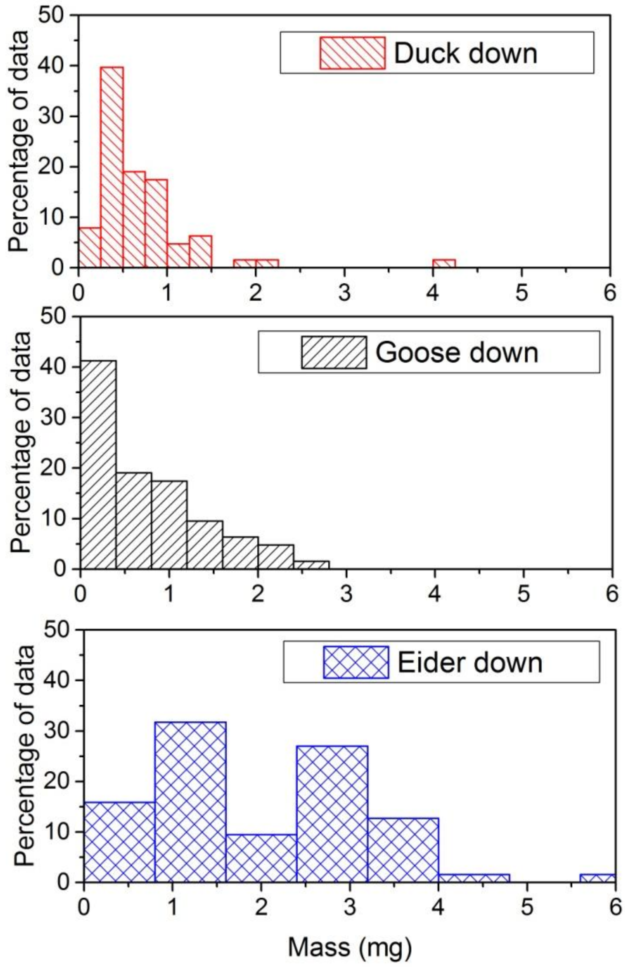


Figure 3-15 - histograms showing the masses of individual goose, duck, and eider down feathers

The box and whisker plots also show the close similarities between the goose and duck down mass distributions but a difference to the masses of the eider down. The mass distribution of goose and duck down feathers were approximately normal (one outlying measurement in the duck down) but the distribution of eider feathers may be bimodal (greatest frequencies at 0.8-1.6 and 2.4-3.2 mg, but low frequency of feathers at 1.6-2.4 mg). This would require the measurement of more feathers to confirm this.

The greater mass of eider down plumes versus goose or duck down is likely due to two factors. Firstly, the feathers are plucked by the eider birds themselves and the bird may be able to identify and pluck only mature feathers which are likely to be more massive than the feathers picked from geese and ducks at slaughter, where feathers are removed regardless of their stage of development. Secondly, the feathers are a different morphology (discussed more in section 3.3): the barbs and barbules on eider down are more densely-packed than those of either goose or duck down and this influences the overall mass of each feather. A bimodal distribution in the eider down feathers may suggest that the down feathers are grown either to occupy maximum space (large feathers) or are genetically limited in their maximum size (small feathers) so as to fill the gaps in between the larger feathers. Further experimentation, preferably with direct access to source birds, would be required to confirm this, but is outside the scope of this work. Certainly, bimodal distributions of feathers have been observed in other species (Gosler & King 2011; Broughton et al. 2008; Mckeegan & Savory 1999; Janiszewski 2006), though this is often a result of sexual dimorphism (the difference in shape and size between the male and female birds of the same species (Owens & Hartley 1998)) which is not applicable here as only female eider birds occupy nests, and so most down feathers are from females.

Feather size may impact on fill power, the outdoor industry's standard method used to determine down quality. Larger feathers occupy more space than smaller ones and the space between each feather is also greater meaning each feather cannot pack together very effectively. How this impacts on thermal performance is currently uncertain: larger, heavier feathers have more surface area and so a greater resistance to radiative heat transfer, but if the gaps between large plumes can be easily disturbed by agitation or small compressive forces, then the potentially-greater bulk resulting from larger feathers would be negated.

This work confirms that eider down feathers are heavier than goose or duck down and, as the densities of keratins are very consistent, the greater mass is due to more and/or longer barbs and barbules of eider down plumes. The mass and subsequent size of goose and duck down feathers are extremely similar.

3.3 Characterisation of down barbs, barbules, and tertiary structures

The morphology of barbs and barbules in the plumes of goose, duck and eider down feathers was studied in this section by using optical microscopy and SEM as described in section 2.2.1 and 2.2.2. The diameter of barbs was measured using the method described in section 2.4.

3.3.1 Down barbs

Barbs in goose, eider, and duck down will be discussed concurrently.

The core in each plume had similar geometry and the barbs divided in all directions from it (Figure 3-16) in a manner reminiscent of islands-in-the-sea bicomponent fibres that can divide under mechanical stresses (Ndaro et al. 2007). The multi-directional emanation of barbs from the central core imparted excellent space-filling ability that maximises insulating properties.

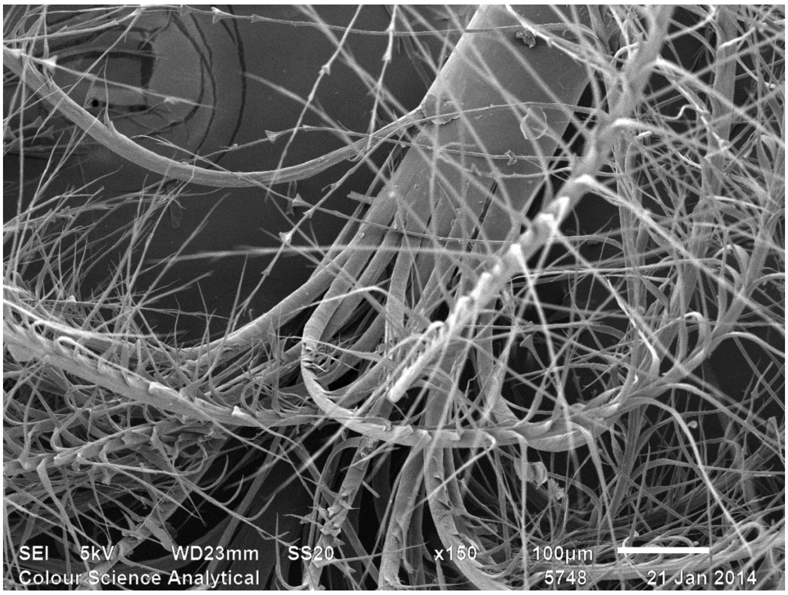


Figure 3-16 – SEM micrograph showing barbs leaving the core of a duck down plume

Larger down plumes which had a greater number of barbs had a slightly different geometry. In these, barbs divided from the length of the core, as shown in Figure 3-17:

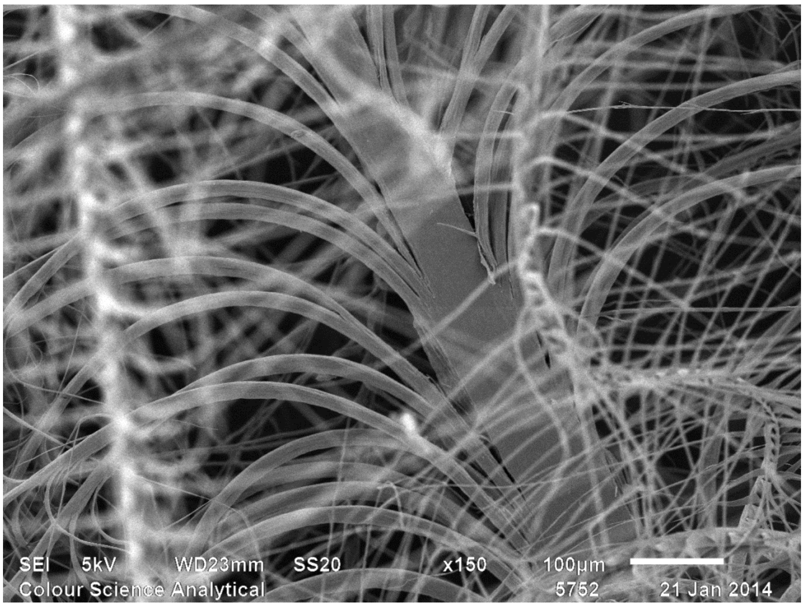


Figure 3-17 – SEM micrograph showing barbs leaving the core of a goose down plume

Barbs from the same plume tended to be very similar in length and, consistently, eider down barbs were longer than those from goose or duck down. Barbs from each type of down could be bent to extreme angles without apparent damage and could be straightened or untangled (see Figure 3-18) by shaking the plumes. Despite the extreme flexibility of down barbs, on occasion they could be broken. When they did, the interlocking barbules would often stop the complete separation of the broken barb, as shown in Figure 3-19:

Figure 3-18 – tangled goose down barbs

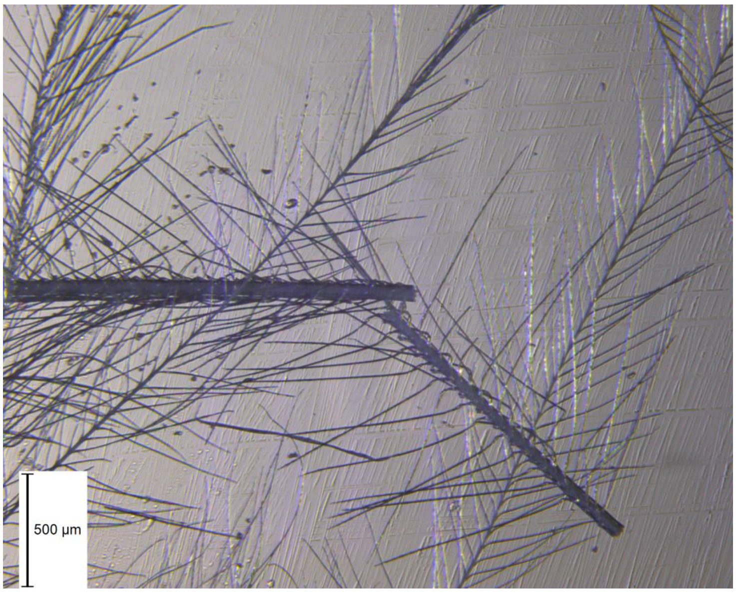


Figure 3-19 – broken duck down barb

The diameter of each barb was very consistent along their length, varying by less than 5 %: only the extreme distal end of the barb differed in diameter from the remainder. The diameters of goose and duck down barbs have previously been reported by numerous academic groups (Skelton et al. 1985; Loconti 1955; Gao, Yu & Pan 2007b) but to the author's knowledge, eider down has not been measured previously in this way. The mean and standard deviation values for the diameters of goose, duck and eider down barbs are shown in Table 3-4:

Table 3-4 - mean and standard deviation values for the diameter of goose, duck and eider down barbs

Down	Mean diameter (µm)	Diameter SD (µm)
Eider	20.81	4.24
Goose	18.93	4.69
Duck	19.21	4.72

The distributions of barb diameters are shown in the histograms in Figure 3-20:

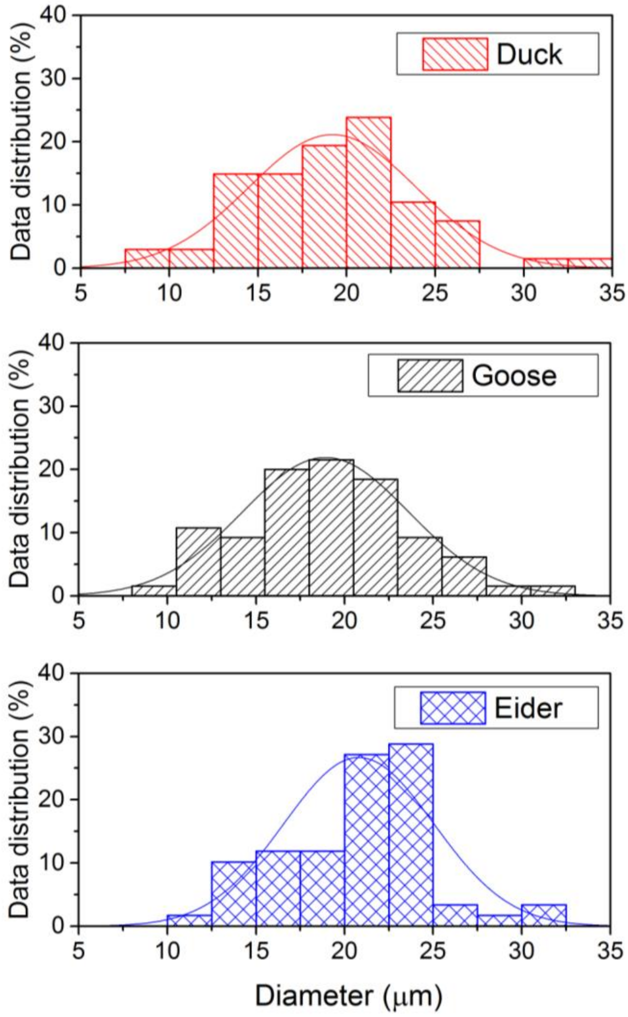


Figure 3-20 - histograms demonstrating the spread of diameters in duck, goose and eider down barbs
Curves are normal (Gaussian) distributions added using Origin Pro 8.1

As shown in Figure 3-20, the distributions of goose and duck down were approximately normal, typical of many real data populations. However, the distribution of barb diameters in the eider down was narrower (i.e. a positive degree of kurtosis), from 20-25 µm. Given that sampling took place in the same manner for all birds' feathers, and that the same number of data points was obtained for each, it seems likely that the distribution of eider down barb diameters is non-normal, and instead is closely controlled.

ANOVA statistics showed that goose and duck down's barb diameters were not significantly different to one another, but goose and duck down's barb diameters were significantly smaller than those of eider (p = 0.049 for eider compared to duck; p = 0.022 for eider compared to goose).

A comparison of the results in Table 3-4 with those of other researchers (Table 3-5) shows the great variability in these natural materials:

Table 3-5 – diameters of down feather barbs reported by previous researchers

Source bird	Diameter (µm)	Reference
Unstated	16-65	Skelton et al. (1985)
Goose	20-24	Loconti (1955)
Goose	8-20	Gao et al. (2007b)

The only previous direct comparison between the diameters of goose and duck down barbs was carried out by Bonser et al. (1999) who noted no measurable difference between the diameters of goose and duck down barbs, though they did not publish the mean values. Despite the variability in the diameters of goose and duck down, the values reported by Loconti are extremely close to those found here, and the spread of data is similar. The wide variability in diameters reported by Gao et al. and Skelton et al. may be a result of highly-variable down batches from birds of very different ages (it is assumed that feathers from older birds are likely to possess wider barbs), or it may be that the values they reported are the ranges, and not the means.

3.3.2 Down barbules

Barbs were furnished at regular intervals and along their entire length with barbules, as shown in Figure 3-21:

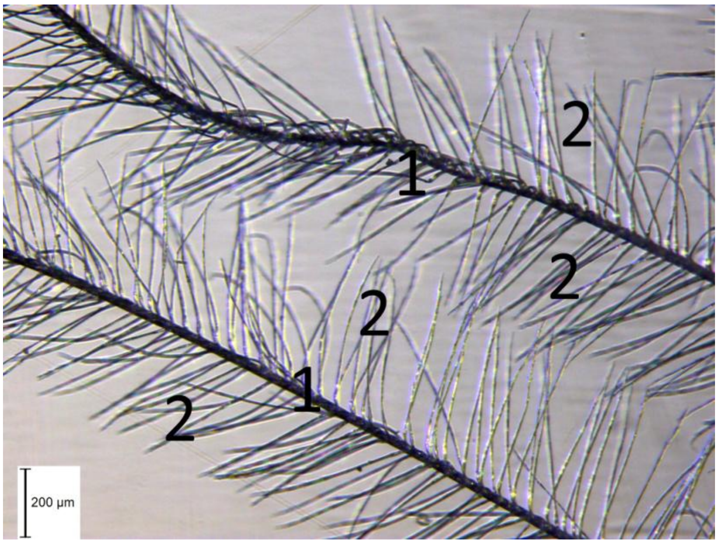


Figure 3-21 – goose down barbs and barbules
1 – barb; 2 - barbules

Barbules varied significantly in length depending on where they were located on the barb, and on the size and development of the feather. Larger feathers possessed the greatest discrepancy between barbule lengths: barbules at the proximal end of the barb tended to be shorter than those in the middle of the barb, and the barbule lengths tended to taper towards the distal end of a barb. This is expressed diagrammatically in Figure 3-22:

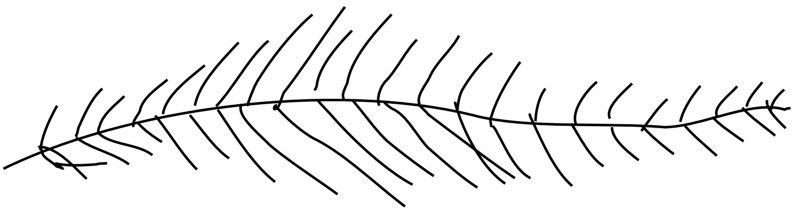


Figure 3-22 - diagrammatic representation of the changing lengths of barbules along the length of a duck, goose, or eider down barb
They are shortest at the proximal and distal ends

This disparity in barbule length was more exaggerated in eider down than in goose or duck down (see Figure 3-23):

Figure 3-23 - optical microscope image of an eider down barb showing the disparity in length between barbules on the distal and proximal ends, and those in the centre of the barb

In each type of down, shorter barbules near the plume centre (at the proximal end of the barb) ensured that adjacent barbs did not easily become entangled with one another, while also preventing the down plume from collapsing on itself. The longer barbules in the centre of the barb length had a larger area to project into than those nearer the centre of the barb, thus reducing the chance of entanglement with neighbouring barbs and barbules. The ability of the down plume to block radiative heat was maximised by these long barbules' large surface area, and prevented a formidable barrier to convective heat flow by minimising the space for air to move between them. Indeed, free convection cannot operate in a space smaller than approximately 8-13 mm (Morrissey & Rossi 2013), and so free convection in the down assembly can be ignored in most situations (it has been proven to be nearly zero in penguin feathers (Dawson et al. 1999)), and in use, down is usually contained in tightly-woven down-proof fabrics which restrict air-flow, so any forced convection must penetrate fabric and then a significant volume of barbs and barbules.

Barbules on a barb's distal end were smaller, which may be because they were less mature or because at this distance from the plume core, their mass and subsequent leverage may cause the barb to deform. Short barbules minimise the chance of this. In addition, shorter barbules reduce the chance of neighbouring plumes becoming locked with one another, which could potentially create an uneven and matted layer of down feathers.

In the feathers from all three source birds, barbules were flat when they split from the barb (see A in Figure 3-24, also Figure 3-25), and became more cylindrical as they extended towards the distal end of the barbule (B in Figure 3-24, also Figure 3-25).

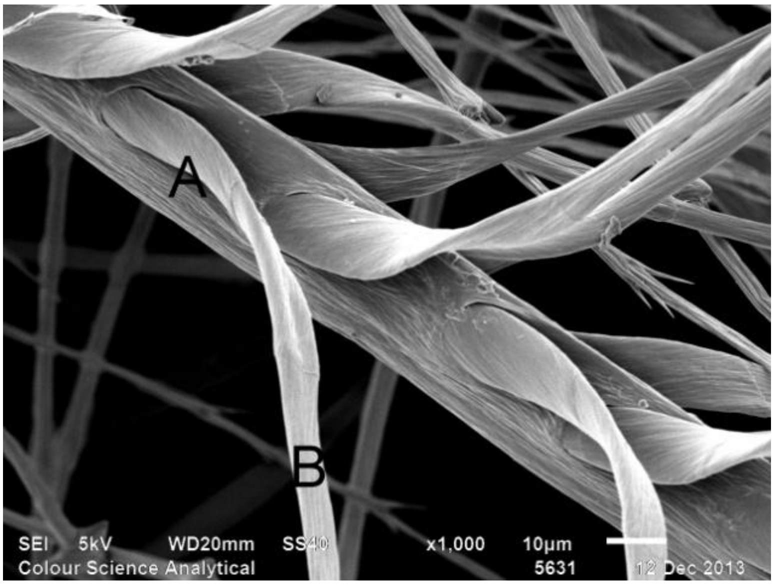


Figure 3-24 - SEM image of goose down barb and barbules
A – barbule is flat as it leaves the barb but – B – thickens to become more cylindrical

The barbules' planar proximal ends encourage bending to occur in one direction and this leads to the formation of a highly space-filling shape, while the more cylindrical barbule length maintains stiffness. The planar barbule proximal ends means that recovery from compression is likely to occur in a predetermined manner which might also minimise the chances of tangling adjacent barbules with one another. This planar barbule morphology is an essential feature in down's compression resistance and compression recovery and has not, to the author's knowledge, been reported previously.

The barbules in eider down (Figure 3-25) left the barbs at larger angles than observed in either goose or duck down (approximately 90 ° in eider down, compared to 60 ° in goose and duck down, quite consistent with the values measured in goose and duck down by previous researchers (Skelton et al. 1985; Gao et al. 2009; Yildiz et al. 2009; Wilde et al. 2006)). This created a hollow space-filling shape (Figure 3-26) that was first reported by Loconti (Loconti 1955), though his claim that the superstructure is cylindrical is perhaps exaggerated and potentially misleading. Regardless, this barbule superstructure forms a very effective three-dimensional network which might help resist compression and trap still air, both of which are crucial qualities for down feathers to act as effective insulators. In goose and duck down, similar superstructures were observed created by the barbules leaving the barbs (Figure 3-27 and Figure 3-28). In each of the birds' plumes, the barbules tended to alternate quite strictly in the direction that they left the barb: this maximised space-filling and made for a consistent shape with maximum strength along its length.

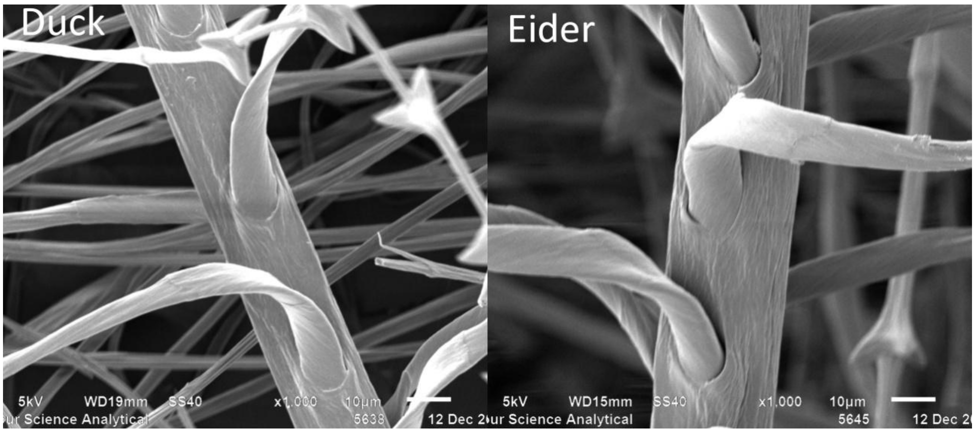


Figure 3-25 - SEM images of barbules dividing from barbs in duck (left) and eider (right) down Note the planar barbules as they leave the barb

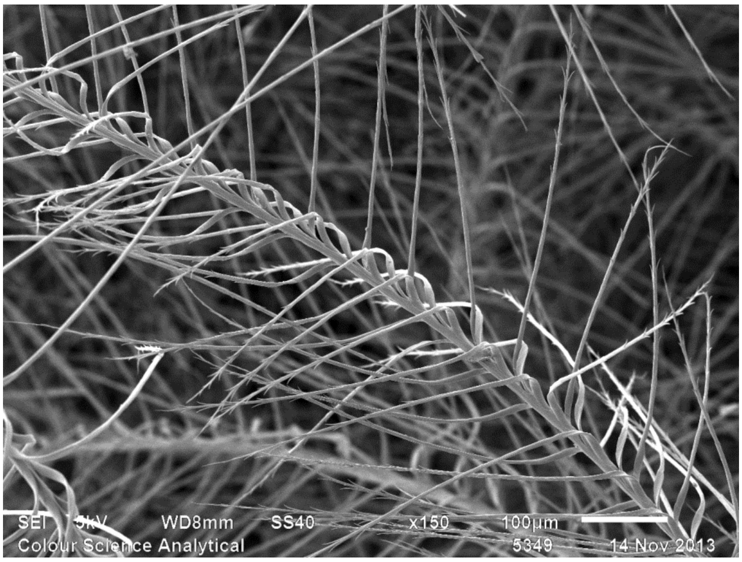


Figure 3-26 - SEM image of eider down barbules leaving the barb to create the superstructure of an almost cylinder-like shape, first noted by Loconti (1955)

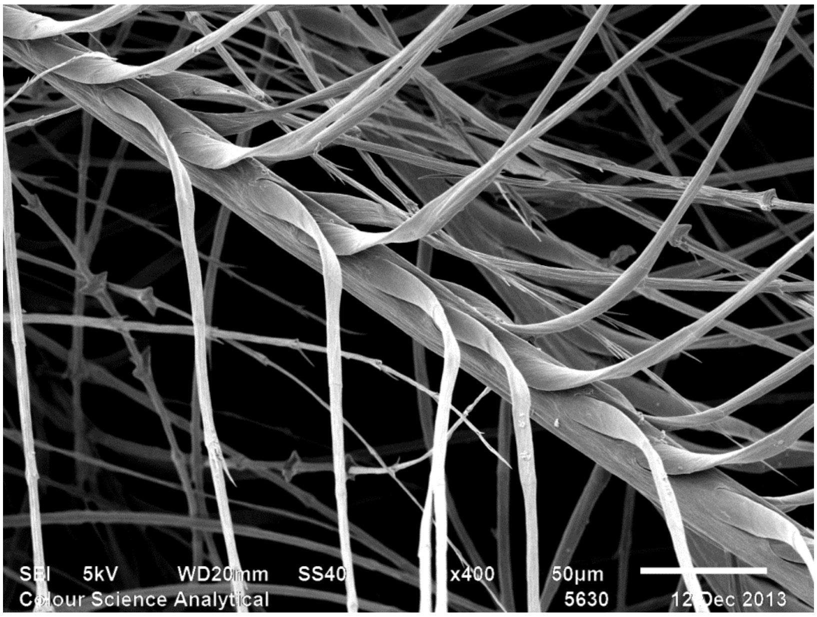


Figure 3-27 - SEM image of goose down barbules leaving the barb

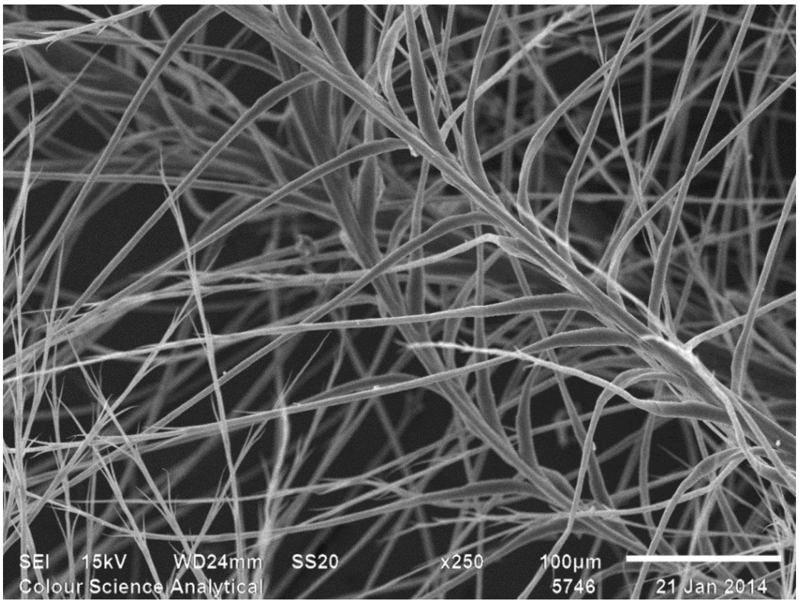


Figure 3-28 - SEM image of duck down barbules leaving the barb

Unlike the smooth fibres observed in many synthetic insulations, down's barbs and barbules were textured three-dimensional structures (see Figure 3-24, Figure 3-25). This is likely to be a result of growth rather than a result of trying to further minimise convection or thermal radiation. Despite the roughness of the barbs and barbules, ratchet-like scales that occur in wool or hair were not observed in down.

3.3.3 Tertiary structures

The morphology and prevalence of the tertiary structures, a term used to describe both nodes and prongs emanating from barbules (Wilde et al. 2006), were the greatest difference between goose, duck, and eider down plumes. As such, the tertiary structures in each of the bird's down plumes will be discussed separately.

3.3.3.1 Goose down

Most goose down barbules possessed nodes (Figure 3-29) but no prongs, though a small portion possessed prongs near the distal end of the barbules (Figure 3-24). Goose down's nodes were found in two different forms: large pyramids (see Figure 3-29) and in more stunted bud-like structures (Figure 3-30). On barbules at the distal end of goose barbs, nodes seldom occurred; when they did, they tended to be the more poorly-developed and stunted type.

The nodes in goose down (Figure 3-29) were trilobal and projected towards the distal end of the barbule to form a structure to stop the sliding of other surrounding fibres. Nodes may also increase the stiffness of the barbule without adding significant weight, as diaphragms do in bamboo (Chung & Yu 2002). Nodes were 3-4 times the diameter of the barbule, which compared favourably with the results of previous researchers (Gao, Yu & Pan 2007a; Skelton et al. 1985). Goose down's nodes have previously been described as isosceles triangles (Yan &

Wang 2009) and irregular tetrahedrons (Zhang et al. 2011), but these terms do not accurately describe what is a complex shape with few parallels in regular geometry. There was an average of two nodes per barbule, though some smaller barbules possessed no developed nodes at all (Figure 3-31).

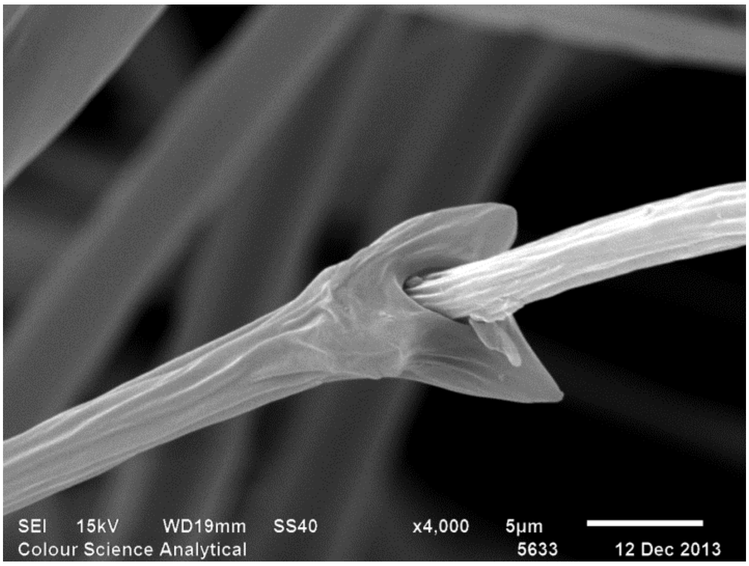


Figure 3-29 - SEM micrograph of a goose down node
The nodes of goose down were trilobal with a greater internal angle than those found in duck down nodes

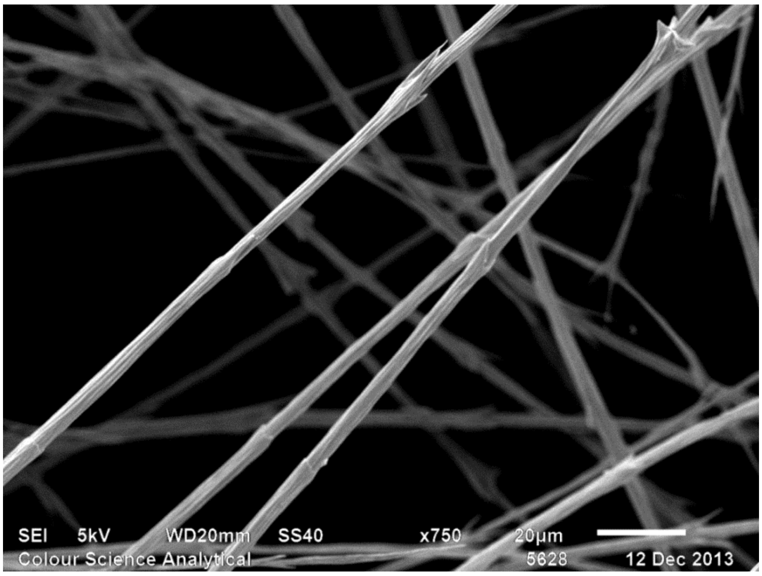


Figure 3-30 - SEM micrograph of stunted goose down nodes

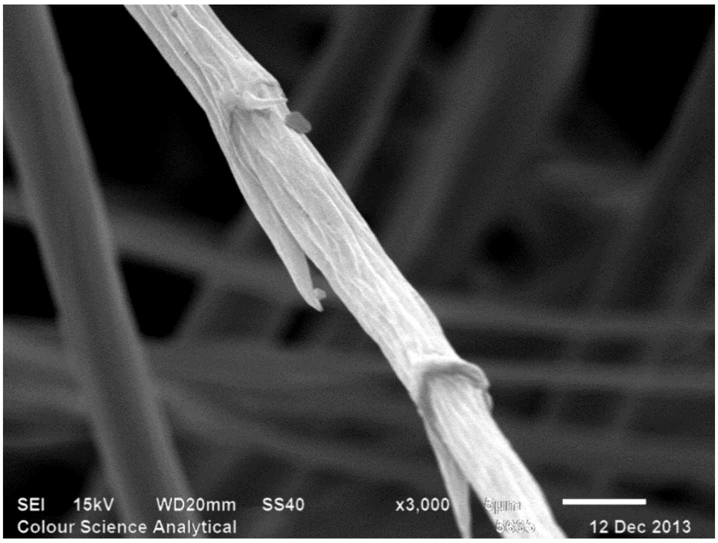


Figure 3-31 - SEM micrograph of very stunted goose down nodes

3.3.3.2 Duck down

Prongs and nodes were found on almost all duck down barbules. Duck down's prongs (Figure 3-32) extended in two directions from the barbule to form a fork-like geometry and nodes (Figure 3-33) extended in three directions.

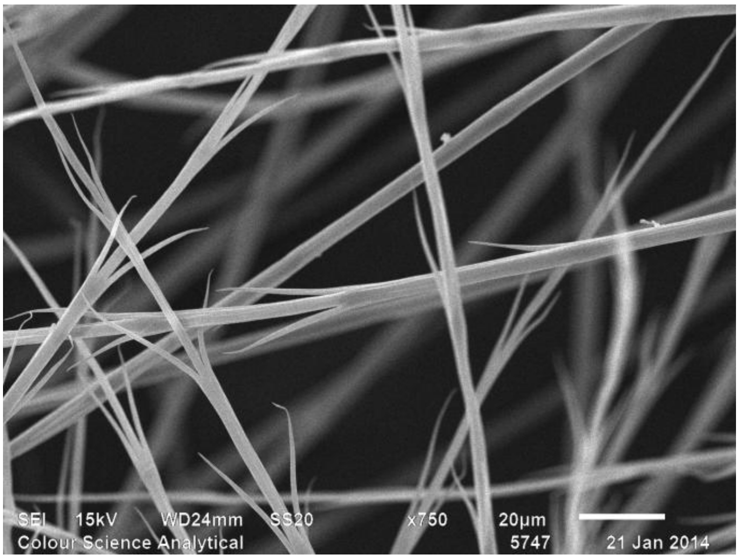


Figure 3-32 - SEM image of duck down's prongs

As reported in previous research (Zhang et al. 2011), duck down's nodes tended to be larger than those of goose down, being 5-6 times the diameter of the barbule, compared to approximately 3-4 times in goose down, and the distance between nodes in duck down was smaller (10-20 µm, see Figure 3-33) than in goose down (20-30 µm (Yan & Wang 2009)). There were approximately 5-8 nodes per barbule in duck down while there were approximately 2 nodes per barbule in goose down.

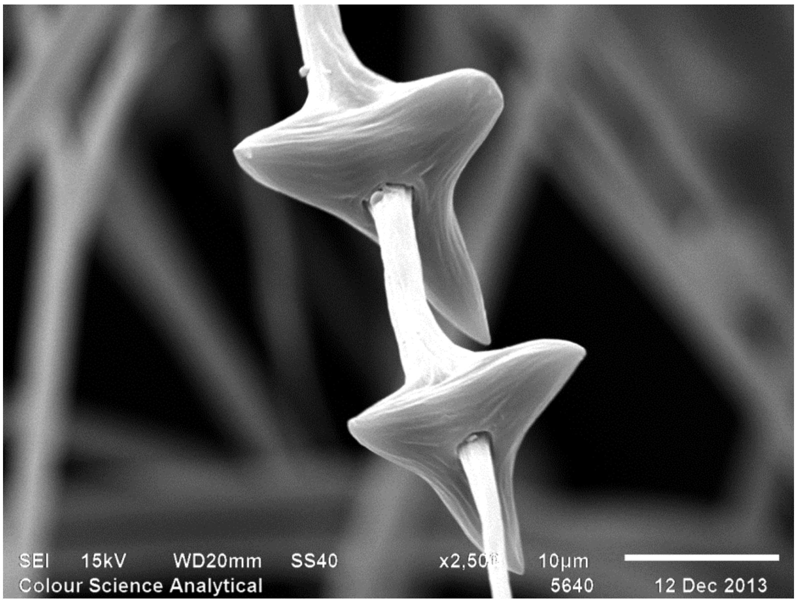


Figure 3-33 - SEM image of duck down nodes highlighting the small gaps between these structures and the difference in shape when compared to goose down's nodes

In addition to their greater size and number, duck down's nodes and prongs occurred only towards the distal end of a barbule (as reported in previous research (Gao, Yu & Pan 2007b; Gao, Yu & Pan 2007a)). Prongs did not occur on the same barbule as one that had nodes, and some shorter barbules had no nodes at all and instead they were lined entirely with prongs. The prevalence of prongs rather than nodes at the distal end of barbules is likely to be because of their lower mass reducing leverage and potential bending of the barbules. Prongs are also assumed to be less metabolically strenuous to grow than nodes, as they are smaller.

The most notable difference between the nodes of goose and duck down was their projected shape (see Figure 3-34 for a picture of duck down's). Duck down's nodes have been described as equilateral triangles (Yan & Wang 2009) and regular tetrahedrons (Zhang et al. 2011), and they extended further orthogonal to the barbule axis than goose down's but barely projected towards the barbule's distal end. This shape is assumed to trap other barbs and barbules less effectively than goose's node (because it is flat, as opposed to slightly hooked), but this potential ineffectiveness is counteracted by the greater number of nodes and by the presence of prongs, which also resist the slippage of barbs and barbules.

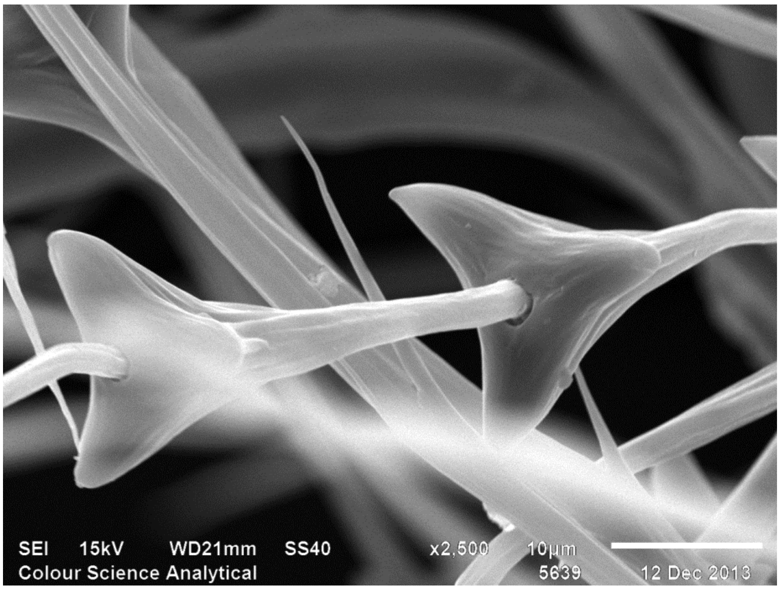


Figure 3-34 - SEM image of duck down nodes showing their frontal projection

The greater frequency of nodes and prongs (structures that maintain distance between plumes, stop excessive clumping of feathers, and provide compression resistance of individual feathers (Wilde 2004)) on duck down's barbules when compared to goose down may reflect the greater stresses that ducks, which are more aquatic than geese (Kear 2005b), must counteract.

3.3.3.3 Eider down

A greater number of nodes and prongs were found in eider down than in either goose or duck down. Eiders' down feathers had trident-like prongs, as can be seen in Figure 3-35 and Figure 3-36, rather than the fork-like prongs of commercial ducks' down feathers (see Figure 3-32). There were a great number of these large and trident-like prongs (Figure 3-36), present on the distal ends of each barbule.

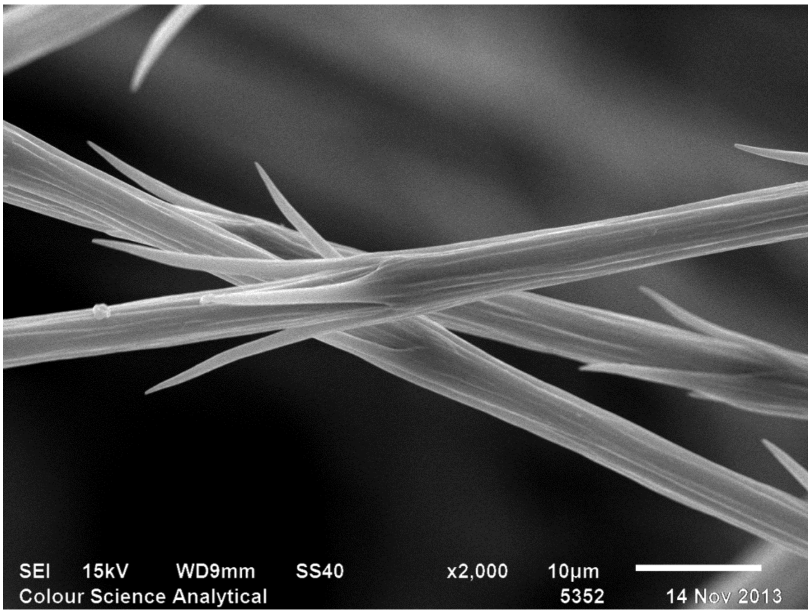


Figure 3-35 - SEM image of the trident-like prongs found in eider down

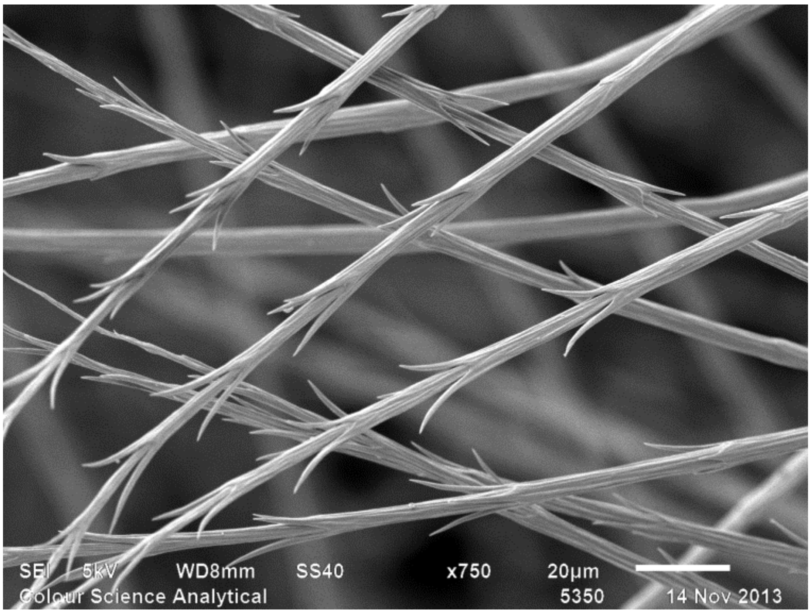


Figure 3-36 - SEM image of eider down's trilobal prongs

There were also two types of node observed in eider down, 'Type 1' and 'Type 2' (see Figure 3-37 and Figure 3-38). It is not certain if these nodes are two distinct structures or the same features in different stages of maturity. The nodes of Type 1 were somewhat similar to goose down's, with projecting lobes and little development orthogonal to the fibre axis (see Figure 3-37):

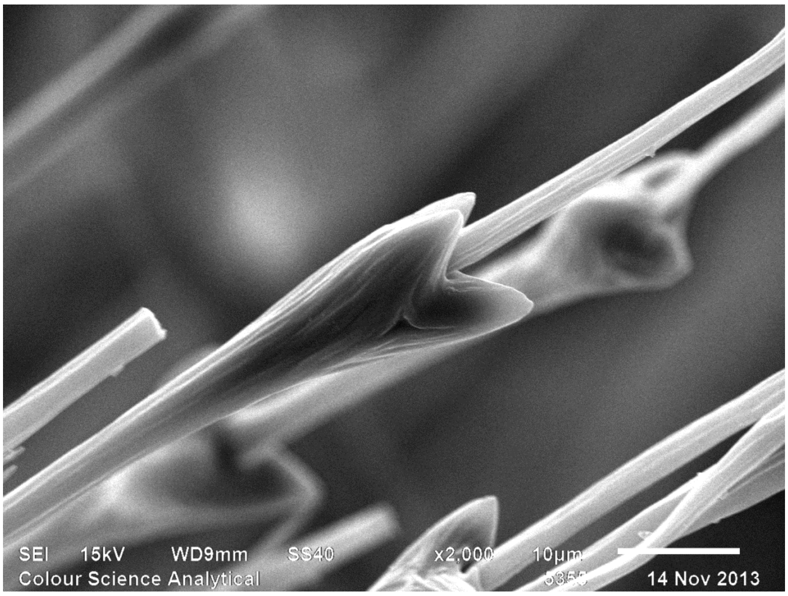


Figure 3-37 - SEM image of a 'Type 1' eider down node that was somewhat similar in shape to goose down's nodes

The nodes of Type 2 (Figure 3-38) were more similar to the nodes of duck down as they possessed more thickness orthogonal to the fibre axis but less projection towards the distal end of the barbule when compared to goose down's nodes.

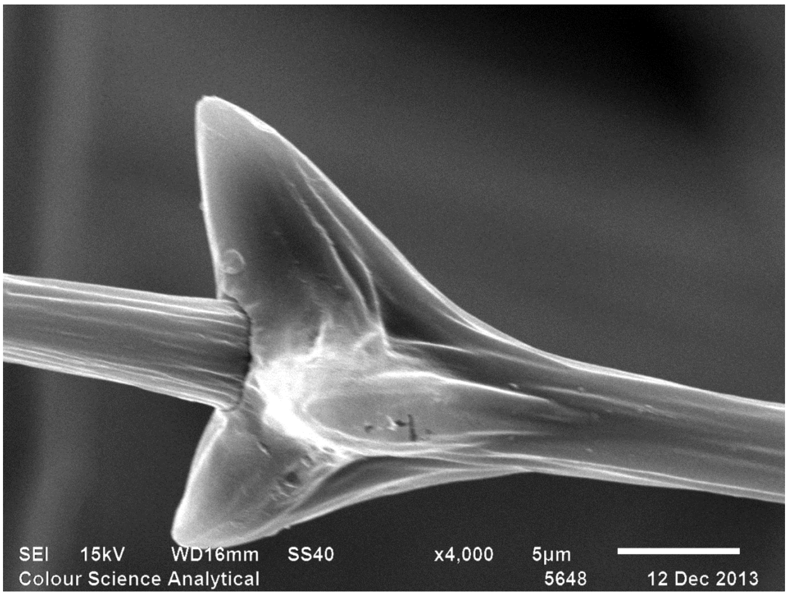


Figure 3-38 - SEM image of a 'Type 2' eider down node

There were significantly more nodes in eider down than in goose and duck down (see eider down Figure 3-39, goose down in Figure 3-27, and duck down in Figure 3-32), and while in duck and goose down some barbules had neither nodes nor prongs, all barbules in eider down seemed to possess them. These differences may indicate a greater feather maturity in eider down, because they are plucked by the bird itself during nest preparation (Bedard et al. 2008).

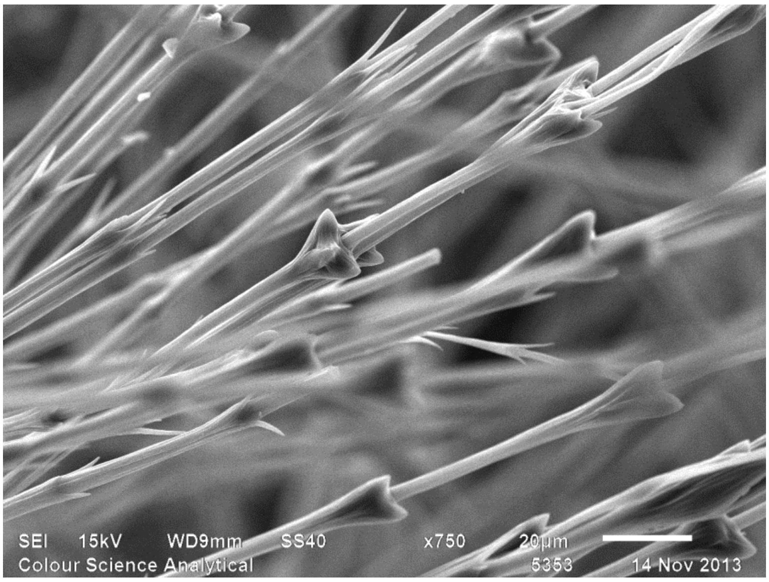


Figure 3-39 - SEM image of eider down's greater number of nodes when compared to either goose or duck down

In use, more nodes and prongs, coupled with longer barbules, leads to a greater interaction between barbs and plumes in eider down. These strong interactions are likely to impart a significant resistance to compression that would be desirable to a seabird that will be buffeted considerably more than farmed birds such as geese and ducks. However, once removed from the bird, the cling between eider down feathers that results from their prevalence of nodes and prongs could potentially lead to them becoming overly-matted and stuck together, making them unsuitable for long-term use in outdoor equipment. This may be a contributing factor in the lower fill power of duck down (IDFL 2010a) when compared to goose down, and why eider down has an inferior fill power (its high fill power is a "myth" (Sandvoss 2013)): it may be that the same structures that increase stress resistance can prevent eider down plumes lofting to their maximum capacity.

3.4 Summary

Some of the fundamental properties of the two major types of down feather used in the outdoor industry have been assessed alongside eider down, a much rarer and more expensive material. Each down feather serves the same purpose for its source bird – to provide insulation – and as a result, their properties are very similar in many regards. However, small details differ between the birds' feathers, and this reflects the environments that the birds inhabit.

1) Duck and goose down are very similar on initial inspection but eider down plumes are noticeably larger and heavier (average plumes masses: eider – 2.02 mg; goose – 0.77 mg; duck – 0.70 mg).

2) The diameter of eider down barbs is significantly greater than in goose and duck down (a mean diameter of 21.8 µm in eider versus 18.9 µm and 19.2 µm in goose and duck, respectively).
3) The near-spherical shape of down plumes can be approximated by the point group K_h.
4) The densities of goose and duck down are very similar (duck: 1.44 g cm^{-3} when undried, 1.27 g cm^{-3} when dried; goose: 1.42 g cm^{-3} when undried; 1.25 g cm^{-3} when dried). Thus, any differences in performance between these two materials are not merely the result of differences in density.
5) Barbs leave the down feather core in all directions and barbules emanate from these, initially utilising a planar cross-section to predetermine recovery from compression (a 'memory effect'), enabling both a greater resistance to compression and a better compression recovery.
6) The lengths of barbules are adapted to their position on the barb to maximise the filling of space while minimising mass and the potential entangling of adjacent plumes.
7) Eider down has the most tertiary structures (prongs and nodes), duck down the second most, and goose down the least. Goose down has very few prongs while duck and eider have numerous.
8) The nodes of each type of down are distinct from one another.
9) The greater mass, barb diameter, and number of tertiary structures all indicate that eider down is a structure designed to be extremely resilient and resistive to stresses. This reflects the harsh conditions that this sea bird must survive. Farmed geese and ducks, however, live relatively placid lives where they do not need to battle such severe sea and weather conditions and so their feathers do not require such stress resistance.
10) When used in outdoor equipment, goose and duck down, based on inspection here, are expected to have very similar properties, with duck down perhaps being marginally more resistant to compression owing to its greater number of tertiary structures. Eider down is expected to provide the greatest compression resistance, though its prohibitive cost and scarcity is a barrier to its widespread use.

Chapter 4. The microstructure of goose and duck down feathers

Comprehensive information regarding the microstructure of down feathers is vital to understanding their unique mechanical and thermal properties. While the microstructures of many keratin fibres including wool (Onions 1962; Fraser et al. 1972) and flight feathers (Filshie & Rogers 1962; Astbury & Beighton 1961; R. Schor & Krimm 1961; Pauling & Corey 1951) have been studied for decades, down feathers - which are morphologically very different to flight feathers - have been largely ignored. In this chapter, the microstructures of goose and duck down plumes are characterised by employing techniques including transmission electron microscopy (TEM), atomic force microscopy (AFM), X-ray diffraction (XRD), and differential scanning calorimetry (DSC).

4.1 The cross sections of down barbs and barbules

Despite numerous papers examining down's external structure, to the author's knowledge, only one paper has studied the cross-section of goose down. Gao et al. (Gao, Yu & Pan 2007b) used transmission electron microscopy (TEM) to identify an epicuticle, cuticle, "skin", cortex, macrofibrils, and fibril-matrix, but as described in section 1.3.2 their pictures were unclear and lacked important details. Here, both goose and duck down will be analysed, and where relevant, their structures compared to those of wool and hair.

The cross sectional shape of down barbs and barbules has previously been assumed by some academics to be circular (Bonser & Farrent 2001; Bonser & Dawson 1999), but the single TEM image supplied by Gao et al. (2007b) suggested that they may be more irregular in shape. This required further investigation, but the low throughput of TEM and its complex sample preparation meant that it was not a good method for studying many different down plumes at once, and SEM offered a much better method. In this experiment, a Hardy Microtome was used to section goose, duck, and eider down plumes, and SEM carried out. These techniques are described in section 2.2.2.2.

For analysis of fine structure in the down feather cross sections, TEM and AFM were used. TEM is regarded as the most important method used to observe keratin fibres' ultra-fine internal structure because of its relative ease of use, very high resolution, and ability to differentiate between regions of different chemical properties (Kitano et al. 2009). The method used to prepare the down samples for TEM is described in section 2.2.3.1 and the TEM procedure is listed in section 2.2.3.2. The staining in the preparation method increased contrast between the electron-scattering heavy metals and the electron-transparent lighter elements (Phan 1991).

To the author's knowledge, down feathers have not previously been characterised by AFM, though other keratins have been studied (Smith & Swift 2002). AFM's exceptional resolution is hoped to resolve some of the smallest features of down: their microfibrils, estimated to be 3 nm in diameter (Filshie & Rogers 1962). The method used for embedding the AFM samples is described in section 2.2.3.1, and the AFM method is described in section 2.2.4.

4.1.1 Duck down

When viewed using SEM the barb and barbule cross sections appeared as irregular shapes surrounded by resin, as shown in Figure 4-1:

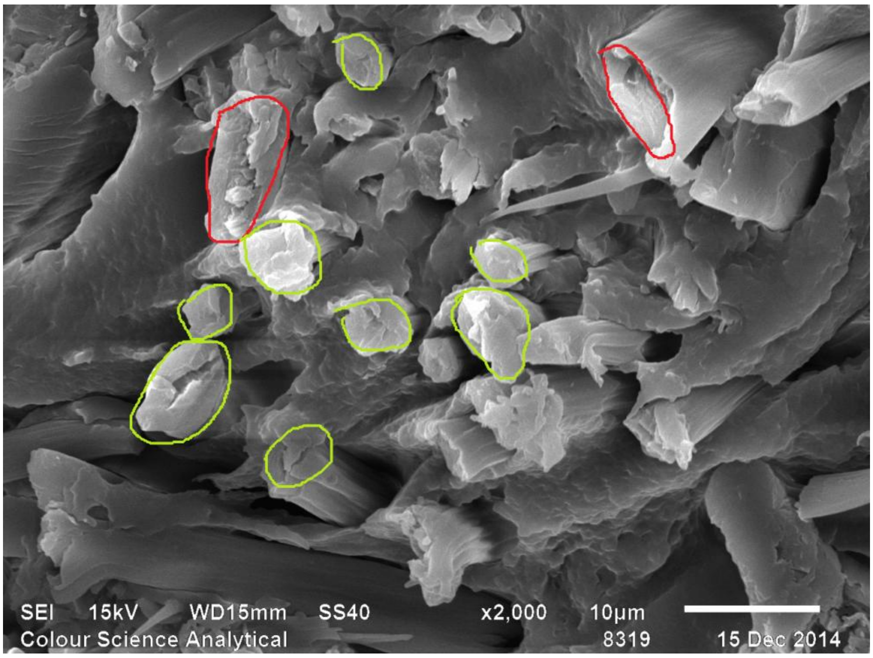


Figure 4-1 – SEM image of the cross sections of duck down barbs and barbules
Green outline added to highlight the barbule cross sections; red outline added to highlight the barb cross sections

In each of the samples there was great variety in the shapes of the barbules – some were approximately circular, whereas others were much more oval-shaped. The barbs were less varied in shape, and were approximately oval-shaped. However, the cutting process involved in preparing cross sections for SEM analysis can deform the barbs and barbules, so more accurate (but lower throughput) cross-sectional geometries were obtained using TEM. In the TEM analyses, the duck down barbules and barbs were sectioned alongside one another and possessed quite similar morphologies, as shown in Figure 4-2:

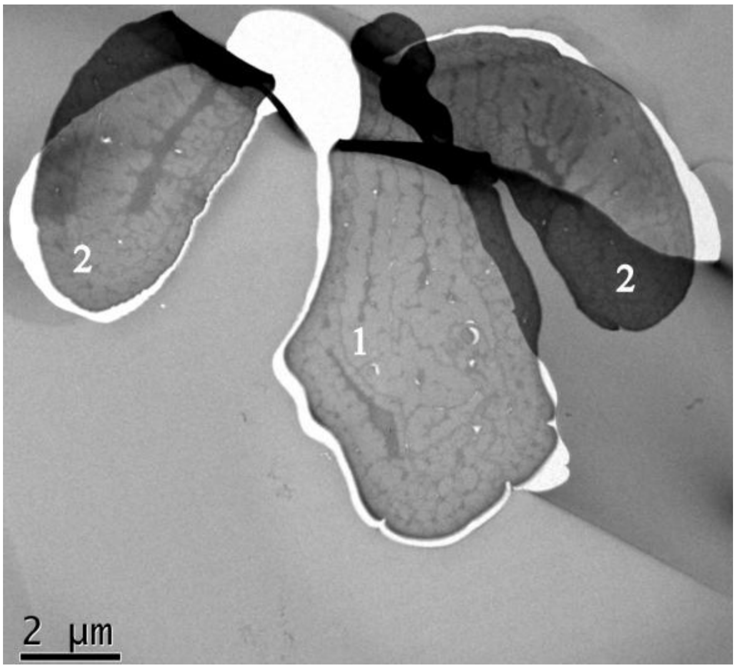


Figure 4-2 - duck down feather cross section
1 – barb; 2 - barbules

As in wool and hair (Rogers 1959), each duck down feather's barb's and barbules' cross sections were composed of a cuticle and a cortex. The cortex was further divided into two quite distinct regions: the orthocortex and paracortex. These features can be seen in Figure 4-3:

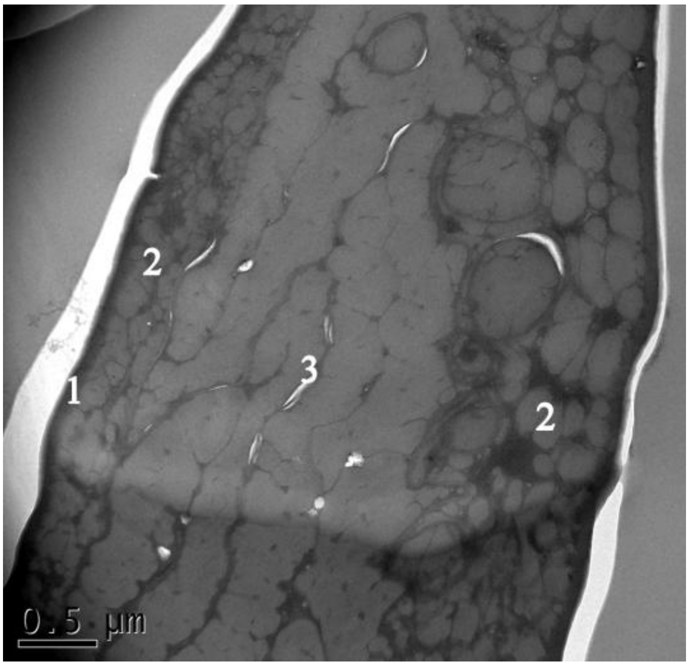


Figure 4-3 - duck down barb cross section showing the cuticle, orthocortex, and paracortex
1 – cuticle; 2 – orthocortex; 3 – paracortex

4.1.1.1 The cuticle

The electron-dense cuticle absorbed high concentrations of the heavy metal stains and thus appeared as a dark boundary to the barbs and barbules (Figure 4-4), as areas with highest electron density correspond to those with elements of highest atomic number (Imai 2011). Wool's cuticle comprises an epicuticle, exocuticle and endocuticle (Marshall et al. 1991) but the very small thickness of down's cuticle (approximately 60 nm, versus approximately 500 nm (Bradbury 1973) in wool) meant it was not possible to reliably differentiate between all these layers in the down samples, though the endocuticle was identifiable (Figure 4-4) as a slightly lighter-dyed portion of cuticle. In wool, the endocuticle has lower cysteine content than other parts of the cuticle (Leeder 1986) because it is inhomogeneous and non-keratinous (Phan 1991) and this would explain its relative transparency to the electron beam. The endocuticle is regarded as the most well-defined layer in the cuticle in wool (Bradbury 1973), consistent with the observation of this layer rather than the others. The "skin" layer that Gao et al. (2007b) observed was not identifiable in the present samples and perhaps requires a more precise definition. The epicuticle that they identified was also not observed. As the epicuticle is approximately 13 nm wide in wool (Swift & Smith 2001) and 3 nm wide in hair (Jachowicz 1987), it seems unlikely that such a small structure could be identified with certainty in down barbs, which are much finer than wool or hair fibres.

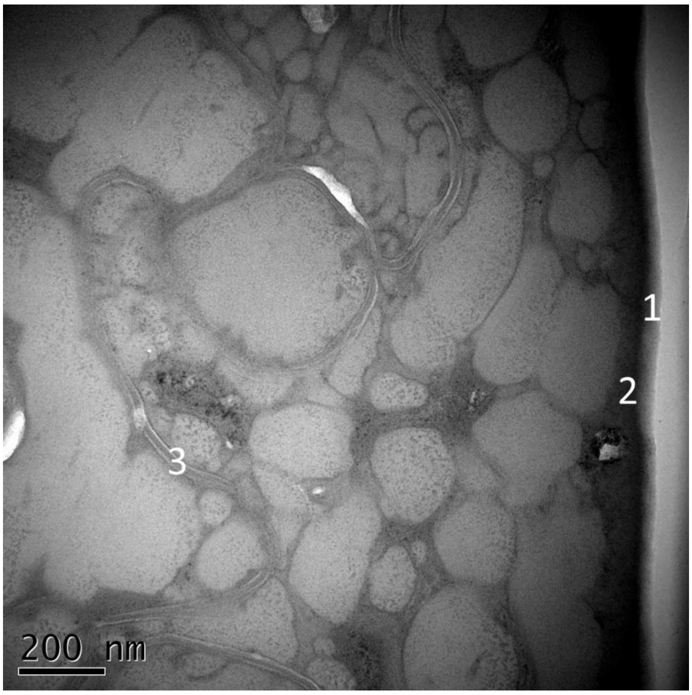


Figure 4-4 – duck down cross section
The cuticle (1) is present as a dark area surrounding the down cross section. The endocuticle (2) is the innermost part of the cuticle. 3 is the cell membrane complex displaying distinct β and δ layers

In wool (Phan 1991) and hair (Wolfram 1971) the cuticle is chemically-protective and contributes to hydrophobicity. It seems logical that the cuticle functions in the same way in down, providing resistance to potentially-harmful agents that may come into contact with the down feather; all chemical interactions with the environment must occur through the cuticle. Because the cuticle is such a small part of down's cross section, it is unlikely to contribute to down's tensile properties.

Previously, cuticle cells have thought to be "restricted to wool and hairs" (Marshall et al. 1991) and previous papers (Filshie & Rogers 1962) analysing flight feathers have not made mention to a feather cuticle. Flight feathers, however, are very different structures to those of down: flight feathers' components comprise barbs, calamus, rachis, medulla, and pulp-cap (Earland et al. 1962), very different to those of down. Researchers have often analysed one of these components independently of the others but the previous identification of a cuticle in feathers has not been found by the author. It may be that down feathers can be thought of as a morphological combination of feathers (being fractal (J. Gao, Pan & W. D. Yu 2007) and composed of barbs and finer structures such as nodes) and wool (being much finer than flight feathers and possessing a cuticle). Considering their function as insulation, and potentially as buoyancy aids (Stephenson 1993) it is unsurprising that down's fine morphology differs from that of flight feathers, the primary purpose of which is to provide lift during flight.

4.1.1.2 The cortex

In the present investigation, numerous details in the cortex were revealed that were not reported by past researchers (Gao, Yu & Pan 2007b), such as the distinct difference between the interior and exterior of the cortex: the paracortex and orthocortex, respectively (see Figure 4-3). The down cortex was the largest part of the duck down barbs and barbules (approximately 95 % of area), and the paracortex accounted for 50 % of the cortex's total area in the barbs (n = 3) and was dominant in the barbules (56 % of total area; n = 6). As the barbs were of greater diameter than the barbules, this followed the general trend observed in wool (Marshall et al. 1991) that as the diameter of the fibre increases, so does the proportion of orthocortical cells. Nutritional stress is thought to increase the proportion of orthocortical cells in wool (Hynd 1989; Orwin et al. 1984) and assuming the same pattern is seen in birds, this would explain the orthocortex to paracortex ratios observed in these samples from the well-fed ducks of commercial farming. Comparisons between wild ducks – assumed to have less available food – and farmed ducks would be interesting to determine whether this same correlation is observed.

The paracortex was in the centre of the down barbs and barbules, as in many wool samples (Bradbury 1973). It was composed of individual cells each separated by a cell membrane complex (CMC). These paracortical cells were further subdivided into individual macrofibrils, separated by intermacrofibrillar material. These features are described in Figure 4-5 and are also observed in wool (MacLaren & Milligan 1981).

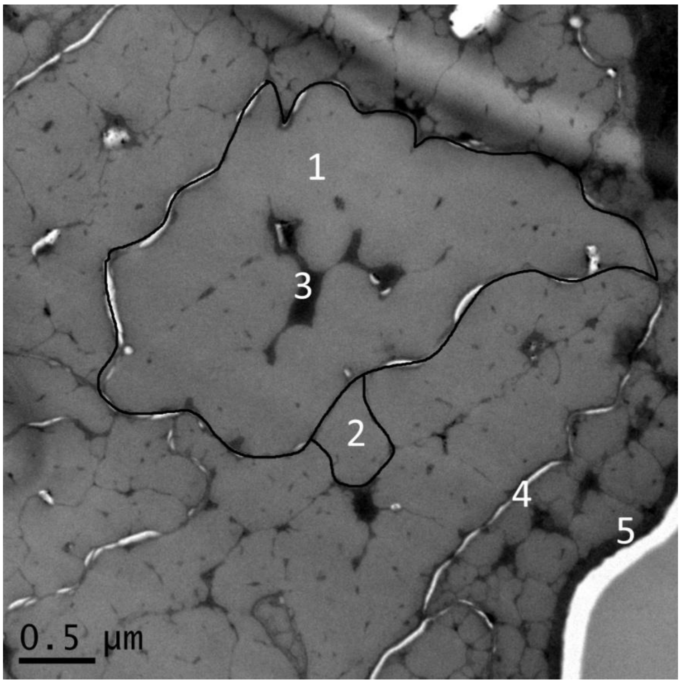


Figure 4-5 - annotated TEM micrograph of the paracortex in a duck down barb
1 - paracortical cell (black border added for emphasis); 2 - paracortical macrofibril (border added for emphasis); 3 - nuclear remnant; 4 - orthocortical/paracortical cell divide; 5 - cuticle

The relative size of the duck down barbs and barbules when compared to a wool fibre meant that there were rarely more than 4 cells in the paracortex whereas a wool fibre cross section might typically contain 20 paracortical cells (MacLaren & Milligan 1981). Paracortical cells varied significantly in shape and size, though the 5 µm diameter of typical paracortical cells in wool (MacLaren & Milligan 1981) was comparable to that found in the duck down samples.

The boundaries of individual macrofibrils in the paracortex were quite difficult to determine, mainly due to the relative lack of densely-dyed intermacrofibrillar material when compared to the orthocortex. This has also been noted in wool (Jones et al. 2006). Because of the relative difficulty in reliable determination of their size, no measurements of macrofibrils were made, but the 0.1-0.3 µm diameter macrofibrils reported in wool (MacLaren & Milligan 1981; Caven 2010; Phan 1991) is approximately comparable to that observed here.

In the paracortex, the osmiophilic nuclear remnants (Figure 4-6) were either found in the centre of the cell or stretched into dendrites (long branched projections). In the barbules, the nuclear remnants often formed a greater portion of the overall fibre volume than in the barbs, as seen in Figure 4-2. These nuclear remnants are also found in wool and are the remains of the once-living cell (Bradbury 1973).

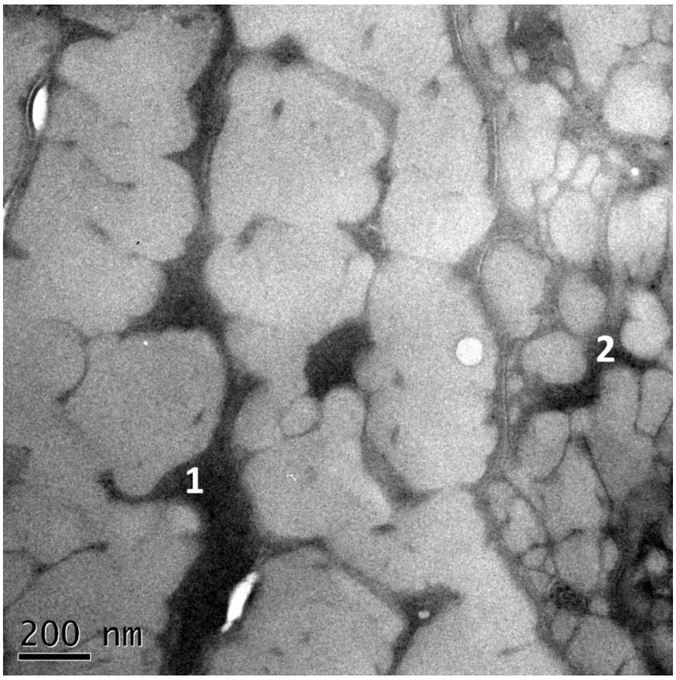


Figure 4-6 - duck down barb cross section highlighting densely-stained nuclear remnants
1 - nuclear remnants in the paracortex; 2 - nuclear remnants in the orthocortex

The smallest features of the keratin cells observable using this iteration of transmission electron microscopy were the microfibrils. Microfibrils are crystalline components packed consistently along the fibre axis (Rogers 1959). In wool they are 7-8 nm across (Anderson et al. 1972) and in flight feathers are approximately 3 nm in diameter (Filshie & Rogers 1962). Conventional lead hydroxide or osmium tetroxide stains used either alone or in combination cannot resolve them (Filshie & Rogers 1962) but lead hydroxide can (Filshie & Rogers 1962), and the Reynolds' solution (lead citrate) as used in the preparations here functions in the same way (Reynolds 1963). Even with this staining procedure, however, the microfibrils were not particularly distinct in these samples and this is thought to be due to the thickness of the cross sections reducing electron flux and subsequent resolution. Very faint microfibrils tended to be observed at the perimeter of the samples, suggesting that a longer exposure time in the lead citrate may allow it to penetrate further and aid resolution of the microfibrils. Microfibrils can be observed in Figure 4-4, and in Figure 4-7, though at such high magnification it is difficult to differentiate between noise and the microfibrils.

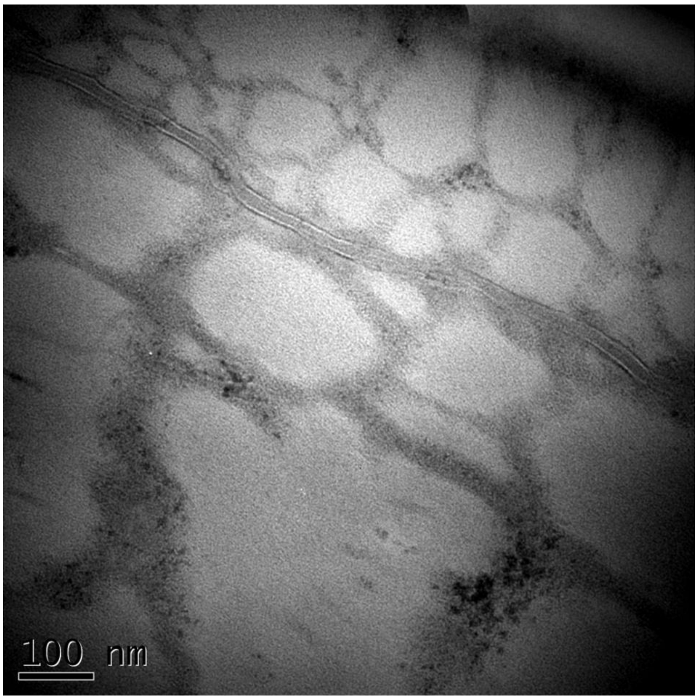


Figure 4-7 - duck down sample viewed through TEM
Indistinct microfibrils in both the paracortex and orthocortex are observable as a 'texture' to the lesser-dyed macrofibrils

Individual orthocortical cells were easy to identify due to the cell membrane complex that surrounded them. The orthocortical macrofibrils inside these were arranged irregularly (see Figure 4-8), as in wool (Onions 1962), and easy to differentiate between due to the non-keratinous material between them (Rogers 1959). This intermacrofibrillar material is expected to make the orthocortex more penetrable to liquids than the paracortex (Bradbury 1973), meaning that once the hydrophobic cuticle is penetrated by water, the orthocortex will readily uptake water and swell the down feather.

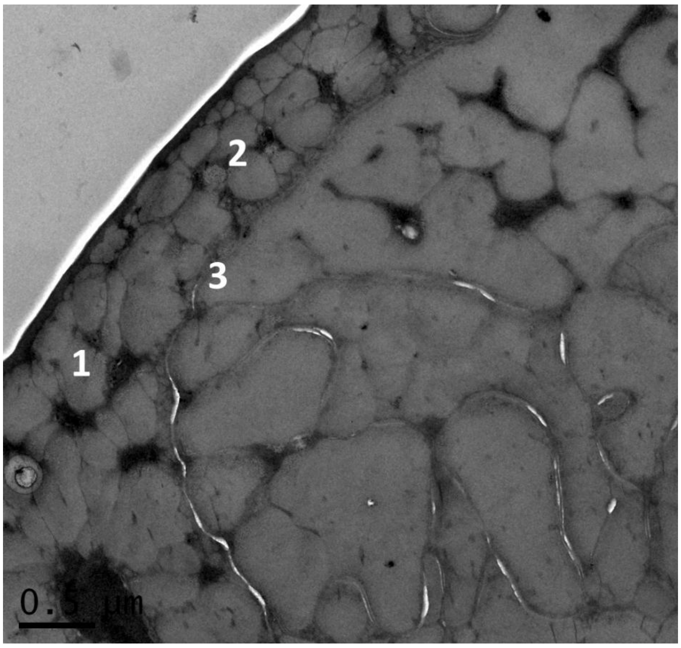


Figure 4-8 - duck down barbule cross section viewed through TEM
1 - orthocortical macrofibril; 2 - intermacrofibrillar material; 3 - cell membrane complex dividing orthocortex and paracortex

The cell membrane complex (CMC), present in wool as a three-layer “tramline” (Leeder 1986) structure of the densely-stained β layer surrounding the lightly-stained δ layer (Rogers 1959; Leeder 1986), was observed in each duck down barb and barbule. It is shown in Figure 4-4 and Figure 4-9 and averaged 29 nm in width (n = 59 from 7 samples, SD = 5 nm). This is consistent with the “approximately 30 nm” reported by Phan (1991) in studies on wool.

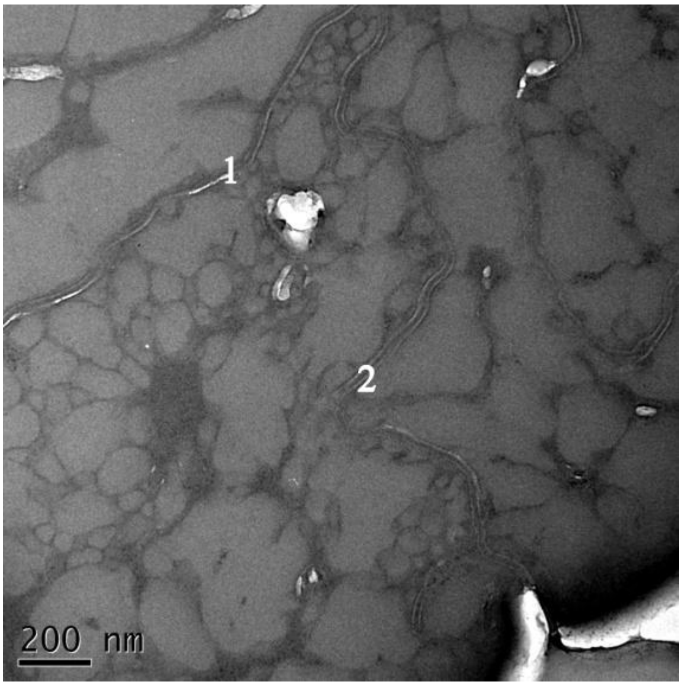


Figure 4-9 - duck down barb viewed with TEM showing the cell membrane complex at points 1 and 2

Despite its diminutive size, the CMC is thought to be important to down's properties, as it is in wool (Leeder 1986). This is because it holds cortical cells together, and a composite structure is only as strong as its weakest link (Mercer 1961). As seen in the white areas of Figure 4-8, TEM samples often separated or internally fibrillated at the CMC under mechanical stresses (such as during sample preparation) and this is evidence for their weak structure. In wool, an increase in humidity has been found to swell the CMC but also increase its abrasion resistance (Nhan & Denby 1979), probably because the swollen structure is more able to dissipate applied stresses (Leeder 1986). It is likely that the same effect would be observed in down, but the contravening effect of decreasing modulus (Bonser & Farrent 2001) would be more important in determining the overall properties of the feathers.

4.1.2 Goose Down

SEM analysis (Figure 4-10) indicated that the goose down barbs may be hollow. As observed in duck down, there were a large variety of cross-sectional shapes in the barbs and barbules (Figure 4-10 and Figure 4-11):

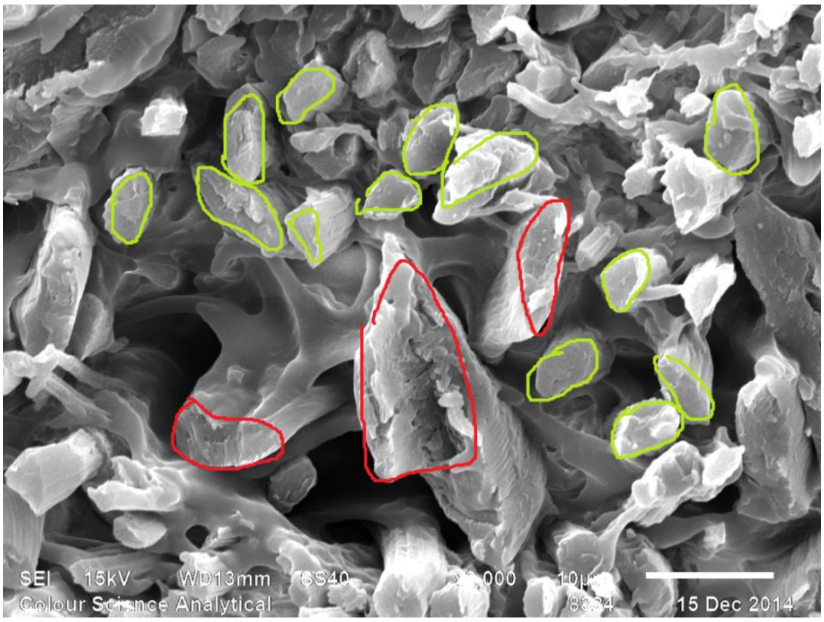


Figure 4-10 - cross sections of goose down barbs and barbules observed using SEM
Note the possible hollow centre to the central goose down barb. Green outline added to highlight the cross sections of barbules; red outline added to highlight the cross sections of barbs

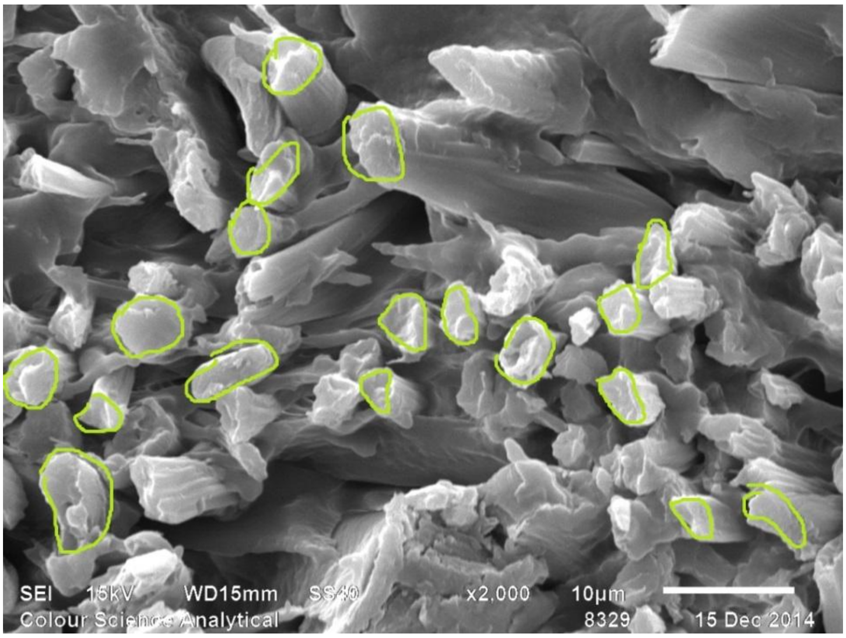


Figure 4-11 - cross section of goose down barbules
Green outline added to highlight barbule cross sections

The TEM analysis showed that many of the features present in duck down were also found in goose down, though there were also crucial differences. A distinctive characteristic of the examined goose down barbs was their cross-sectional shape (see Figure 4-12) as unlike the rounded cross-sections of the duck down barbs, each goose down barb was approximately triangular. The barbules' cross sections were approximately elliptical, as had been observed in duck down. There is evidence that a triangular cross-section can impart a greater bending modulus than a circular or rectangular cross-section (Bond et al. 2002) and so the triangular cross-section of goose down fibres could impact on their compression resistance. This will be determined in subsequent chapters.

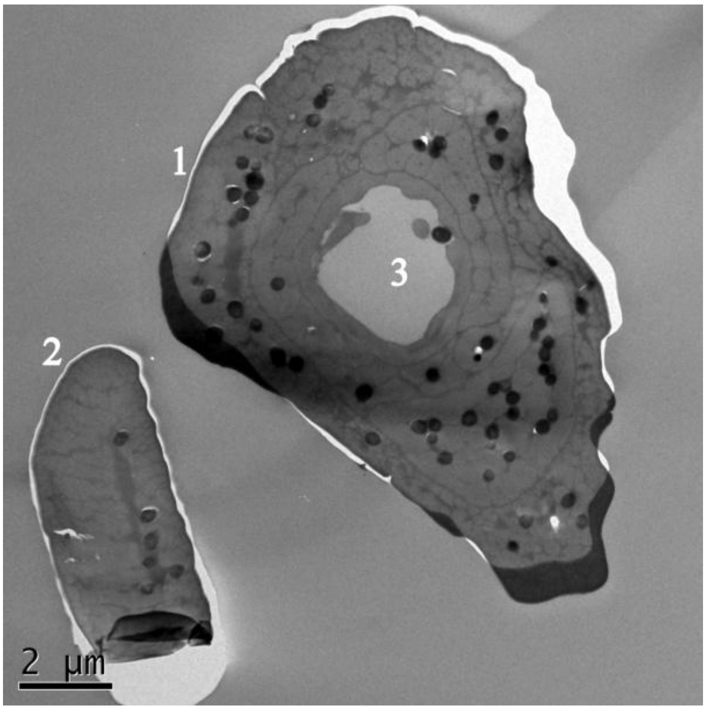


Figure 4-12 - cross section of goose down viewed with TEM
1 – barb; 2 – barbule; 3 – possible void/medulla

4.1.2.1 The cuticle

As in the duck down samples, a dark region surrounded each of the goose down fibres and represented the cuticle. This can be seen in Figure 4-13 and is thought to function in the same way as the equivalent structure in duck down:

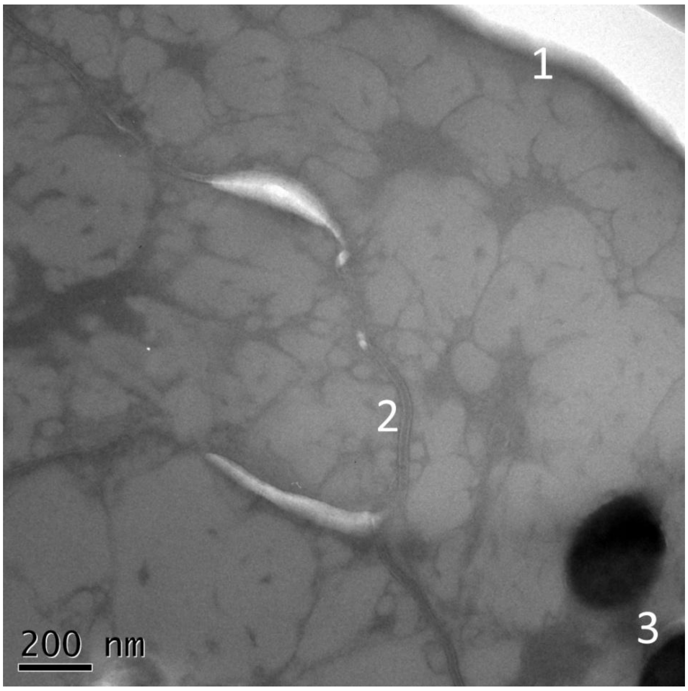


Figure 4-13 - goose down section viewed with TEM
1 - cuticle; 2 - CMC, 3 - possible melanin granules

4.1.2.2 The cortex and possible void or medulla

The greatest differences between the goose and duck down samples were in the cortex. Each of the goose down samples contained a central section distinct from the paracortex that was highly transparent to the electron beam and appeared to be of identical composition to the resin surrounding the samples. This can be seen in Figure 4-12 and Figure 4-14:

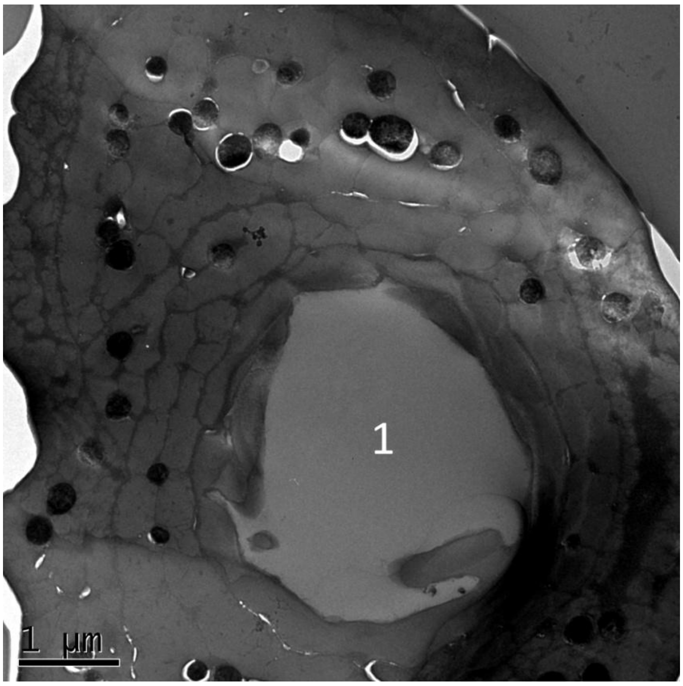


Figure 4-14 - goose down barb cross section
1 = possible void or medulla

From the TEM images it was unclear whether this was a part of the fibre that had failed to be penetrated by staining agents, whether this was a new morphology, or whether this was a void in the barb and it was actually hollow, as previously suggested by some researchers (Bonser & Dawson 1999). The structure also bared resemblance to wool's air-filled network of membranes and interstices (Onions 1962) known as the medulla. The central structure was present in each of the goose down samples and averaged an area of 7.7 μm^2 (n = 3), making up 10 % of the total fibre cross section. In each barb it was surrounded by paracortical cells, and occurred very near the centre of each barb. It was not observed in the barbules.

As in duck down, there was a distinct division of paracortex and orthocortex in the barbs, though this difference was less obvious in the barbules (see Figure 4-12). In the barbs, 43 % of the total area was unmedullated paracortex and 47 % of the area was orthocortex (n = 3), comparable to the ratios in duck down.

The cortex's cells in the barbs were clearly divided by cell membranes (see Figure 4-15). This structure was 27 nm in diameter (n = 41 measurements, SD = 12 nm), similar to the 29 nm measured in duck down, and also contained the distinct β and δ layers. The cell membrane complex was present in both the barbs and barbules.

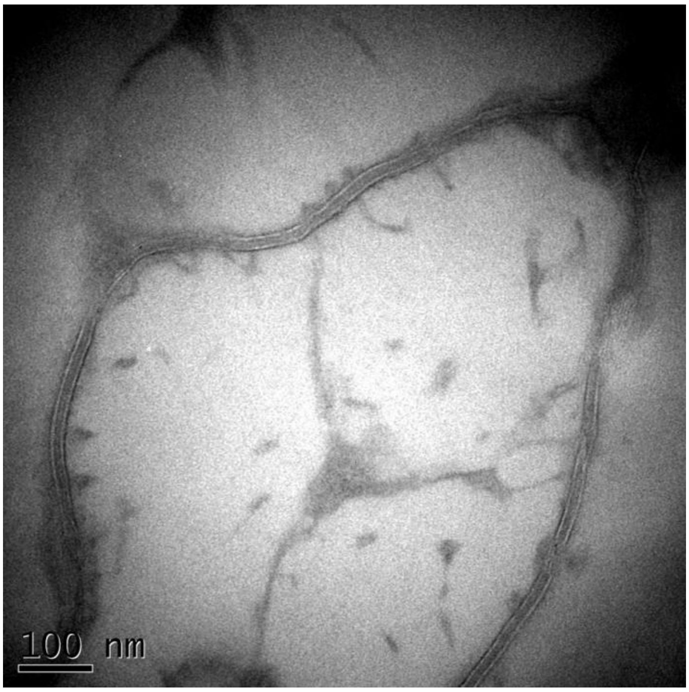


Figure 4-15 - goose down barb cross section highlighting the cell membrane complex

Unlike in duck down, the division of paracortical cells was quite clear, as intermacrofibrillar material divided them. This can be seen in Figure 4-16:

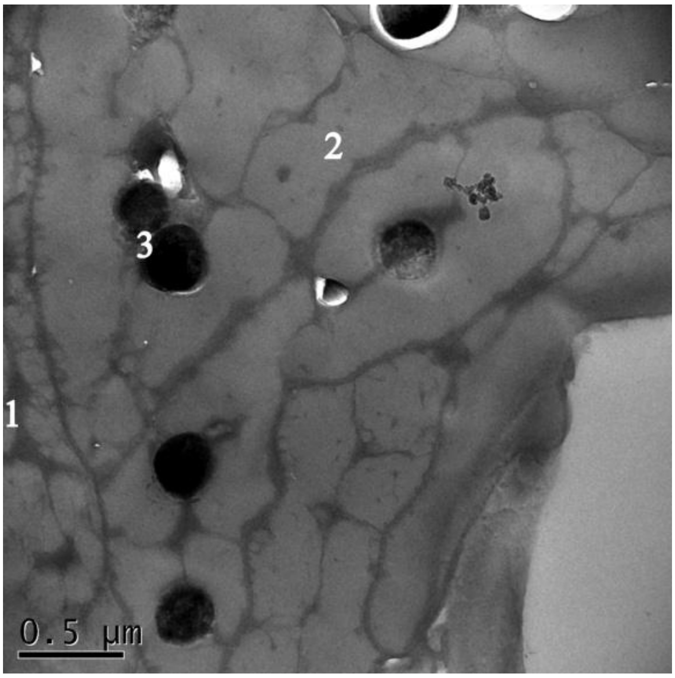


Figure 4-16 - goose down cross section showing paracortical cells and macrofibrils
1 – orthocortex; 2 – paracortical macrofibril; 3 – possible melanin granules

Another major difference between the goose and duck down morphologies was the presence of osmiophilic near-spherical areas present in the goose down samples (see Figure 4-16 and Figure 4-17). These have not been previously observed in down. The concentration of these granules was greater in the barb than in the barbules, and they were present in both paracortex and orthocortex. These granules were not uniformly stained (see Figure 4-17), indicating possible further substructure. The granules tended to aggregate to form approximately ring-like shapes around the barbs and barbules. It was unusual for them to be found very near the fibre perimeter. Their average area was 0.78 μm^2 (n = 63, SD = 3.3×10^{-3} μm^2) and they shared a striking similarity to melanin granules present in some wool and human hair fibres (Kitano et al. 2009).

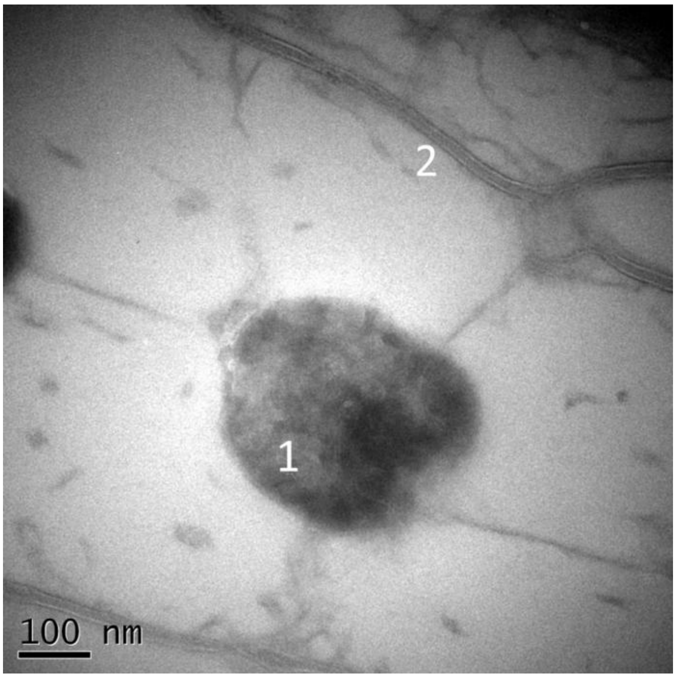


Figure 4-17 - goose down sample highlighting a possible melanin granule
1 – possible melanin granule; 2 – cell membrane complex

The intensely-stained nuclear remnants that had been prevalent in the duck down samples were less apparent in the goose down samples, though they were still present. It is possible that the granules were actually nuclear remnants that had not formed the dendritic shape associated, in wool and in duck down, with these structures. The dendritic nuclear remnants that were present in goose down were more prevalent in the barbules than in the barbs, and granules tended to form near them, particularly in the barbules.

The shape and staining of the granules meant that it seemed highly probably that they were melanin, but because of the limitations of TEM (Hock & McMurdie 1943), such as potential unspecific staining, further analysis was required for their identity to be made certain. This will be carried out in section 5.1.

AFM also highlighted the appearance of the possible melanin granules in goose down (Figure 4-18):

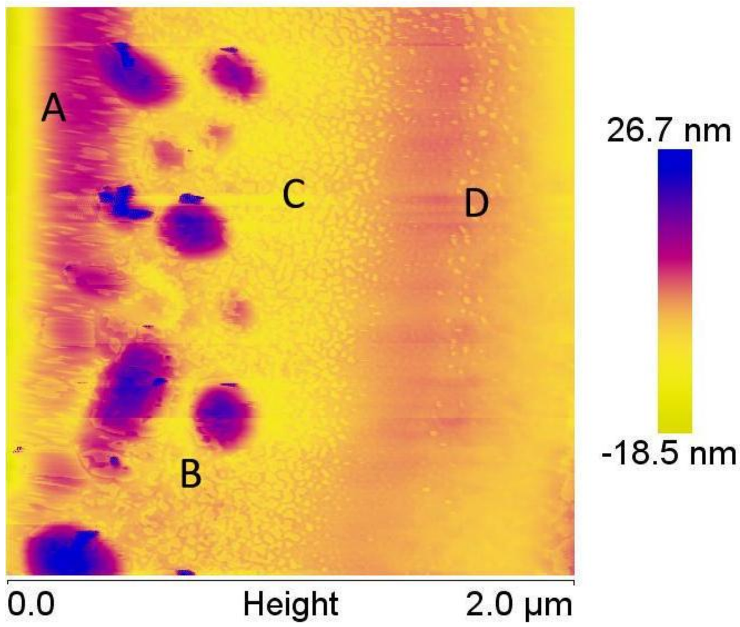


Figure 4-18 – AFM image of part of a goose down barb in resin
A – artefact of preparation, B – possible melanin granules, C – uneven surface, enlarged in Figure 4-19, D - artefact of preparation

Disregarding the raised linear strips at A and D in Figure 4-18 that are a result of sample preparation (imperfections in the cutting of the sample using the ultramicrotome), there are two distinct features present in Figure 4-18. One is the proposed melanin granules at point B, which are of similar shape and approximate diameter (200 nm) to those observed using TEM. The clear difference in height between the granules and the surrounding surface is indicative of possible differences in mechanical properties between the two materials. An image of the granules taken at greater resolution is shown in Figure 4-19:

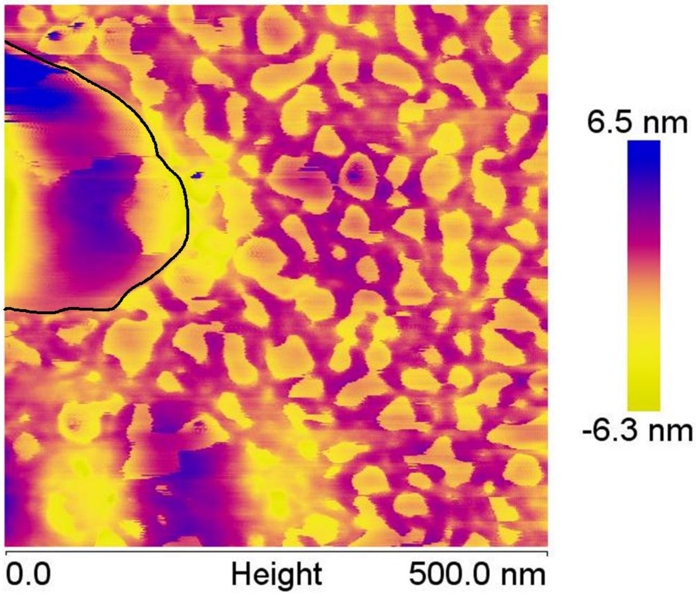


Figure 4-19 – AFM image of a goose down sample in resin displaying the edge of a probable melanin granule (outlined in black) and the surface roughness thought to be indicative of immature macrofibrils

Figure 4-19 shows that, in addition to the proposed melanin granules, the seemingly-uniform material surrounding the granules was actually two-phase, with complex shapes forming channels between raised areas. These areas are not a product of the sample preparation procedure, being too uniform in size and too organically shaped. They are also not mature macrofibrils, as they are too small, when compared to the 200-300 nm diameter structures that were observed using TEM (see Figure 4-20):

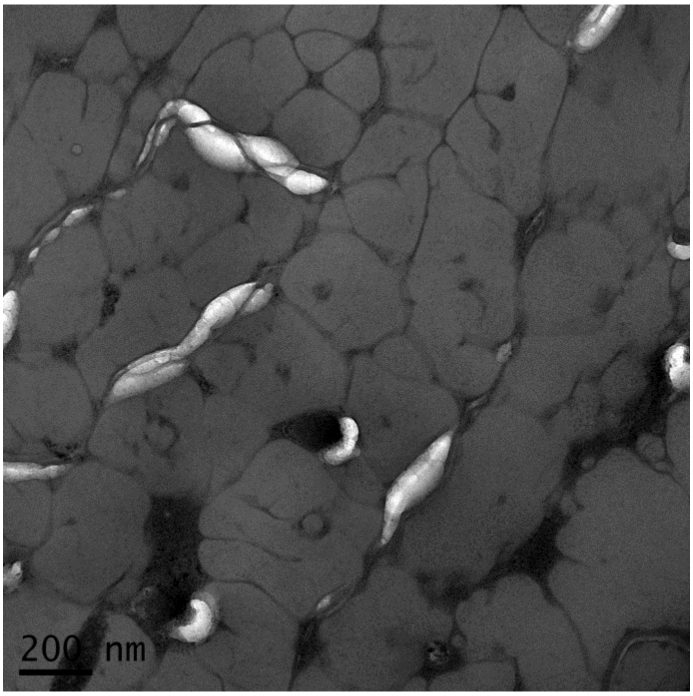


Figure 4-20 - TEM micrograph of a goose down feather cross section
Mature macrofibrils are approximately 200-300 nm in diameter

The complex structures in Figure 4-19 are approximately 25-50 nm in diameter and so are difficult to observe using the present iteration of TEM, but faint structures of similar size could be observed in some TEM images (Figure 4-21). AFM images such as Figure 4-22 show these structures in much greater detail than is possible when using TEM.

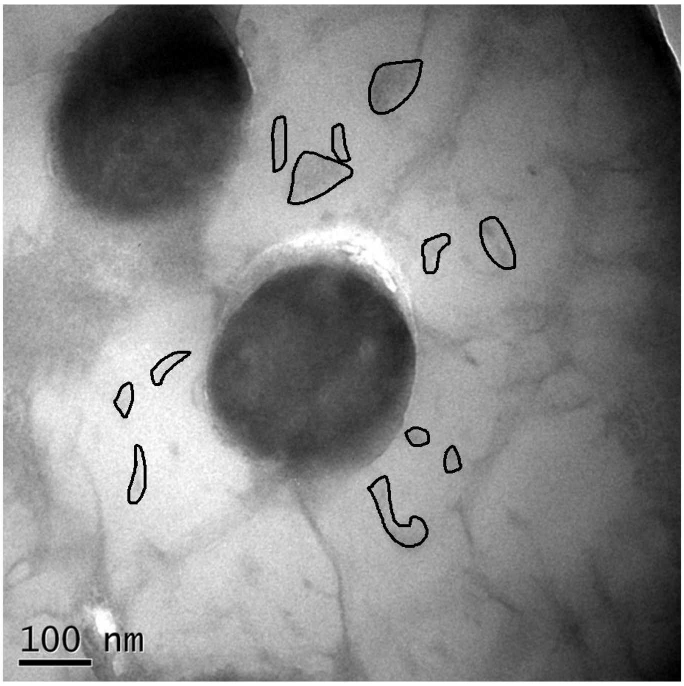


Figure 4-21 - TEM micrograph of goose down feather cross section mounted in resin
Note the small structures (25-50 nm long) that are outlined in black

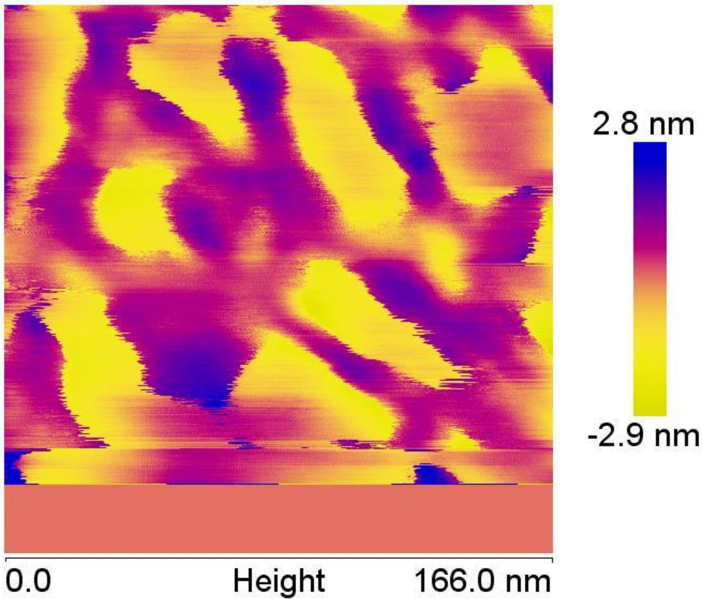


Figure 4-22 – AFM image of a goose down sample in resin highlighting the surface irregularities of the immature macrofibrils in resin

Structures very similar in size and morphology to those in Figure 4-19 and Figure 4-22 have been observed by Filshie and Rogers (1962) in flight feathers. They described them as fibrils in a relatively early stage of development, before coalescing and forming macrofibrils. It is likely that the structures observed here are also premature macrofibrils, as they are too small to be mature macrofibrils or cells, and too large to be microfibrils, which are approximately 3 nm in diameter. The clarity with which AFM has distinguished these structures from the surrounding matrix is notable, and far superior to the resolution achieved by use of TEM either in this work or when carried out by past researchers (Filshie & Rogers 1962). This is due largely to AFM's different mode of operation, being limited less by dyeing and staining procedures which struggle to differentiate between materials with very similar chemistries: instead, as described in section2.2.4, it relies on forces to differentiate between mechanical properties.

In goose down's barbules, nuclear remnants formed long elongated domains and from these, channels of smaller remnants grew that divided macrofibrils between them. This can be seen in Figure 4-23:

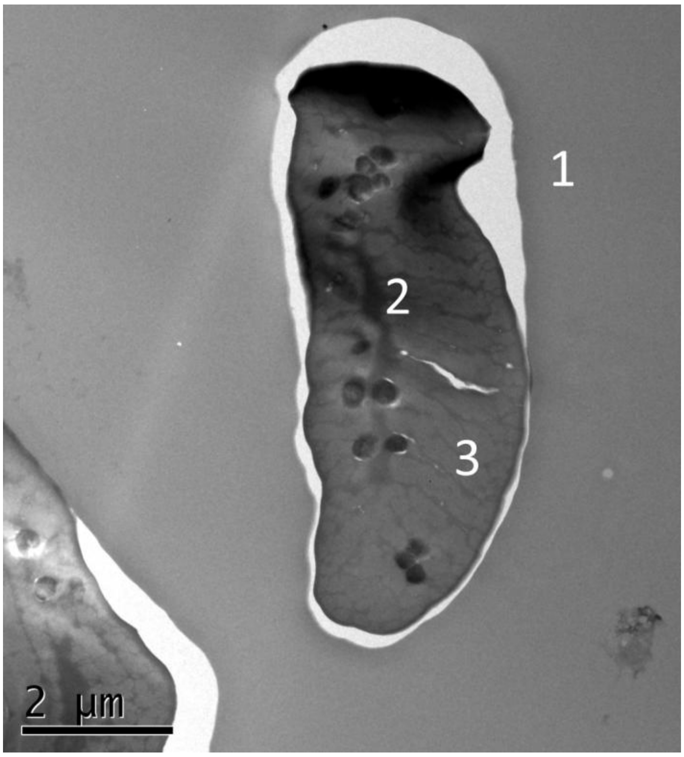


Figure 4-23 – goose down barbule viewed through TEM
1 - the barbule; 2 - nuclear remnants in the centre of the barbule; 3 -elongated nuclear remnants separating macrofibrils

4.2 Analysis of the crystal structure of down feathers using X-ray diffraction

Analysis of down feathers using X-ray diffraction has been, to the author's knowledge, only attempted by one previous research group (Zhang et al. 2011). Despite the difficulties in obtaining structural information from feathers (discussed in section 2.3.3), it has been stated (Schroeder et al. 1955) that they produce a well-defined X-ray diffraction pattern, indicating an ordered material. The first X-ray diffraction studies of feathers were by Astbury and Marwick (1932) and studies have interpreted the X-ray diffraction pattern of feathers as indicating a helical conformation (Briki et al. 2000; Fraser et al. 1972; Cameron et al. 2003). It has been found that the avian keratin pattern has a shorter axial repeat (3.1 Å versus 3.5 Å) than most β-keratin and is more elaborately developed (Mercer 1961).

This X-ray diffraction study is hoped to elucidate structural information from down feathers that was not made clear by previous researchers (Zhang et al. 2011) and to enable direct comparison between goose and duck down. It also serves as an excellent study into the preparation methods required for the accurate study of complex geometries by X-ray diffraction. The impact of water on the diffraction patterns on down was also hoped to be determined. The four methods used to manufacture samples are described in section 2.3.3 and were developed as testing went on in attempts to increase resolution. The methods described may be useful not just to the testing of down feathers but to any fibrous or low-density material of complex geometry. The results from each individual sample preparation method will be treated individually.

4.2.1 Method 1-3

Using Method 1, described in section 2.3.3.1, two obvious peaks were observed in the spectra of both goose and duck down, as shown in Figure 4-24. Using this method indicated that improvements in the sample preparation were needed, however, as the high noise in the spectra made more detailed analyses impractical. In addition, the intensities of the diffraction patterns were not very high due to the small density (relatively low number of atoms causing diffraction) of the rolled down feathers.

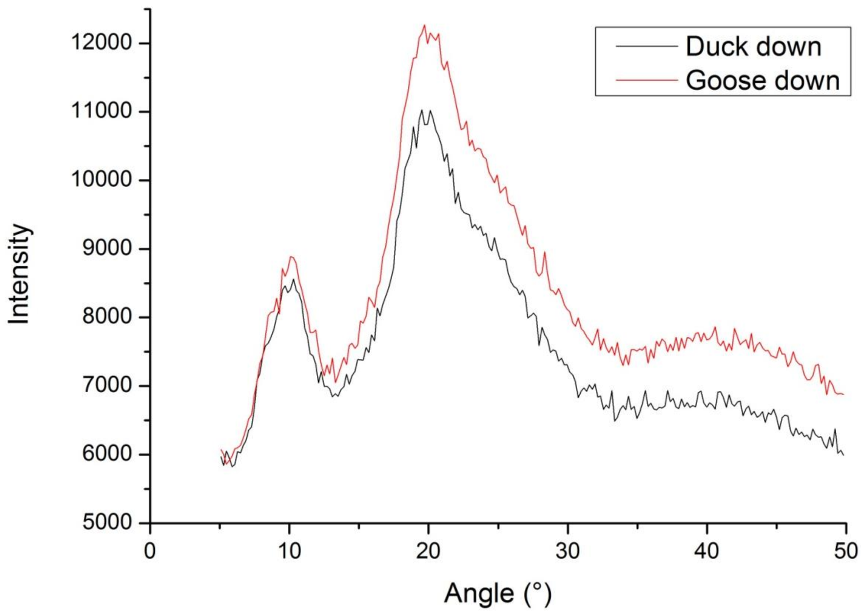


Figure 4-24 – X-ray diffraction patterns of duck and goose down feathers rolled and mounted on a silica crystal

The results from the samples prepared using Methods 2 and 3 offered no greater resolution or intensity than those prepared using Method 1. The ‘powdered’ samples made using Method 2 were too low in density, causing too few X-rays to interact with the down. This was due to the difficulty in cutting the down and creating samples that were thick, uniform, and also closely-packed. Method 3 suffered from the problem of measuring low-angle X-ray diffraction patterns in transmission mode, which tends to result in broad signals from the propagation of beam spread (He & Preckwinkel 2002).

4.2.2 Method 4 – nonwoven samples

This was almost certainly the first time a down-feather-based nonwoven had been made for the purposes of X-ray diffraction analyses. Indeed, it was probably the first time that hydroentangling has been used for the sole purpose of making X-ray diffraction samples from fibres that are otherwise difficult to analyse, and was also one of the first times that down feathers have been used in the manufacture of a nonwoven fabric.

The prepared nonwovens were approximately 2.5 mm thick and mechanically strong enough to be analysed in the X-ray diffractometer. By using hydroentanglement and no chemical binders to secure the down feathers together, no materials that would influence the diffraction pattern were introduced. The prepared samples were analysed using SEM to ensure that the structure of the down had not been damaged by the hydroentangling process and that the hydroentangled samples would be representative of the as-received down feathers. Recorded images are shown in Figure 4-25 and Figure 4-26:

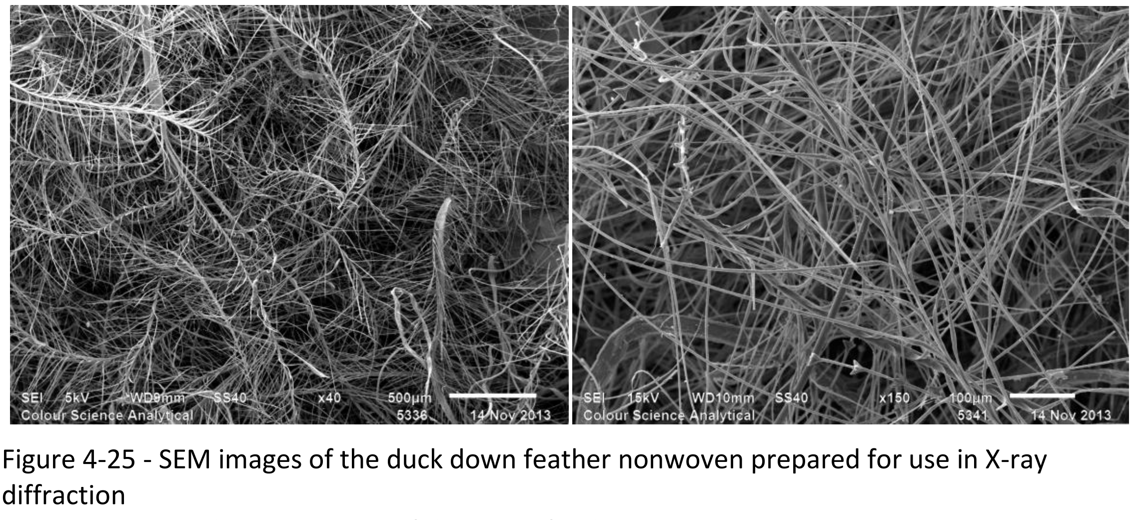


Figure 4-25 - SEM images of the duck down feather nonwoven prepared for use in X-ray diffraction
No damage was observed in any of the down feathers

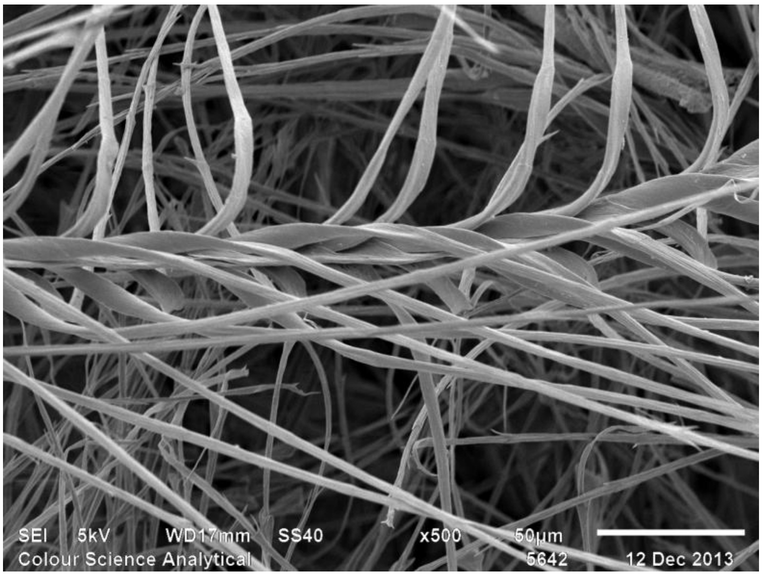


Figure 4-26 - SEM image of the goose down feather nonwoven prepared for use in X-ray diffraction
No damage was observed in any of the down feathers

The SEM analyses showed highly tortuous and cross-linked materials but with no evidence of damaged barbs or barbules.

The nonwoven down samples had a greater density of down feathers than the other samples and were also much flatter, which enabled greater diffraction intensities to be recorded and also for noise to be reduced. Results from both goose and duck down are shown in Figure 4-27:

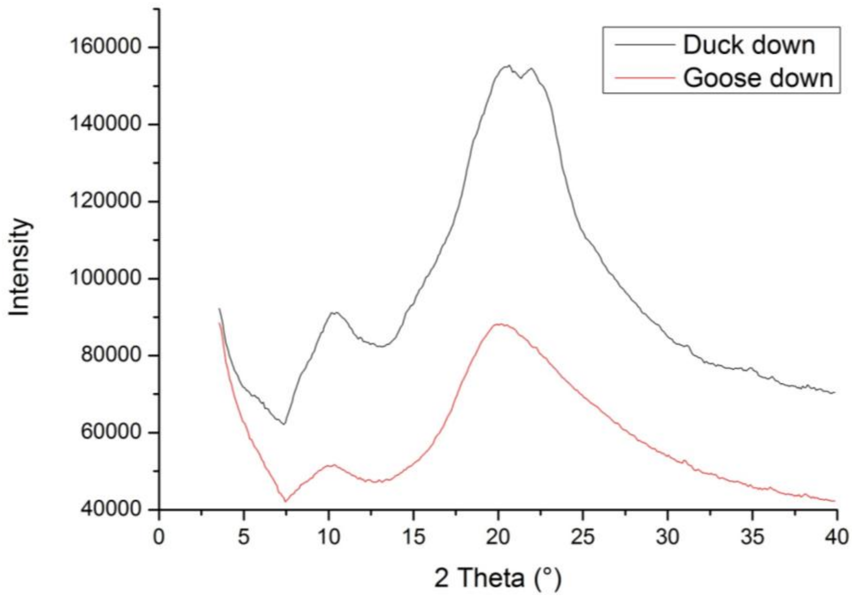


Figure 4-27 – X-ray diffraction patterns of goose and duck down feather nonwovens

The greater intensity of the duck down results in Figure 4-27 was due to differences in sample preparation – a greater density of down feathers as part of the hydroentangled web caused greater X-ray diffraction to occur, so the data from Figure 4-27 is normalised against the maximum intensities in each type of down in Figure 4-28:

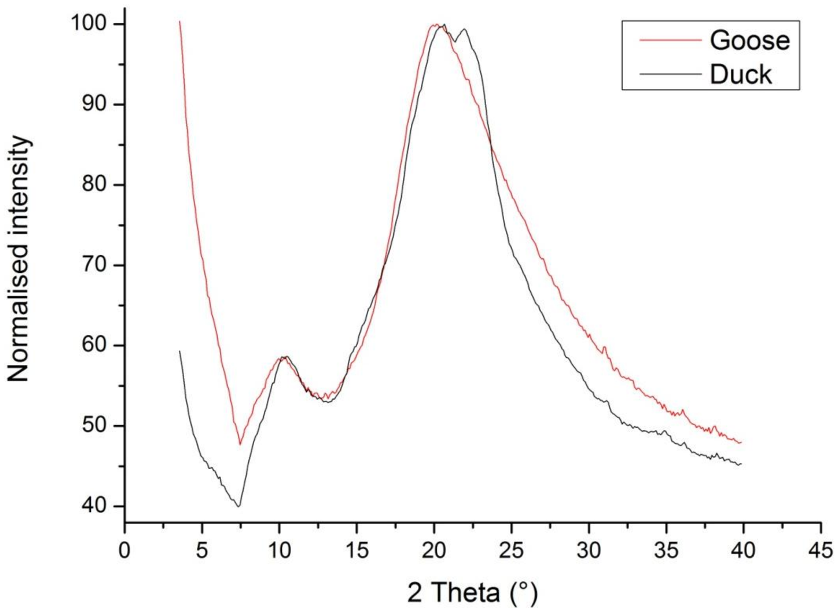


Figure 4-28 – X-ray diffraction patterns of goose and duck down feather nonwovens normalised against the maximum intensities in each respective material

The normalised results in Figure 4-28 show exceptional similarity between the two materials and so despite the differences in morphology between goose and duck down shown earlier in the present chapter, and in Chapter 3, the crystal structure of goose and duck down are very similar. This is representative of the similarity in the keratins that make up these down feathers: the differences between them are macroscopic and therefore larger than which is measurable using X-ray diffraction.

The graphs in Figure 4-28 show maxima at 2θ of 11 and 21 ° which is similar to the results found in wool (maxima at approximately 10 and 21 ° 2θ (Cao & Billows 1999)) and to the results from chicken feathers (maxima at 2θ of 10 and 21 ° (Reddy & Yang 2007)). The peaks were relatively broad, indicating that avian keratin possesses significant amorphous regions and does not possess the defined crystal forms that may occur in minerals or other crystalline materials. This is one factor that makes the determination of a unit cell for feather keratin very difficult; indeed, few models have even been suggested (Reddy & Yang 2007).

In order to decipher the peaks in Figure 4-28 and resolve the features contributing to diffraction, peak-fitting software was used to model the data. The data from the duck down is annotated in Figure 4-29, overleaf. When 3, 4, or 5 peaks were used to model the data the fit was poor, but 6 peaks modelled the data much more accurately.

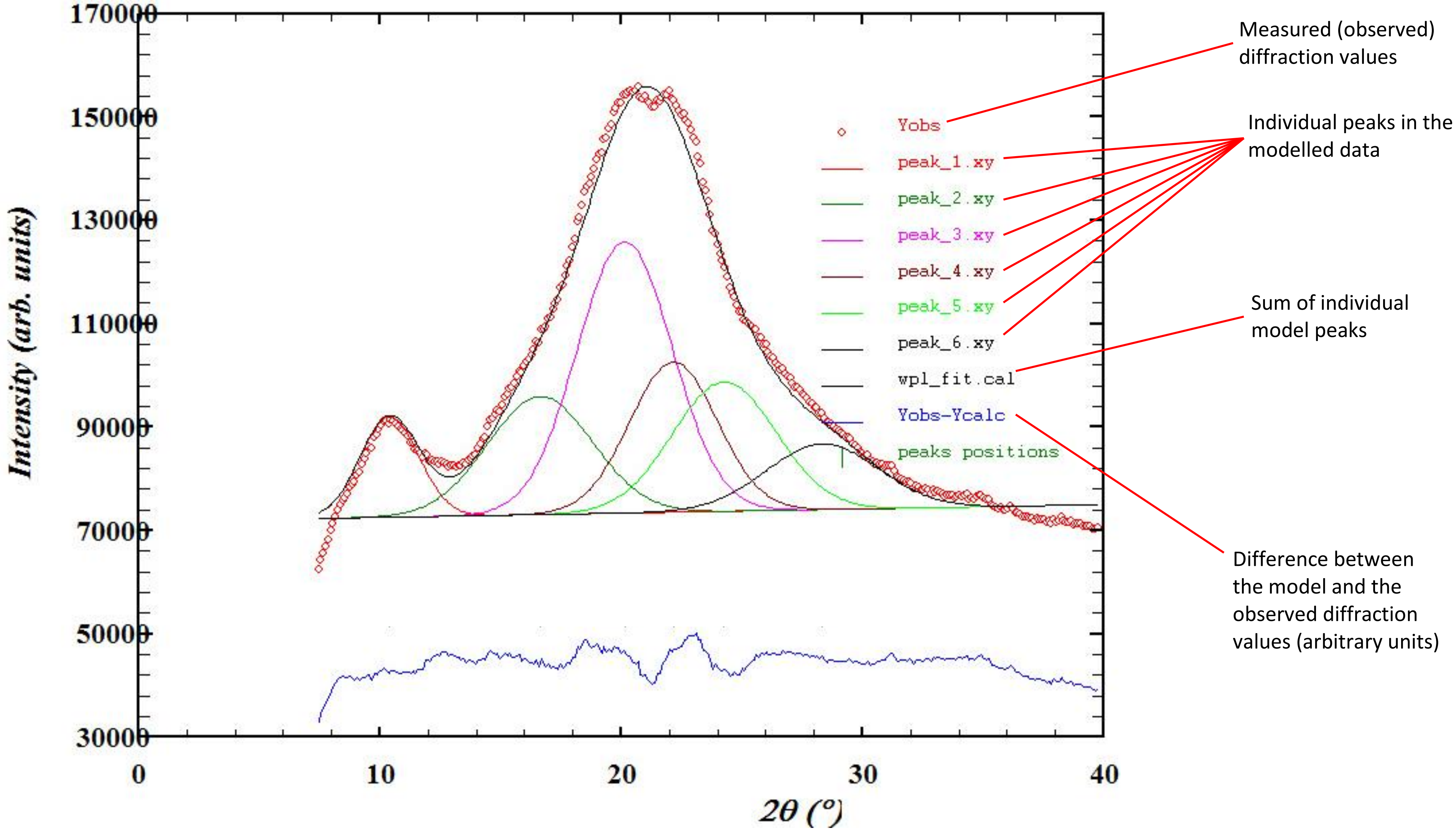


Figure 4-29 - modelling of data from the duck down nonwoven using WinPLOTR from FullProf suite (FullProf Team 2006) with annotations shown

The FullProf model assumed a relatively flat background stretching from 9 to 40 ° 2θ. The 6 peaks in the model are shown in Table 4-1. The diffraction spacings are also shown and were determined using Bragg's Law, Equation 2-1:

Table 4-1 - description of peaks used to model duck down

Peak	Position (2 Theta °))	Intensity (arbitrary units)	Diffraction spacing (Ångströms)	Diffraction spacing (nm)
1	10.40	58909	8.50	0.85
2	16.64	124374	5.32	0.53
3	20.15	259127	4.40	0.44
4	22.16	131957	4.01	0.40
5	24.27	134466	3.66	0.37
6	28.33	73663	3.15	0.31

Of the modelled peaks shown in Table 4-1, Peak 3 was most intense, and equated to a diffraction spacing of 4.40 Å. This same peak was observed as a reflection in the barbs of chicken feathers (4.37 Å (Reddy & Yang 2007; Pauling & Corey 1951)). Peak 1 also correlates with values determined in chicken feathers (8.50 Å here, 8.56 Å in chicken feathers (Reddy & Yang 2007)). Peak 6, that correlates with a diffraction spacing of 3.15 Å, correlates extremely well with the 3.1 Å diffraction spacing obtained by Astbury and Marwick and later by Schor and Krimm (Astbury & Marwick 1932; R. Schor & Krimm 1961) and it is this diffraction spacing that is indicative of the helical crystallites of avian β-keratin (Fraser & Parry 2011) and differentiates it from the 3.33 Å spacing of stretched mammalian keratin.

Though there has been relatively little recent work carried out on the X-ray diffraction patterns of feathers (Reddy & Yang 2007), the model now accepted by most academics is that of a two-strand rope of β-sheet crystallites that form a high modulus framework (Fraser & MacRae 1980) where a quarter to a third of the protein is in β conformation (Fraser et al. 1972; Fraser et al. 1971). It is highly likely that down shares this conformation, which is shown in Figure 4-30 (Fraser & Parry 2008):

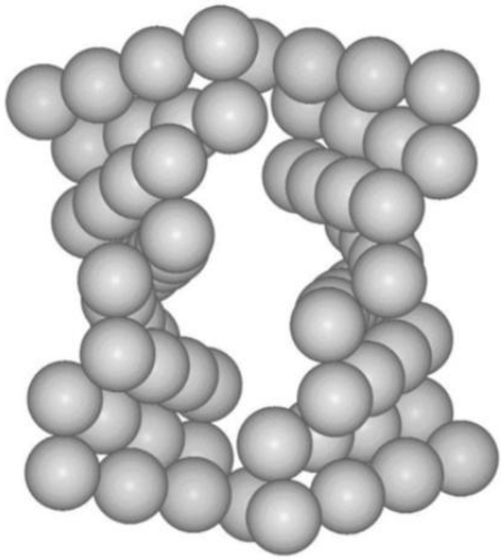


Figure 4-30 - the repeating unit in a feather keratin microfibril
Each sphere represents a protein residue. A 4-chain by 8-residue segment forms a beta-pleated sheet, and two of these segments twist around one another in a right-handed manner, and are arranged on a left-handed helix with 4 pairs of molecules per turn (Fraser & Parry 2008)

The high degree of homology found here between two quite disparate birds from separate orders corroborates the findings of Marshall et al. (1991), who reviewed considerable similarities between the keratins of different mammals. Feathers may offer a unique diffraction pattern (Fraser & Parry 2008), but that same pattern is replicated across many different avian species.

The only known previous X-ray diffraction study of down feathers was by Zhang et al. (2011), but their results correlate poorly with the results here and also with the results from previous studies (Reddy & Yang 2007) of feathers. From their results, they also determined the crystallinity of goose and duck down, but this is fraught with potential errors, and as such this analysis has not been carried out here. When determining crystallinity it is essential that both the crystalline and amorphous contents of the diffraction patterns are known (Cao & Billows 1999), but this is very difficult in a material such as down where the peaks are broad as it is not certain whether the broadening is due to sample preparation, differences in equatorial and meridional reflections, or discrepancies in crystal sizes, and as such it is not possible to separate the amorphous and crystalline contents of these materials. The complexity of determining the degree of crystallinity in keratin is reflected in the relatively few reported measurements of wool's crystallinity. Those that have been published were reviewed by Cao et al. (1999).

Attempts to determine the effects of water on the X-ray diffraction patterns of down were unsuccessful: the X-ray diffraction pattern of water overlapped with that of down and masked the relevant signals (see Figure 4-31). Thus, it was not possible to determine whether the swelling of down in water affected its X-ray diffraction pattern.

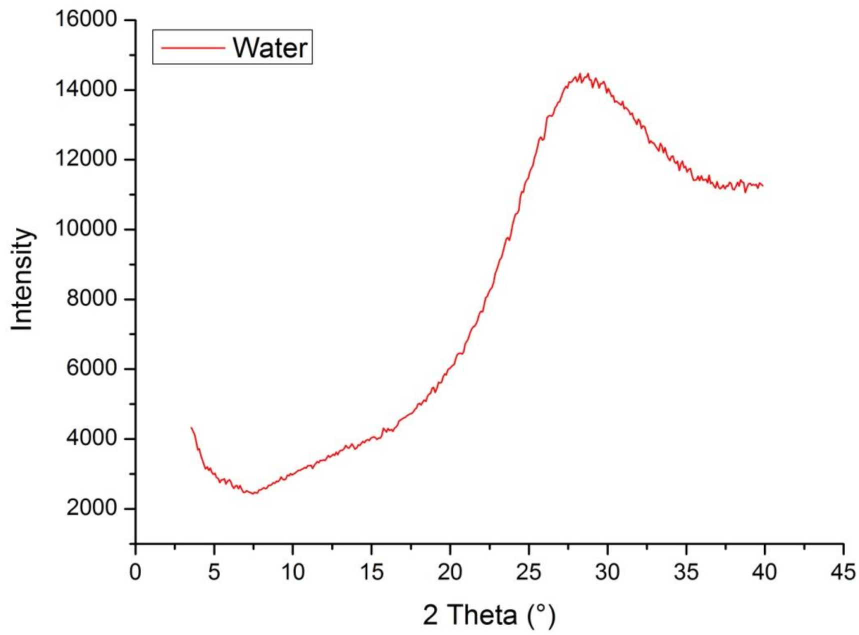


Figure 4-31 - the X-ray diffraction pattern of liquid water captured using a spinner mount
The close similarity between the spectra of goose and duck down and water made determination of the effect of water on down's X-ray diffraction pattern very difficult

A plot containing normalised data from dry duck down, liquid water, and wet duck down, is shown in Figure 4-32, but the signals from the wet down and from the water were too alike to justify further analysis.

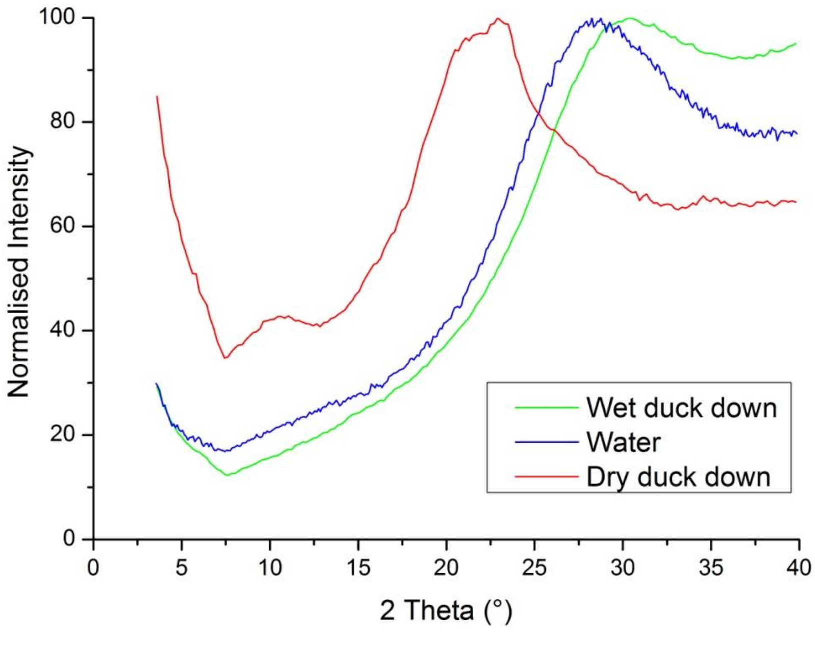


Figure 4-32 - X-ray diffraction data for liquid water, wet duck down, and dry duck down normalised against their maximum intensities

The elucidation of further information from dry goose and duck down, such as changes in diffraction pattern along the length of an individual barb, would require extremely high X-ray flux to generate sufficient scattering at such high resolution and was outside of the scope of this work. Future work could use synchrotron radiation to overcome the greatest limitation of normal X-ray diffractometers (their low proton flux and spatial resolution (Margaritondo 1988)). Use of a synchrotron has, for example, allowed the structure of hair to be studied in great detail (Miller & Dumas 2006), revealing features that had previously been very difficult to detect (Briki et al. 2000).

4.3 Analysis of the thermal properties of down feathers by using differential scanning calorimetry

Previous analysis of down feathers using DSC (Ancheng et al. 1990) showed they have two endothermic peaks when heated to 300 °C: one peak at 80 °C and the other at approximately 250 °C. The peaks were very similar in goose down, duck down, and the flight feathers from both birds (Ancheng et al. 1990), implying that the composition of each of the feathers was very similar. However, to the author's knowledge, no other DSC results have been reported, which is very different when compared to the vast literature source available on the thermal transitions of wool (Menefee & Yee 1965). Here, analysis will concentrate on the possible effects of thermal transitions on down's mechanical and sorption properties. The experimental method used for DSC analyses is presented in section 2.3.4.

The DSC traces from the goose and duck down samples are shown in Figure 4-33 and a summary of the peak data from both samples is shown in Table 4-2. It was found that goose and duck down both underwent two transitions when heated from 30 °C to 350 °C: a broad one at approximately 45-140 °C, and a much sharper one at approximately 230-250 °C.

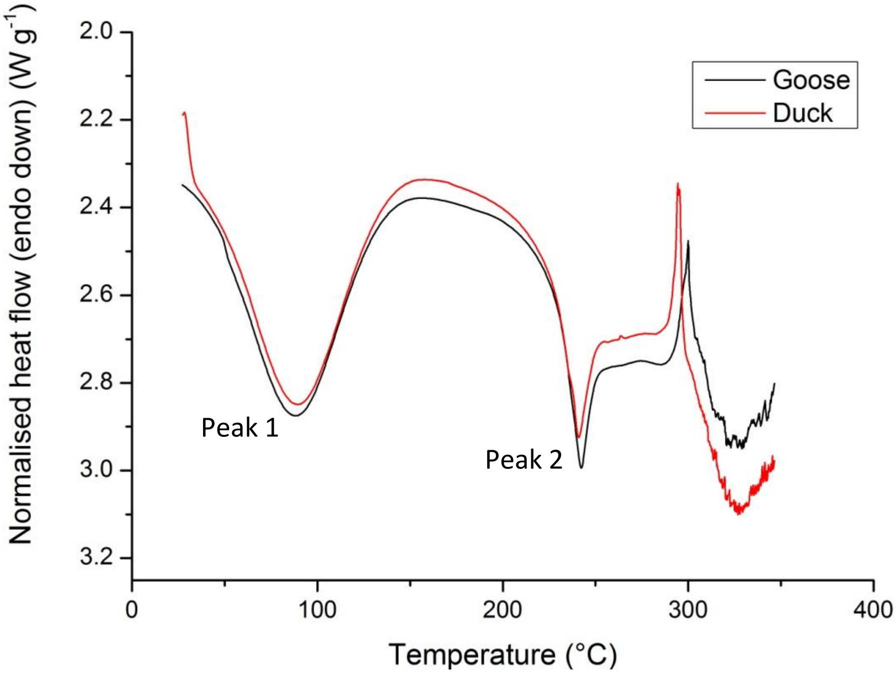


Figure 4-33 - DSC curves for goose and duck down samples heated at a rate of 10 °C min^{-1}

Table 4-2 - summary of the peak data from the DSC curves of goose and duck down

(ΔH is the change in enthalpy of the sample over the course of a reaction or transition)

Sample	Peak 1 onset T (°C)	Peak 1 ΔH (J g^{-1})	Peak 1 (°C)	Peak 2 onset T (°C)	Peak 2 ΔH (J g^{-1})	Peak 2 (°C)
Goose	44.60	161.67	87.95	227.72	123.12	242.27
Duck	46.14	163.27	89.43	228.65	135.30	240.92

The overriding similarity of goose and duck down was the most apparent characteristic of their datasets: each of their peaks was within 2 °C of one another and the enthalpy change of their first transition was extremely similar. Though the measured enthalpies of their second transition varied by approximately 10 %, this may be more due to a discrepancy in the peak-fitting software's differentiation of the peaks from the baseline of each sample. Because of the similarities between the two results, they will be analysed concurrently.

Peak 1, as labelled in Figure 4-33 and Table 4-2, occurs due to the evaporation of water from the down. Water is thought to bind to keratin in three forms (Speakman 1944), and because of hydrogen bonding between water molecules and the keratin structure, some of the water requires the temperature to exceed 100 °C for it to evaporate. Thus, the steady release of water from the sample is a result of activation energy limitations, not thermal lag. The activation energy of water undergoing hydrogen bonding to wool keratin has been estimated at 25.1 kJ mol^{-1} (Menefee & Yee 1965) and it is very likely to be comparable in down, as complete drying of wool requires temperatures of 150 °C (Menefee & Yee 1965), and the same temperatures were reached here before complete drying took place.

Peak 2, the sharp endotherm at 240-242 °C is also found in wool, at 245 °C (Menefee & Yee 1965), in hair at 233 °C (Milczarek et al. 1992), in turkey feathers (Idris et al. 2013), and in poultry feathers at 235 °C (Schmidt & Line 1996). This peak is indicative either of melting (Menefee & Yee 1965) or denaturation (Haly & Snaith 1970) of crystalline phases. The sharpness of this transition indicates a very ordered phase with low polydispersity (Schmidt & Jayasundera 2004) but because keratin is never completely crystalline it is not possible to calculate the percentage crystallinity of these down feather samples, as there are no absolute values to work from. However, direct comparisons can be made: the greater enthalpy of transition 2 in duck down implies that they may be more crystalline than goose down, though this may be a product of inaccuracies in the peak-fitting software. Further analysis would be required to determine this with greater certainty.

One measurement that has not been possible to make from the present DSC spectra is that of glass transition temperature, T_g. The T_g is the point at which amorphous regions in a material turn from a glassy to a rubbery state. The T_g of wool has previously been measured using both mechanical methods and DSC (Wortmann et al. 2006), but it is very difficult to quantify accurately because it is so affected by the presence of water (Phillips 1985), the thermal history of the sample (Huson 1991), and because of the very small change in keratin's heat capacity that occur with a change in T_g (typically 0.1 J g^{-1} K^{-1} (Wortmann et al. 2006)).

Kure et al. (Kure et al. 1997) have highlighted the effect that moisture content has on wool's T_g, measuring the T_g of dry wool as approximately 170 °C, and the T_g of wet wool as approximately -10 °C. This has been attributed to the effect of water as a plasticiser in keratin's amorphous matrix (Milczarek et al. 1992) reducing the internal viscosity between molecules (Lynch & Feughelman 1970). It has even been suggested (Phillips 1985) that the wearer of a woollen jumper sweating profusely could cause wool to exceed its T_g because if moisture content in the wool reached 22 % then the T_g of the fibre would be approximately 35 °C (the approximate skin temperature of the wearer (Phillips 1985)). The great structural and chemical similarity between down and wool indicates that their T_gs are likely to be very similar, and so the T_g of saturated down at room temperature may also be exceeded. This is an important factor that has, to the author's knowledge, previously been overlooked. It shows that the reduced tensile modulus of wet down (Bonser & Farrent 2001) may be due in part to the T_g being exceeded, and may indicate why down is thought to become vulnerable to potential mechanical damage when saturated, as like wool, it loses dimensional stability (Wortmann et al. 1984; Kure et al. 1997). The effect of T_g may also in part explain why tumble drying, soaking in water, or steam treatment of down increases its fill power (IDFL 2010b; IDFL 2010g; IDFL 2011; Fuller 2012) because 'ageing' that occurs in wool, and almost certainly occurs in down, leads to hydrogen bonding between fibres and can decrease their elasticity (Huson 1991). Ageing may reduce down's ability to recover from compressional stresses and by wetting or heating the down, the T_g is exceeded and deageing occurs, breaking the hydrogen bonds that have formed in the down over time and thus allowing it to move freely and loft to its full extent. This would lead to a subsequent increase in fill power.

4.4 Conclusions

TEM, SEM, AFM, X-ray diffraction and DSC have been used to study the fine structure of goose and duck down. To the author's knowledge this is the most comprehensive analysis of down's microstructure presented to date.

1) Duck and goose downs' cross-sectional compositions were found to be relatively similar to one another and closely related, also, to wool and hair.
2) Goose and duck down samples both possessed a cuticle, which had previously been assumed to be unique to wool and hair, suggesting that down may be thought of as a combination of a feather's fractal morphology with some of wool's functionality.
3) The down samples possessed cortices containing the principle components found in wool: orthocortical and paracortical cells, macrofibrils, nuclear remnants, and cell membrane complexes.
4) The avian microfibrils were too small to be distinguished with certainty using this iteration of TEM but may have appeared as faint structures in some images.
5) AFM identified immature macrofibrils that were very difficult to observe using TEM.
6) While the barbules of goose and duck down were similar in shape, goose down's barbs were approximately triangular in cross section, expected to impart them with superior bending resistance to the more rounded cross sections of duck down barbs.
7) Goose down's barbs had a possibly-hollow cross section that will be further analysed in Chapter 5. If hollow, this will influence both thermal and mechanical properties.
8) Osmiophilic granules were observed in goose down but not in duck down. The shapes of these structures suggest that they are melanin but this will be further investigated in Chapter 5.
9) X-ray diffraction studies of down presented great challenges in sample preparation, but these difficulties were overcome by airlaying and hydroentangling down to make

flat nonwoven fabrics. These samples are almost certainly the first down fabrics made in this manner and for this purpose.

10) The goose and duck down feathers produced X-ray diffraction patterns that closely resembled the results from other feathers and they were modelled accurately by six diffraction peaks, including the characteristic peak from a diffraction spacing of 3.15 Å that is indicative of avian keratin.
11) The X-ray diffraction study provided the clearest diffraction patterns known from both goose and duck down, and demonstrated an excellent manner of preparing X-ray diffraction samples from complex-shaped fibres.
12) DSC studies showed the similarities between goose and duck down and wool.
13) The T_g of down could not be measured, but extrapolating the T_g from literature results of wool led to the proposition that the T_g of down may be exceeded in hot conditions or if it is thoroughly wetted. This may help explain why tumble drying or steaming, that cause down to exceed its T_g, or water washing, which lowers down's T_g enough that room temperature exceeds it, can increase fill power.

This work is expected to aid the determination of structure-property relationships in subsequent chapters and it helps fill the significant knowledge gap regarding the microstructure of down.

Chapter 5. The chemical composition and properties of goose and duck down feathers

The structural properties of goose and duck down were characterised in Chapters 3 and 4 but their chemical composition and properties have not yet been explored. Analysis using energy-dispersive X-ray (EDX) and Fourier transform infrared (FTIR) spectroscopies will provide such information: EDX providing details regarding internal structure, to identify the granules in goose down, and to determine whether the goose down barbs are hollow; FTIR spectroscopy to inform regarding surface chemistry.

5.1 Elemental analysis of the chemical composition of down feathers using energy-dispersive X-ray (EDX) spectroscopy techniques

It was found in section 4.1.2.2 that goose down barbs had a possible hollow cross section, and both barbs and barbules contained granules with a shape suggesting that they were made of melanin. Here, EDX analyses were carried out to aid identification of the granules and the void structure in the centre of the barbs. In addition, elemental mapping was carried out on the goose and duck down feathers to determine their elemental concentrations.

The techniques used in EDX analyses are described in section 2.2.3.3. Three goose down cross sections were analysed and one duck down sample.

5.1.1 Identification of the central structure in goose down barbs

The same granules and void/medulla that had been seen in Figure 4-10 to Figure 4-14 in the goose down samples were observed in the EDX analyses (Figure 5-1). The goose down barbs' cross sections were analysed using EDX at the locations labelled EDX1 (the middle of the barb) and EDX2 (the resin surrounding the barb) in Figure 5-1. The elemental compositions in these two locations are shown in Figure 5-2.

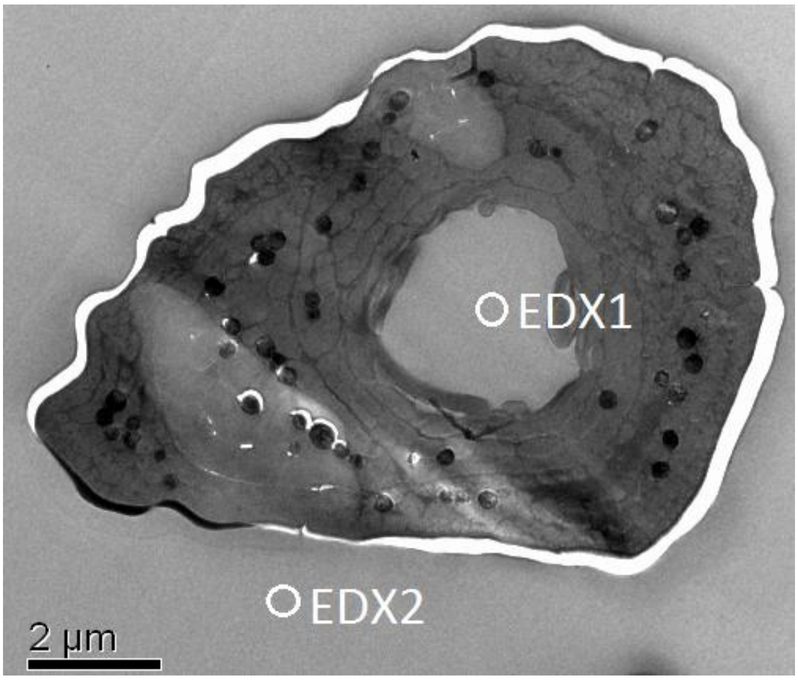


Figure 5-1 - TEM image showing location of the EDX analyses in the centre of the barb (EDX1) and in the surrounding resin (EDX2)

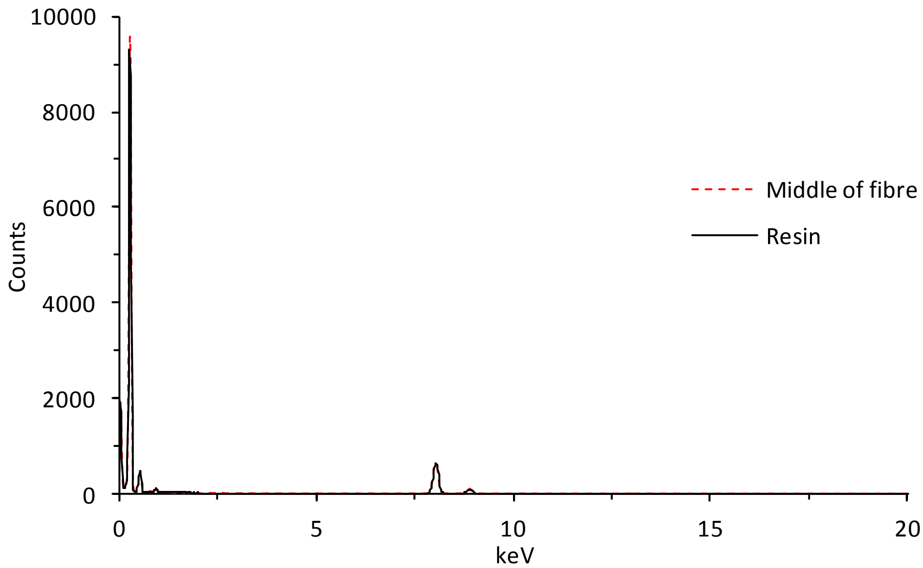


Figure 5-2 – EDX trace of the goose down barb shown in Figure 5-1 (of the resin and the middle of the goose down barb)
Counts refers to the number of detections of certain energy transitions, measured in keV, which are characteristic of different elements. Counts represent the relative numbers of each type of atom and their ability to scatter electrons

Figure 5-2 shows an identical signal from the middle of the goose down barb and from the surrounding resin, indicating the same elemental composition in each area. This was observed in each of the samples and signifies that at all the points of sectioning, the goose down barbs were hollow, showing a strong probability that the barbs are hollow all along their length, despite the barbules being solid.

The hollow cross section in goose down is probably the most striking difference between it and duck down. However, despite all the samples here displaying this pattern, there is no certainty that the hollow cross section occurs in all goose down feathers. Indeed, the hollow medulla in wool does not appear in all wool fibres. Thus, one must be cautious when basing conclusions on a dataset that has been drawn from one type of goose down: in a natural product it would be very bold to claim that all goose down is hollow and all duck down is solid. It may be that, like the medulla in wool, the hollow cross section of goose down is related to diameter, and only occurs when a threshold diameter is reached to reduce mass and subsequent bending moments.

The impact of a hollow and approximately-triangular cross section on the performance of goose down feathers is two-fold. Firstly, the hollow shape gives high bending resistance with economy in weight (Galileo 1914) while the approximately-triangular cross section is thought to reduce the chance of buckling, which is a problem in some hollow cylinders (Fraser & MacRae 1980). Primary flight feathers are often hollow (Purslow & Vincent 1978) and overcome buckling by a utilising a cross section akin to a box girder (Fraser & MacRae 1980). The second influence that the hollow cross section has on down's performance relates to thermal properties: hollow structures trap a greater volume of air and so provide a greater thermal resistance. This is exemplified in hollow synthetic fibres and by other natural hollow fibres such as polar bear fur (Koon 1998). The combined effects of potentially greater thermal and bending resistance properties in goose down may be a factor in its reputation (Bedard et al. 2008; Jacob et al. 2011; IDFL 2010a) as a superior insulating material to duck down.

5.1.2 The elemental composition of goose down barbs

The elemental composition of the cortex of goose down feathers has not been reported previously, and the granules that had been observed in section 4.1.2.1 were of unknown chemical composition. By using EDX, these points would be rectified. The EDX elemental mapping was performed on the same cross sections of the goose down barbs that were used in section 4.1.2.1 and 5.1.1, with the TEM in scanning mode (STEM, scanning transmission electron microscopy) and using ADF (annular dark-field) imaging. Use of STEM allows for much easier conversion between imaging techniques and analytical techniques (Williams & Carter 2009), and use of ADF increases signal strength and contrast. Using these techniques and producing elemental maps is time consuming but provides excellent resolution and is sensitive to atomic number (Crewe & Nellist 2009; Akita et al. 2005) so can clearly highlight differences in chemical composition across a sample. The composite ADF-STEM image of all chemical compositions is shown in Figure 5-3:

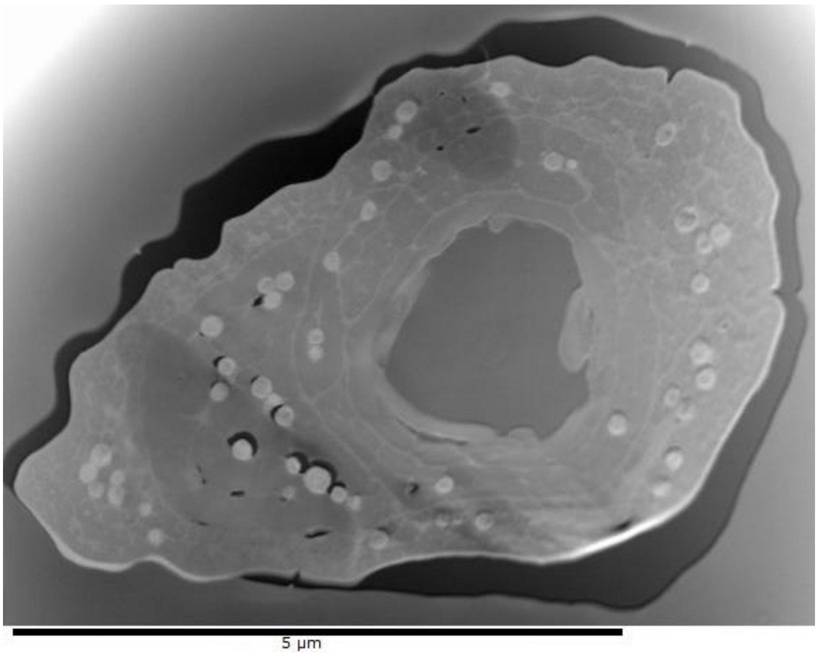


Figure 5-3 - ADF-STEM image of a goose down barb cross section
Lighter areas indicate a higher concentration of electron-scattering elements

The individual features of the cuticle, orthocortex, paracortex, hollow cross-section and the granules were shown very clearly in Figure 5-3 and confirmed the presence of the structures that were identified in Figure 4-12 using conventional TEM. The concentrations of individual chemical elements in each of the component areas of goose down were also mapped using ADF-EDX. In particular, the mapping of sulfur and oxygen were made, which were useful in characterising the barbs. These analyses are shown in Figure 5-4:

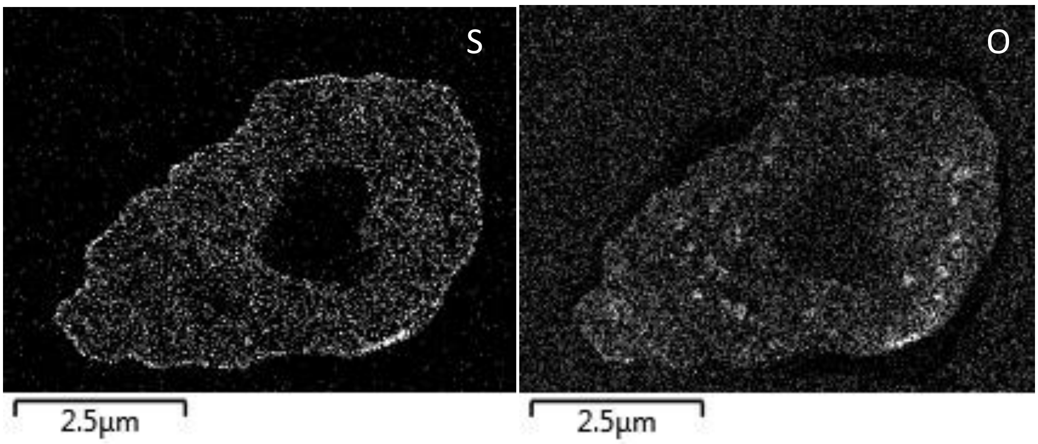


Figure 5-4 – ADF-STEM elemental mappings produced using EDX of sulfur (left) and oxygen (right) concentrations in goose down
Lighter areas indicate a higher concentration of the respective element

Figure 5-4 shows higher concentrations of sulfur and oxygen in lighter colours, and the strong signal of sulfur around the border of the down cross section confirms the identity of the cuticle as, just as in wool or hair (Bradbury & Leytt 1972; Imai 2011; Jones et al. 1990; Carr et al. 1986), it is a thin region of high sulfur content surrounding the fibre. The high oxygen content in the granules bolsters the belief that they are melanin (Imai 2011).

EDX analyses of specific locations in the cross sections were carried out to confirm the findings of the ADF-STEM analyses. The elemental compositions of the cuticle and cortex were analysed at the scanning sites shown in Figure 5-5 (EDX1 for the cuticle; EDX2 for the cortex). The results from these EDX analyses are displayed in Figure 5-6.

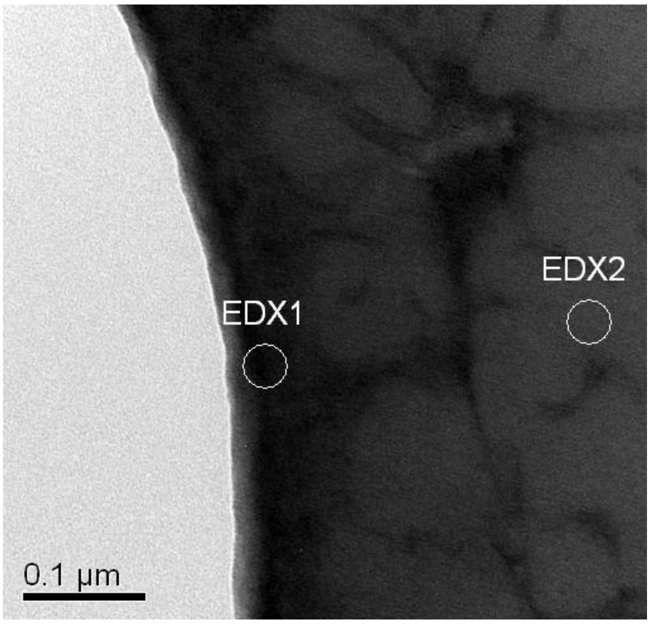


Figure 5-5 – TEM image of a goose down cross section indicating the location of EDX analyses
EDX1 is the cuticle, EDX2 is the cortex

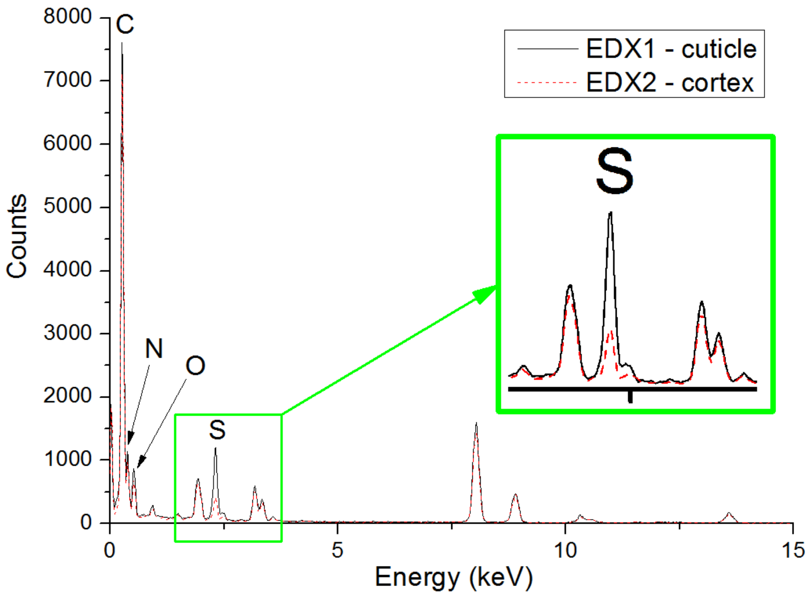


Figure 5-6 – results of the elemental analysis of a goose down barb carried out at EDX1 (the cuticle) and EDX2 (the cortex)
The highlighted area indicates the greater concentration of sulfur in the cuticle than in the cortex. The signals not labelled here are identified in Figure 5-8

In Figure 5-6 the enlarged area of the elemental mapping curve shows that while the ratio of carbon, nitrogen, and oxygen atoms are very similar (C: N: O = 78.2: 13.8: 6.1 % for the cuticle and C: N: O = 79.0: 14.0: 5.6 % for the cortex), there was a large difference in concentration of sulfur (2.0 % in cuticle versus 0.8 % in the cortex). This corresponds with a significantly higher proportion of sulphur-containing amino acids in the cuticle's keratin proteins than in the cortex. This difference is also seen in other keratins (Bradbury & Leytt 1972; Imai 2011; Jones et al. 1990; Carr et al. 1986) and reflects the cuticle's purpose as a protective layer. The sulfurous amino acid cysteine, in particular, is integral to the mechanical structure of keratins by forming disulfide bonds between keratin chains (Jones et al. 2006) and provide keratins with their relatively high wet strength and insolubility (Wilks 2001).

Hydrolysis of cysteine cross-links is thought to reduce the stress resistance of feathers and so, as feathers are wetted, their water resistance will decrease as the vulnerable sulfurous cross-links react (Wortmann & Zahn 1994). The higher sulfur concentration in the cuticle increases its hydrophobicity and resistance to wetting, but once the cuticle has been saturated by water, further penetration of water into the feather will be rapid. This is an indication of why down feathers are sometimes regarded as good insulating materials in moderately-wet weather, but deteriorate quickly if allowed to get very wet.

5.1.3 Identification of the granules in the goose down barb cross sections

The identity of the granules in goose down was uncertain following TEM analysis so EDX was also used to analyse them. Figure 5-7 shows the sites of the EDX analyses performed in the cross section of the goose down barbs. The elemental composition from the EDX analysis is shown in Figure 5-8, and the percentage composition of the major elements in goose down is summarised in Table 5-1.

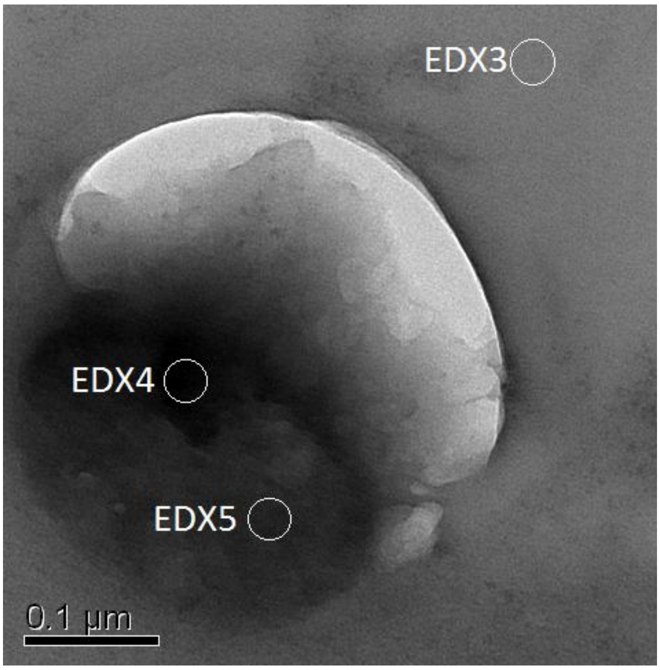


Figure 5-7 – TEM image of a goose down cross section indicating the location of EDX analyses performed on the cross section
EDX3 is the cortex; EDX4 and EDX5 were taken of the granule

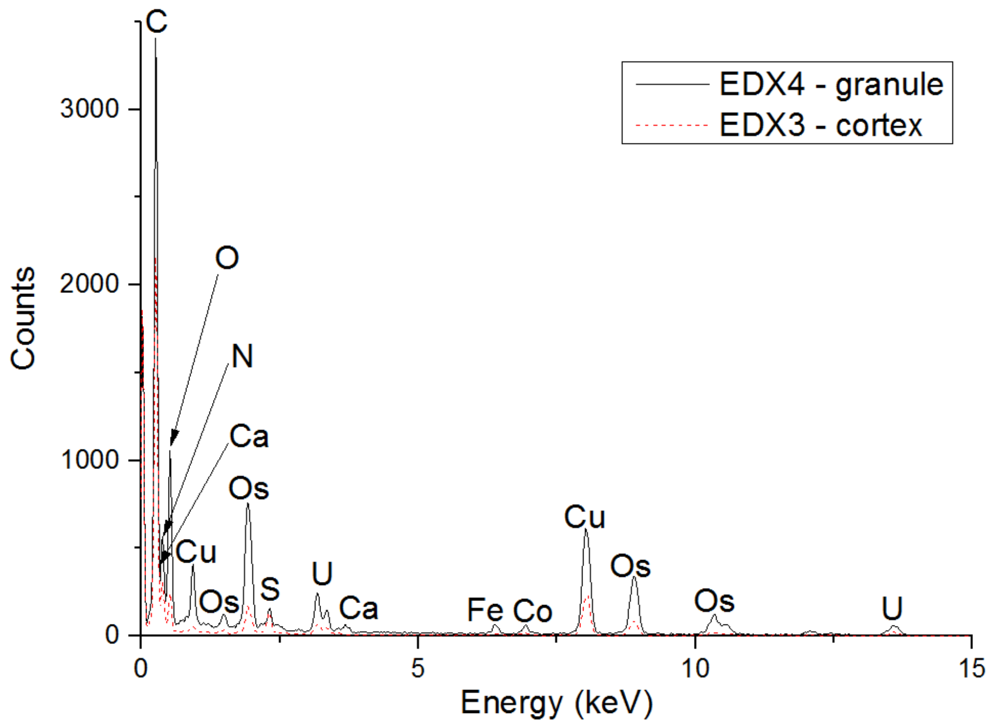


Figure 5-8 – results from EDX measurements taken at two different parts of a goose down cross section
EDX3 is of the cortex; EDX4 is of the granule. The granule shows a much stronger signal than the cortex, which confirms its large uptake of staining agents. The copper artifactual peaks are derived from components of the microscope (Carr et al. 1986). Osmium and uranium peaks are derived from fixative and contrast agents (osmium tetroxide and uranyl acetate, respectively). Data from EDX5 is not shown for the purpose of clarity

Table 5-1 – percentage abundance of elements in goose down barb cross sections (n = 3 for melanin samples and cortex; 2 for cuticle)

	Atomic percentage of element (%)					
	Carbon (C)		**Nitrogen (N)**		**Oxygen (O)**	
Feature	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Granule	72.27	1.07	11.51	2.65	15.72	1.80
Cuticle	78.15	0.25	13.75	0.00	6.07	0.73
Cortex	78.94	1.65	14.00	0.73	5.62	0.69
	Atomic percentage of element (%)					
	Sulfur (S)		**Magnesium (Mg)**		**Potassium (K)**	
Feature	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Granule	0.50	0.15	0.02	0.03	0.57	0.48
Cuticle	2.03	0.47	0.01	0.00	0.27	0.00
Cortex	0.83	0.08	0.03	0.03	0.42	0.03

As shown in Table 5-1, the ratio of carbon, nitrogen, oxygen and sulfur atoms in the granules (C: N: O: S = 72.3: 11.5: 15.7: 0.5) were different to those in the cuticle and cortex (C: N: O: S = 78.2: 13.8: 6.1: 2.03 for cuticle and C: N: O: S = 79.0: 14.0: 5.6: 0.83 for cortex). The quantity of oxygen in the granules (15.7 %) was approximately 2.5 times that in the cortex and cuticle (5.6-6.1 %), and this high concentration has previously been used to positively identify melanin granules (Imai 2011). Melanin granules also have a propensity to uptake metals (Imai 2011) and the high concentrations of potassium (0.57 % in granule, 0.27 and 0.42 % in cuticle and cortex, respectively) in the granules here also suggest that they are melanin. The distinctive shape of the granules (Filshie & Rogers 1962), their high oxygen concentration (Imai 2011), and propensity to uptake metals (Imai 2011), confirms that the granules must be made of melanin, rather than the keratinous proteins of the cortex.

Melanin is derived from the amino acid tyrosine (Kitano et al. 2009) and provides keratinous materials with their colour (Feughelman 1997). It has also been suggested that it may act as a toughening agent (increasing hardness by as much as 39 % (Bonser 1995)) and it may affect the breaking strength of feathers (Burtt 1986). It could thus be metabolically-efficient for a bird to produce melanin to reduce the energetic cost of regrowing damaged feathers (Bonser 1995). Melanin's role in the proposed toughening of feathers can be explained by fracture mechanics: theories and methods that are frequently employed in high-performance composite production (Hayes & Gammon 2008). For example, particles of different properties to the bulk material are often incorporated in a composite matrix to increase toughness. This has the effect of forcing propagating cracks to develop around them, rather than through them (Dowling 1993), which slows the degradation or breakdown of a material. Rubber particles are frequently incorporated in brittle epoxy-based matrices for this purpose (Manson & Sperling 1989). This principle is illustrated in Figure 5-9:

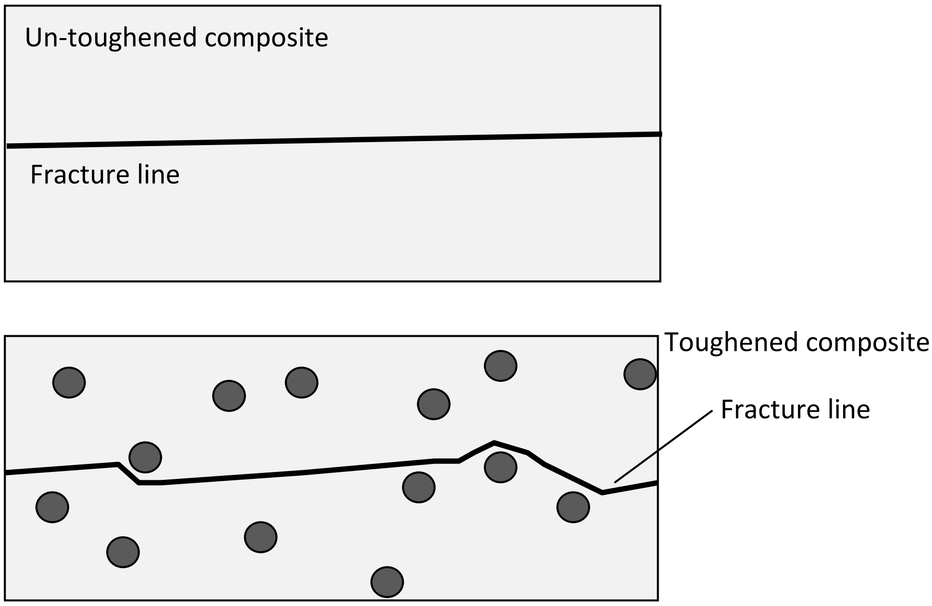


Figure 5-9 – schematics demonstrating the effect of a crack propagating across an un-toughened (top) and toughened (bottom) material
Melanin granules may act like the toughening particles present in the bottom sample

In hair, melanin has been shown to be softer than the surrounding keratinous matrix (Kitano et al. 2009) and this suggests that the melanin may act as a toughening agent in a similar way to rubber in manmade composites (Manson & Sperling 1989). However, it has not yet been determined whether the melanin has a measurable effect on the toughness properties of goose down when compared to duck down, which here possessed no melanin granules. Melanin's role as a toughening agent has been questioned by Butler and Johnson (Butler & Johnson 2004) and, regardless of the presence of melanin, keratins are matrix-filament composites already optimised to maximise their mechanical properties, so the melanin may not provide any additional toughness.

Though the role of melanin as a toughening agent is debateable, its effect on colour is better understood: melanin darkens feathers and other keratins. This may have important commercial implications as coloured down – that which contains melanin – is sometimes cited as less desirable than white down because the colour may sully the appearance of garments incorporating translucent face fabrics (Arc'teryx 2014), or may imply to consumers the presence of unclean or soiled feathers. Thus, melanin and its associated colour may need to be minimised to meet consumer expectations. This can be achieved using chemical treatments, as bleaching human hair has been shown to significantly degrade melanin granules (Imai 2011) and the same effect is expected to occur in down.

5.1.4 The elemental composition of duck down barbs

Following TEM analysis, there were no structures in duck down that required identification, meaning fewer samples were analysed than in goose down. However, elemental analyses of the cortex and cuticle were made. The areas that were analysed are shown in Figure 5-10 and the result of quantitative analyses carried out on these results is shown in Table 5-2:

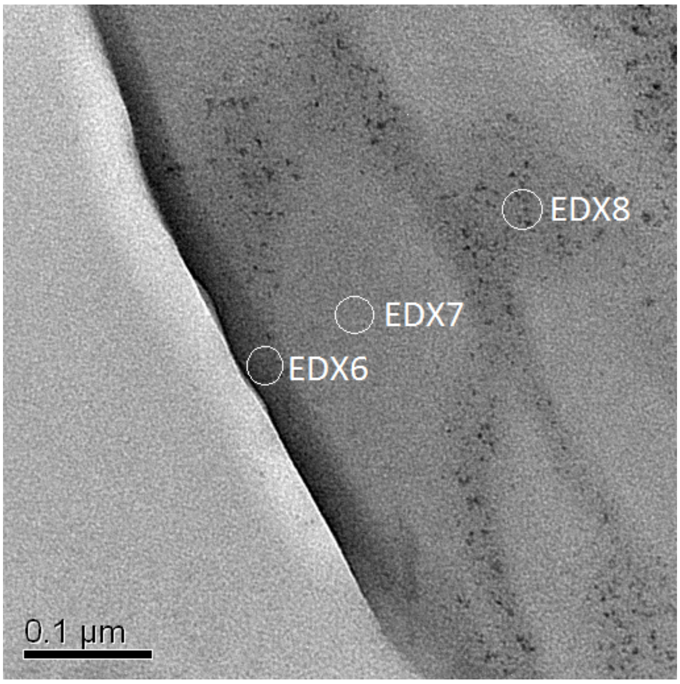


Figure 5-10 – duck down TEM sample undergoing elemental analysis using EDX
EDX6 (cuticle), EDX7 (cortex) and EDX8 (cortex) refer to the areas that underwent analysis

Table 5-2 – the percentage abundance of major elements in duck down cross sections

(n = 2 for cortex samples and 1 for cuticle)

	Atomic percentage of element (%)							
	Carbon (C)		Nitrogen (N)		Oxygen (O)		Sulfur (S)	
Feature	Mean	SD	Mean	Mean	Mean	SD	Mean	SD
Cuticle	84.70	-	9.96	-	3.83	-	1.51	-
Cortex	85.36	0.72	9.74	1.34	4.39	0.47	0.51	0.14

As shown in Table 5-1 and Table 5-2, goose and duck down's elemental compositions were very similar, though the percentage elemental composition of carbon was greater in the duck down than in the goose down (in cortex, duck: 85.36, goose: 78.94). Similarly, oxygen concentrations were slightly lower in the duck down (in cortex, duck: 4.39, goose: 5.62). The reason for this is currently uncertain, but the small number of duck down samples analysed may be a contributing factor. Despite the apparent differences in oxygen and carbon content between goose and duck down, the percentage of sulfur was very similar (in cortex, duck: 0.51, goose: 0.83), and the greater concentration of sulfur in the cuticle (in cuticle, duck: 1.51, goose: 2.03) when compared to the cortex was also observed in the duck down samples, as had been seen in the goose down samples.

5.2 The surface chemistry of goose and duck down feathers identified using attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy

Goose down has been analysed with infra-red spectroscopic techniques by two previous research groups (Gao, Yu & Pan 2007b; Zhang et al. 2011); duck down has been analysed by one (Zhang et al. 2011). A summary of the data from Zhang et al. regarding goose down is given in Table 5-3 (Zhang et al. 2011):

Table 5-3 – infrared spectroscopy results for goose down showing major absorption values (Adapted from Zhang et al. (2011))

Wavenumber (cm^{-1})	**Moiety**
3290	-OH, $-NH_2$
3060	-C-CH
1635	-CONH
1450	$-CH_2$
1335	$-CH_2$
1235	-NH
640	-CS

Very similar results to those in Table 5-3 were reported for duck down (Zhang et al. 2011); the only significant difference between them was the presence of an absorption band at 2400 cm^{-1} in duck down but not in goose. This absorption is characteristic of an -SH group and the authors (Zhang et al. 2011) thought that it may be the reason for the more pungent odour in duck down than goose down, though this has not been further investigated.

ATR-FTIR measurements were made using the technique described in section 2.3.5 to analyse the nonwovens that had been manufactured using the method described in section

2.3.3.4. The nonwovens were chosen for analysis because they were significantly denser than native goose or duck down and therefore yielded stronger signals.

The measured ATR-FTIR spectra for goose and duck down are shown in Figure 5-11 and the assignments of peaks is given in Table 5-4:

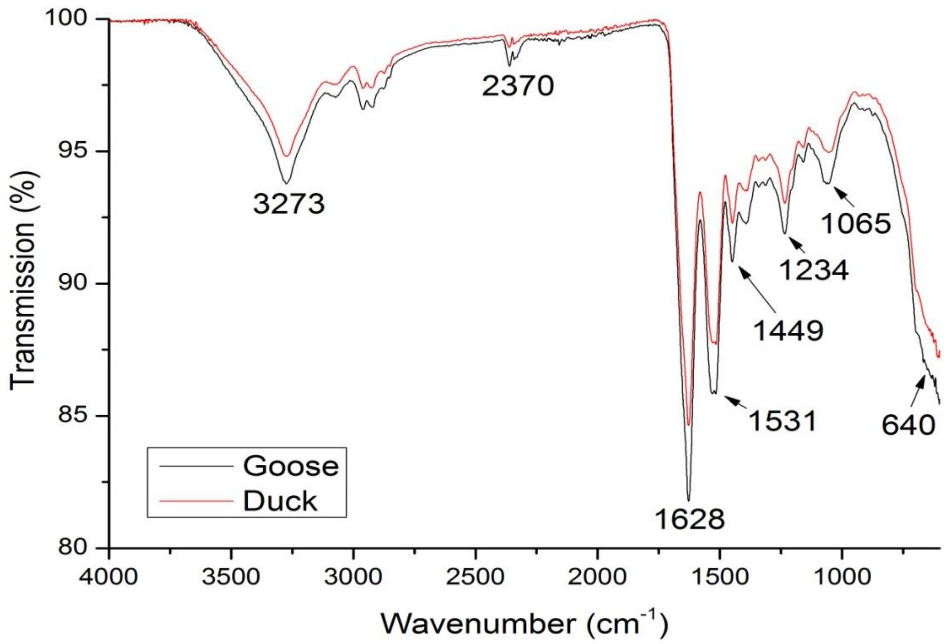


Figure 5-11 – ATR-FTIR spectra of goose and duck down
The wavenumbers of prominent peaks are highlighted

Table 5-4 - assignment of peaks in the ATR-FTIR spectra goose and duck down

(Jabs n.d.; Zhang et al. 2011; Chirgadze & Nevskaya 1976; Boulet-Audet & Holland 2011):

Wavenumber (cm^{-1})	Assignment
3273 (broad)	Amide A: N-H stretch; O-H stretch
3075 (weak)	C-CH stretch
2370 (weak)	S-H stretch
1628 (strong)	Amide I: C=O stretch
1531 (complex)	Amide II: N-H in-plane bend, C-N stretch
1449 (weak)	C-H deformation: CH_2, CH_3 bending modes
1234 (weak)	Amide III: -NH
1065 (weak)	Amide IV

The ATR-FTIR spectra (Figure 5-11) show goose and duck down to be very similar in their infrared properties. This is to be expected, as they are both keratinous, and therefore exhibit similar chemical properties. The differing surface oils of goose and duck down (discussed in section 1.3.4.2.1) are not easily detected using FTIR, because despite the differences in their structures, they are both principally hydrocarbon-based molecules. Zhang et al. (Zhang et al. 2011) obtained very similar spectra to those in Figure 5-11 in their analysis of goose down, though their spectra had much greater absorption at approximately 3500 cm^{-1}, attributed to the O-H stretch as a result of the presence of water. Different degrees of humidity in the test environment could influence this. The results in Figure 5-11 are also very similar to those previously obtained from swan feather rachis (Bradbury & Elliott 1962).

Peptides, such as keratin, elicit up to nine absorption bands (Jabs n.d.). Amide A (here at 3273 cm^{-1}) originates from a Fermi resonance between the first overtone of amide II; the other bands, labelled A, I, II, III, IV, etc. are due to fundamental absorptions or conformation of the keratin proteins. From the ATR-FTIR spectra, the β-sheet structures in avian keratin can be identified: their main contribution to the spectrum is at 1628 cm^{-1} (Chirgadze & Nevskaya 1976).

The absorption at 2370 cm^{-1} attributed to S-H that previous researchers (Zhang et al. 2011) only observed in goose down was present in both goose and duck down. As S-H is believed to cause down to smell (Zhang et al. 2011), and thorough washing of down may remove it (Bonser & Dawson 1999), this difference is probably a result of the varying processing conditions that different batches of commercial goose and duck downs undergo. Comparisons between the FTIR spectra of wool (Yao et al. 2008) and down show the great similarities in their surface chemistries and the only difference between wool, goose and duck down is that wool lacks the S-H stretch at 2370 cm^{-1}. This is likely to be due more aggressive scouring techniques used in the processing of wool.

The ATR-FTIR results show that the surface chemistries of the analysed goose and duck down samples, which influence hydrophobicity and wetting, are almost identical. No great difference in odour between the two samples could be detected, and this is mirrored in the ATR-FTIR results, which show similar absorption values at 2370 cm^{-1}.

5.3 Summary

In this chapter, EDX and ATR-FTIR spectroscopic analyses have been carried out on goose and duck down.

1) Down had a sulfur-rich cuticle (goose down: 2.03 % S in cuticle, 0.83 % S in cortex; duck down: 1.51 % S in cuticle, 0.51 % S in cortex).
2) In goose down, the presence of melanin granules was confirmed by their high oxygen and potassium content. The melanin granules may influence toughness, modulus, and due to their effect on colour, the desirability of a product.

3) The presence of a hollow cross section in goose down barbs was confirmed, as the EDX measurements from the centre of the barb matched that of the surrounding resin. The effect of the hollow centre on bending resistance will be determined in subsequent chapters.
4) ATR-FTIR spectroscopy illustrated the close chemical relationship between down and wool and the close relationships between goose and duck down, which were indistinguishable in this test.
5) Literature results from FTIR testing of goose and duck down showed a difference in the concentrations of sulfurous compounds on the down feathers' surfaces, but this difference was not detected here. It is likely that the concentration of sulfurous compounds and their associated smell is a result of processing methods used before the down reaches market.

In this chapter, goose and duck down have been shown to have similar chemical properties as their cross sections' elemental compositions and their surface chemistries are alike. In Chapter 4, the similarity between goose and duck down was also remarkable, as they had similar keratinous sub-components, underwent the same thermal transitions, and had very similar crystal structures. Thus, it can be concluded that any differences in their bulk properties are likely to be a result of morphological differences, such as the difference in cross sectional shape and the presence of a hollow centre to goose down barbs. Though eider down was not analysed in Chapters 4 and 5, it is likely that this conclusion can be extended to include this material.

Chapter 6. The mechanical properties of down barbs and plumes

The mechanical properties of down feathers, in particular their response to compressive forces, influence their thermal insulation characteristics. When used in insulated clothing and sleeping bags, down is subject to numerous compressive stresses: it is compressed through all directions when in a stuff-sack and must be made as small as possible; and when it is compressed under a sleeping person, by the wind, or by the small forces when in a baffle, it must try to counteract these stresses. The compression resistance of fibrous materials are closely linked to their bending rigidity, buckling properties (Van Wyk 1946), Young's modulus (Morton & Hearle 2008a), and the interactions between fibres. Tensile stresses are also common as down feathers are moved and stretched during use. Down feathers that break reduce in volume and therefore lose insulating ability, meaning that the tensile strength of these feathers is important. While the tensile properties of individual down plumes are very difficult to determine, the tensile properties of individual barbs can be measured.

In this chapter, the compression resistance of individual down plumes will be measured. These results will be correlated with the bulk properties of down feather assemblies in Chapter 7. The tensile properties of individual down barbs will also be measured, using two different instruments, and their cross-sectional shape determined in the TEM analyses of section 4.1, will be used to ensure accurate results. The barbs broken during tensile testing will be studied to yield further information regarding structure-property relationships.

6.1 The tensile properties of down barbs

The barbs and barbules that constitute down plumes are extremely fine and break under small stresses. Previous testing (Bonser & Farrent 2001; Bonser & Dawson 1999) of individual down barbs was performed using a tensile testing device purpose-built for the measurement of very fine fibres, equipped with a load cell with a maximal capacity of 2 N. While commercial devices of this level of sensitivity are very uncommon, a machine recently developed by Agilent Technology (USA) intended for testing nanofibres offers exceptional precision and resolution (the maximum load is 500 mN (Agilent Technologies 2013)). The relatively low throughput of this machine compared to most universal testing rigs, however, means that testing large numbers of samples is difficult, and so here the Agilent T150 is used mainly to ensure the accuracy of results from a Zwick Roell universal tensile testing machine which was easier to access and work with.

6.1.1 The tensile properties of down barbs tested using an Agilent nanomechanical testing rig

The test method used in this testing is described in section 2.4.1. Measurements from 25 goose and 22 duck down samples were analysed.

Typical stress-strain curves for goose and duck down barbs are shown in Figure 6-1 and the mean tensile properties shown in Table 6-1, with the Young's modulus and ultimate strength values based on the assumption that the barbs were circular in cross section:

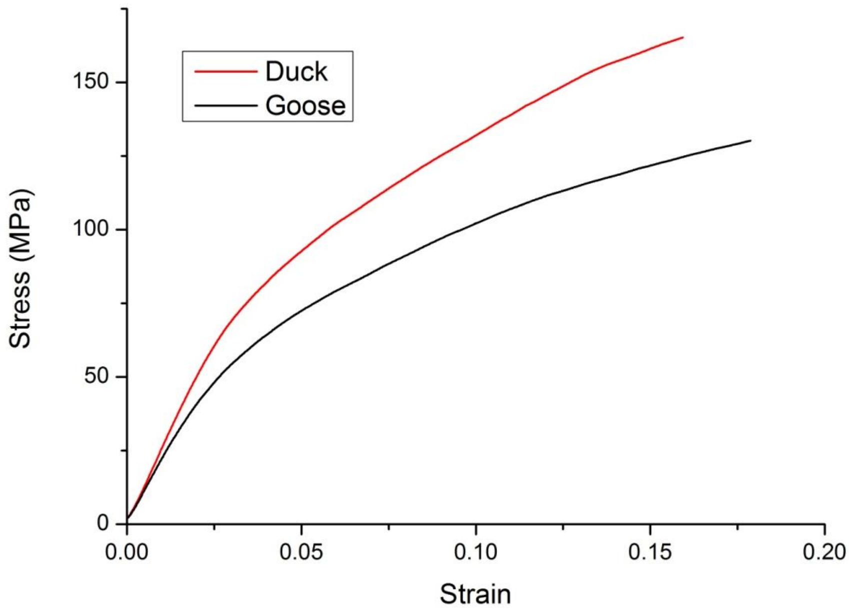


Figure 6-1 - typical stress-strain graphs for individual goose and duck down barbs undergoing extension

Table 6-1 - mean and SD tensile properties of goose and duck down barbs tested using the Agilent T150 and assuming barb circular cross sections

Sample	Fibre width (µm)		Young's Modulus* (GPa)		Strain at break		Ultimate strength* (MPa)	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Duck Down	17.96	4.57	2.41	1.48	0.14	0.05	158.28	87.60
Goose Down	19.05	4.52	1.94	1.10	0.16	0.06	136.74	87.08

(*Young's modulus and ultimate strength based on the assumption that both duck down and goose down possessed a circular cross-section)

Single-factor ANOVA analyses of the tensile properties of the down barbs shown in Table 6-1 indicated that the differences in diameter, Young's modulus, the strain at break, and ultimate strength between the duck and goose down barbs were not statistically significant ($p > 0.05$ in each case).

The mean values of Young's modulus and stress at break were very similar to those previously reported for duck and goose down (Bonser & Dawson 1999; Bonser & Farrent 2001) but previous researchers found a statistically-significant difference between the tensile properties of the goose and duck down barbs (for example, they found a greater modulus in duck down (Bonser & Dawson 1999)) which was not found in this experiment. The high SD values here are quite typical when testing natural fibres: numerous factors, including the genetic and environmental influences on the bird, and the processing conditions that the feathers undergo, all affect the final properties of the material. The high SD values are a factor in the non-significant differences between the properties of the two types of down feathers.

The values in Table 6-1 are very similar to those obtained from other feathers (Bonser 2001; Bonser 1996; Bonser & Purslow 1995), and the Young's modulus and ultimate strength are comparable to those obtained from wool and hair (Jachowicz 1987; Feughelman 1997). However, wool and hair extend much more before breaking (50-60 % strain (McKittrick et al. 2012), compared to 14-16 % here).This is because, while the Hookean and yield regions in Figure 6-1 are very similar those from wool and hair, the post-yield region that correlates with the straightening of wool and hair's α-helices is much larger (Jachowicz 1987). Avian keratins, however, cannot unravel and elongate under stress because their proteins are essentially fully-elongated in their native state (Wortmann & Zahn 1994).

6.1.1.1 The effect of down barbs' cross sectional geometry on their tensile properties

As found in section 4.1, down barbs are not circular in cross section, as assumed in Table 6-1, and this affects some of their tensile properties, such as Young's modulus and ultimate strength. While the previous section was useful for making comparisons to past results, in this section, assumptions were made that each duck down cross section was elliptical (Figure 6-2), and that each goose down barb was of the approximate shape shown in Figure 6-2. These values are thought to be closer to the real tensile properties of down barbs.

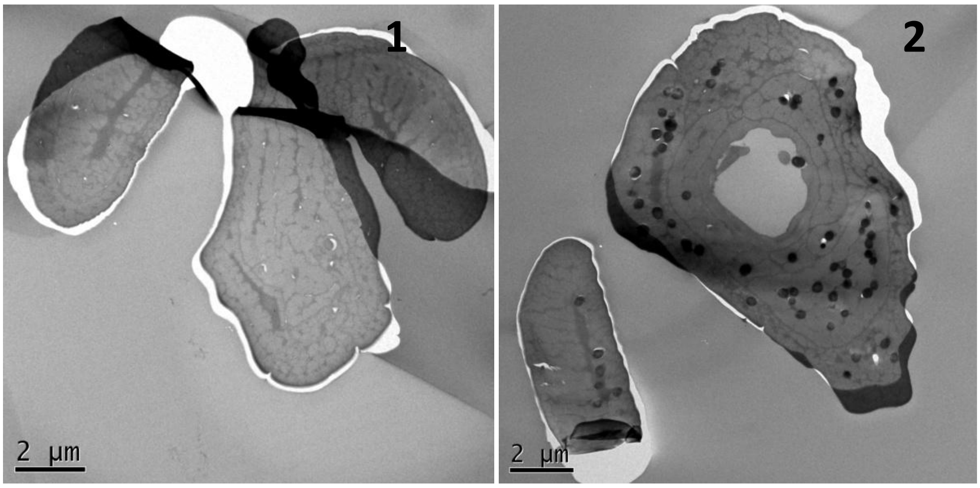


Figure 6-2 – TEM cross sections of down feathers used to estimate the tensile properties of barbs outlined in Table 6-2
1 – duck down (the central barb was used for analyses) ; 2 – goose down (the hollow barb was used for analyses)

The shapes in Figure 6-2 accurately represent the morphology of the down barb cross sections, but the TEM cross sections have been dehydrated and are thus far smaller than the down barbs when in their native state. This is considered in the following calculations.

Using the assumption that duck down's barbs were elliptical and possessed the dimensions in Figure 6-2 (height 9.7 µm, width 5.3 µm), one can calculate their area (Equation 6-1):

$$area\ of\ duck\ down\ barb\ (elliptical) = \frac{\pi \times 9.7 \times 5.3}{4} \quad (6\text{-}1)$$

$$area\ of\ duck\ down\ barb\ (elliptical) = 40.4\ \mu m^2 \quad (6\text{-}2)$$

If the average duck down barb (mean fibre width as shown in Table 6-1 is 17.96 µm) were assumed to be circular in cross section, the area of the barb is shown in Equation 6-3:

$$area\ of\ duck\ down\ barb\ (circular) = \pi \times \left(\frac{17.96}{2}\right)^2 \quad (6\text{-}3)$$

$$area\ of\ duck\ down\ barb\ (circular) = 253.3\ \mu m^2 \quad (6\text{-}4)$$

These values can then be used to create a scaling factor to convert the dimensions of the dehydrated cross sections into values consistent with those of hydrated feathers (Equations 6-5 to 6-7):

$$scaled\ area\ of\ duck\ down\ barb\ (elliptical) = \frac{d_{hydrated}}{d_{dehydrated}} \times \frac{d_{hydrated}}{d_{dehydrated}} \times area\ of\ duck\ down\ barb\ (elliptical) \quad (6\text{-}5)$$

where $d_{hydrated}$ is the diameter of the hydrated barb and $d_{dehydrated}$ is the diameter of the dehydrated barb

$$scaled\ area\ of\ duck\ down\ barb\ (elliptical) = \frac{17.96}{9.7} \times \frac{17.96}{9.7} \times 40.4 \quad (6\text{-}6)$$

$$scaled\ area\ of\ duck\ down\ barb\ (elliptical) = 138.5\ \mu m^2 \quad (6\text{-}7)$$

By using the area of the circular duck down barb and the existing Young's modulus and ultimate strength values from Table 6-1, the Young's modulus of the duck down when assumed to be elliptical can be determined, as shown in Equations 6-8 to 6-12:

$$Young's\ modulus\ (elliptical) = Young's\ modulus\ (circular) \times \frac{area\ (circular)}{scaled\ area\ (elliptical)} \quad (6\text{-}8)$$

$$Young's\ modulus\ (elliptical) = 2.41 \times \frac{253.3}{138.5} \quad (6\text{-}9)$$

$$Young's\ modulus\ (elliptical) = 4.41\ \text{GPa} \quad (6\text{-}10)$$

The ultimate strength can be determined in the same way:

$$ultimate\ strength\ (elliptical) = ultimate\ strength\ (circular) \times \frac{area\ (circular)}{scaled\ area\ (elliptical)} \quad (6\text{-}11)$$

$$ultimate\ strength\ (elliptical) = 289.5\ \text{MPa} \quad (6\text{-}12)$$

These calculated values of Young's modulus and ultimate strength are shown in Table 6-2.

Goose down can be modelled in a similar way to duck down, though this time using the shape shown in Figure 6-2's Picture 2, rather than an ellipse. The area of the barb is 77.0 μm^2, and the area of the hollow section is 7.6 μm^2 (measured using ImageJ software), thus giving an area of 69.4 μm^2 for the sum area of solid barb. The length of the longest side of the barb in Figure 6-2 is 10.8 µm, and this can be scaled to the mean diameter of the goose down barbs in Table 6-1 (19.05 µm) (Equation 6-13):

$$scaled\ area\ of\ goose\ down\ (irregular\ polygon) = \frac{19.05}{10.8} \times \frac{19.05}{10.8} \times 69.4 \quad (6\text{-}13)$$

$$scaled\ area\ of\ goose\ down\ (irregular\ polygon) =\ 215.6\ \mu m^2 \quad (6\text{-}14)$$

This area can then be used to calculate Young's modulus and ultimate strength:

$$Young's\ modulus\ (irregular\ polygon) = Young's\ modulus\ (circular) \times \frac{area\ (circular)}{area\ (irregular)} \quad (6\text{-}15)$$

$$Young's\ modulus\ (irregular\ polygon) =\ 2.56\ \text{GPa} \quad (6\text{-}16)$$

The value of ultimate strength is shown, alongside the other values for goose and duck down modelled using the shapes described, in Table 6-2:

Table 6-2 - mean and SD tensile properties of goose and duck down barbs tested using the Agilent T150 and assuming irregular cross sections

Sample	**Fibre width (µm)**		**Young's Modulus** (GPa)**		**Strain at break**		**Ultimate strength** (MPa)**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
Duck Down	17.96	4.57	4.41	2.71	0.14	0.05	289.48	160.21
Goose Down	19.05	4.52	2.56	1.45	0.16	0.06	180.76	115.10

(**Young's modulus and ultimate strength based on the assumption that duck down possessed an elliptical cross-section, and that goose down was in an approximately triangular shape with a hollow centre, akin to the shapes in Figure 6-2)

The non-circular cross section of goose and duck down influenced the Young's modulus and ultimate strength, as these values are defined per unit area. Similar effects were noted by researchers studying irregularly-shaped flax fibres (Baley 2002). The irregular cross sections resulted in goose down's modulus and ultimate strength increasing versus the circular cross section, but duck down's modulus and ultimate strength increased by a much greater degree, owing to the more elliptical cross section. The greater difference between the duck and goose down barbs' Young's modulus and ultimate strength values suggest differences that may be significant, as previous researchers found (Bonser & Dawson 1999). This will be discussed in greater detail in section 6.1.2.1.

6.1.2 The tensile properties of down barbs tested using a Zwick Roell universal testing rig

Limited access to the Agilent T150 testing machine meant that the tensile properties of eider down barbs – a material for which no apparent tensile testing results exist – could not be determined using this equipment. As such, eider down was tested using a Zwick Roell universal testing machine and samples of goose and duck down also tested to ensure good correlation between the two different testing machines. The test method used in is described in section 2.4.1 and 2.4.2.1.

Typical stress-strain graphs from the tensile testing of eider, goose, and duck down are shown in Figure 6-3 and assuming a circular cross section, a summary of the results from the testing is shown in Table 6-3:

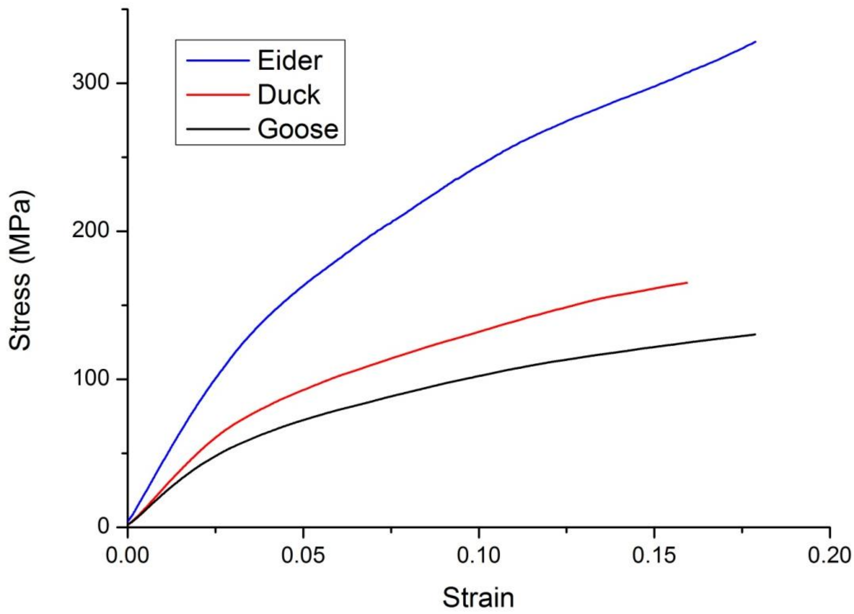


Figure 6-3 – typical goose, duck, and eider down barbs tested using the Zwick tensile testing machine

Though in this graph the duck barb appeared to have higher ultimate strength values than the goose down sample, the goose and duck down samples were on average extremely similar in this regard. The eider samples, however, broke at consistently higher stresses

Table 6-3 - tensile testing results from eider, goose and duck down barbs tested using the Zwick Roell universal testing machine

Sample	Barb diameter (µm)		Young's modulus (GPa)*		Strain at break		Ultimate strength (MPa)*	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Goose	19.87	4.59	2.76	1.51	0.10	0.05	164.6	70.8
Duck	21.10	4.25	2.12	1.52	0.11	0.05	144.8	96.8
Eider	21.76	4.99	3.20	1.21	0.19	0.04	283.4	116.6

(*Young's modulus and ultimate strength values based on the assumption that the duck, goose, and eider down barbs possessed a circular cross-section)

As in the testing in section 6.1.1, no significant differences (ANOVA single factor, $p > 0.05$) were detected between the tensile properties of goose and duck down. However, some of the values from eider down were significantly different: the Young's modulus of eider down was significantly greater than that of duck down, and the strain at break and ultimate strength of eider down was significantly greater than the values from both goose and duck down. When comparing the results of eider down testing with the results from goose and duck down from section 6.1.1, significant differences were found in each of the measured properties: diameter, ultimate strength, strain at break, and Young's modulus.

The differences in values in Table 6-3 reflect the environment in which the birds live: eider are wild ducks that live at sea while commercial geese and ducks live relatively docile lives primarily on land. Eider ducks have evolved very damage-resistant and stiff down feathers to resist the forces of the sea while minimising the metabolic requirements of growing new feathers. This is shown in the barbs' greater modulus, strength, and strain resistance versus goose or duck down barbs and also reflects the findings of Chapter 3. Future biomimetic insulations, should, like down feathers, possess mechanical properties optimised to their environment and end use. The greater tensile properties of eider down could be beneficial in outdoor clothing that needs to resist significant forces, such as under rucksack straps or in the underside of a sleeping bag. However, the extreme cost of eider down makes its use relatively prohibitive, and so the small differences in mechanical properties between it and goose or duck down are difficult to justify. However, the luxury and associated prestige of an expensive and rare material may make it desirable to some consumers.

The values generated from the Zwick Roell tensile testing are compared to those from the Agilent machine (section 6.1.1) in Table 6-4. Because literature values are also included in Table 6-4, the barbs are assumed to be circular in cross section to aid comparisons.

Table 6-4 – a summary of all previous literature results and values determined by the present testing regarding the tensile properties of goose, duck, and eider down barbs

Sample	Diameter (µm)		Young's modulus (GPa)		Strain at break		Ultimate strength (MPa)		Ref or section
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	
Goose	19.05	4.52	1.94	1.10	0.16	0.06	158.28	87.60	6.1.1
Goose	19.87	4.59	2.76	1.51	0.10	0.05	164.6	70.8	6.1.2
Goose	-	-	1.31	0.81*	0.12	0.03*	104	81*	(Bonser & Dawson 1999)
Duck	17.96	4.57	2.41	1.48	0.14	0.05	136.74	87.08	6.1.1
Duck	21.10	4.25	2.12	1.52	0.11	0.05	144.8	96.8	6.1.2
Duck	-	-	2.21	1.08*	0.10	0.03*	148	73*	(Bonser & Dawson 1999)
Duck	-	-	2.07	0.82*	0.14	0.05*	166.23	68.33*	(Bonser & Farrent 2001)
Eider	21.76	4.99	3.20	1.21	0.19	0.04	283.4	116.6	6.1.2

(*Standard error of the mean (*SEM*) values were originally quoted for these values; SD was calculated from *SEM* using the equation: $SD = SEM \times \sqrt{N}$, where N is the number of samples (Myers et al. 2013).) - implies that no value was given. Each value shown assumes that the cross section of the barbs were circular

Table 6-4 highlights the high variability of goose and duck down barbs' tensile properties. Even when testing feathers from the same supplier and batch, high standard deviation values were recorded in all data sets, indicative of the variations in this natural material. However, the diameters of goose and duck barbs were consistently between 18 and 21 µm, and the Young's modulus of duck down's barbs was consistently between 2.0 and 2.5 GPa, though the variability in goose down barbs' modulus was greater (1.3-2.8 GPa). The strain at break was quite consistent in both goose and duck down (0.10-0.16), and but for one literature value from goose down (Bonser & Dawson 1999), all ultimate strength values were between 135 and 170 MPa. As a result, though previous testing (Bonser & Dawson 1999) found that goose and duck down were significantly different in their strain at break, ultimate strength, and modulus, this is certainly not true for all down samples, and indeed was not true for all of the down samples studied here. Some of the differences in structure between goose down and duck down found in Chapter 3 and 4 (such as differences in their tertiary structures) were not anticipated to impact on the tensile properties of individual barbs, but goose down's hollow cross section might have been, as hollow polyester fibres have been found to be of lower strength than solid fibres (Khoddami et al. 2009). Similarly, the presence of melanin granules in goose down may have coincided with a reduction in modulus (Hwang et al. 1989) owing to the presence of these relatively pliant and energy-absorbing areas, but this was not observed in this testing.

Despite the similarities between the tensile properties of goose and duck down barbs, the differences between those of eider down and those of goose and duck were very apparent in Table 6-4 and shows that eider down barbs are of greater diameter, greater modulus, higher ultimate strength, and break at greater strain, than either goose or duck down barbs.

Down feather barbs are rarely subjected to maximal tensions in nature or when used in outdoor equipment, as a barb is never isolated and pulled. However, the forces encountered by a feather are significant, such as when sliding over other feathers, when buffeted by water or during flight, during grooming, when compressing or decompressing a garment, or during washing. That the goose, duck, and eider feathers possess a high resistance to tension and a relatively high strain at break means they can combat moderate tensional stresses without risk of breaking. Broken feathers are poor insulators and of little use to either a bird or man.

6.1.2.1 The effect of cross sectional geometry on the tensile properties of goose, duck, and eider down barbs

As described in 6.1.1.1, goose and duck down barbs are not circular in cross section, and so the same analyses that were carried out on the results from the Agilent T150 machine were carried out here on the results obtained from the Zwick Roell universal testing machine. The results from these analyses are thought to be closer to the 'true' mechanical properties of the down barbs, but comparisons to the results of past researchers are not possible.

Using the same techniques that were used in Equations 6-1 to 6-12, the area of the elliptical duck down barbs can be calculated from those in Table 6-3 assuming that they possess the elliptical shape shown in Figure 6-2:

$$area\ of\ duck\ down\ (elliptical) = 40.4\ \mu\mathrm{m}^2 \qquad (6\text{-}17)$$

If the average duck down barb (mean fibre diameter as shown in Table 6-3 is 21.10 µm) were assumed to be circular in cross section, the area of the barb can be calculated:

$$area\ of\ duck\ down\ (circular) = \pi \times \left(\frac{21.10}{2}\right)^2 \qquad (6\text{-}18)$$

$$area\ of\ duck\ down\ (circular) = 349.7\ \mu\mathrm{m}^2 \qquad (6\text{-}19)$$

Using the same method that was used in Equations 6-5 to 6-7, scaling factors can now be used to ensure the dehydrated and hydrated feathers are accurately represented:

$$scaled\ area\ of\ duck\ down\ (elliptical) = 191.1\ \mu m^2 \quad (6\text{-}20)$$

By using the circular and elliptical areas of the duck down and the existing Young's modulus and ultimate strength values from Table 6-3, the Young's modulus and ultimate strength assuming elliptical cross sections can be determined to give:

$$Young's\ modulus\ (elliptical) = 2.12 \times \frac{349.7}{191.1} \quad (6\text{-}21)$$

$$Young's\ modulus\ (elliptical) = 3.88\ \mathrm{GPa} \quad (6\text{-}22)$$

$$ultimate\ strength\ (elliptical) = ultimate\ strength\ (circular) \times \frac{area\ (circular)}{area\ (elliptical)} \quad (6\text{-}23)$$

$$ultimate\ strength\ (elliptical) = 265.0\ \mathrm{MPa} \quad (6\text{-}24)$$

These calculated values of Young's modulus and ultimate strength are shown in Table 6-5.

Goose down was modelled in an identical way to that shown in Equations 6-17 to 6-24 to give the values shown Table 6-5. Eider down was modelled using the highlighted barb cross section in Figure 6-4 (approximately elliptical, an area of 124.1 µm^2, a width of 12.5 µm, and a length of 23.1 µm) and the same method as was used for duck down:

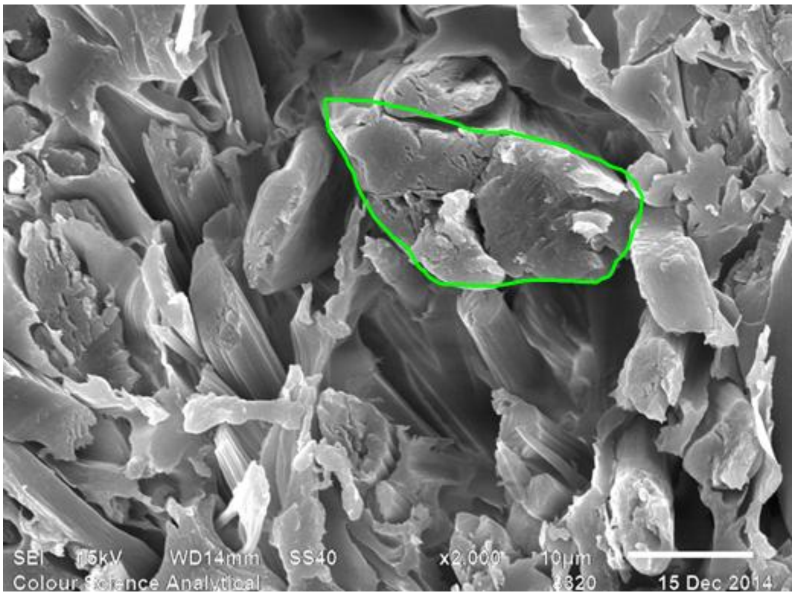


Figure 6-4 - cross section of an eider down barb and barbules
The dimensions of the barb highlighted in green were used to calculate tensile properties

Table 6-5 - tensile testing results from eider, goose and duck down barbs tested using the Zwick Roell tensile testing machine modelled assuming a non-circular cross section

Sample	Width (µm)		Young's modulus (GPa)**		Strain at break		Ultimate strength (MPa) **	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Goose	19.87	4.59	3.64	1.99	0.10	0.05	217.29	93.5
Duck	21.10	4.25	3.88	2.78	0.11	0.05	265.0	177.2
Eider	21.76	4.99	5.91	2.24	0.19	0.04	523.7	215.4

(**Young's modulus and ultimate strength results based on the assumption that duck down and eider down barbs possessed an elliptical cross-section similar to that in Figure 6-2 and Figure 6-4, respectively, and that goose down was in an approximately triangular shape with a hollow centre, akin to that in Figure 6-2

As shown in Table 6-2 and Table 6-5, the Young's modulus and ultimate strength of the goose and duck down barbs differed quite considerably when the cross sectional shape was considered. The values for ultimate strength, in particular, implied that duck down may be stronger than goose down (duck: 290 and 265 MPa; goose: 180 and 217 MPa). The Young's modulus was more similar (duck: 4.4 and 3.9 GPa; goose: 2.6 and 3.6 GPa), but again implied a slightly higher value in the duck down barbs. The hollow cross section of goose down's barbs may be the cause of their seemingly lower strength (Khoddami et al. 2009), and the potentially-lower Young's modulus may be due to the presence of melanin granules, which could act like rubber particles in plastics, which lower modulus (Manson & Sperling 1989). The impact, if any, of these features on compression resistance and recovery properties – arguably more important in an insulating material than tensile properties – will be determined in section 6.2 and Chapter 7.

When modelled using the elliptical cross section depicted in Figure 6-4, eider down barbs were, as found in section 6.1.2, significantly stronger and of significantly higher Young's modulus than either goose or duck down. This reinforces the conclusion that eider down feathers are quite distinct to goose and duck down, which can be considered relatively alike. This is almost certainly a result of the very different environments that the birds have evolved in.

6.1.3 Fracture mechanics

Studying fracture mechanics is an essential part of understanding many modern high-performance materials, such as carbon fibres (Tanaka et al. 2014; Naito et al. 2009) and their composites (Hojo et al. 1996). Despite being a high performance material, to the author's knowledge down barbs have not previously been analysed in this way, though other keratin fibres have been (Hearle 2002b; Hearle 2002a).

Goose and duck down feathers that were broken during tensile testing were analysed using SEM as described in section 2.2.2.2. The results from the goose and duck down barbs were very similar so will be analysed together.

The down barbs broke in a manner analogous to wool and hair: they split axially, granularly, or by a combination of both (Hearle 2002b). Granular fractures are shown in Figure 6-5 and axial fractures in Figure 6-6:

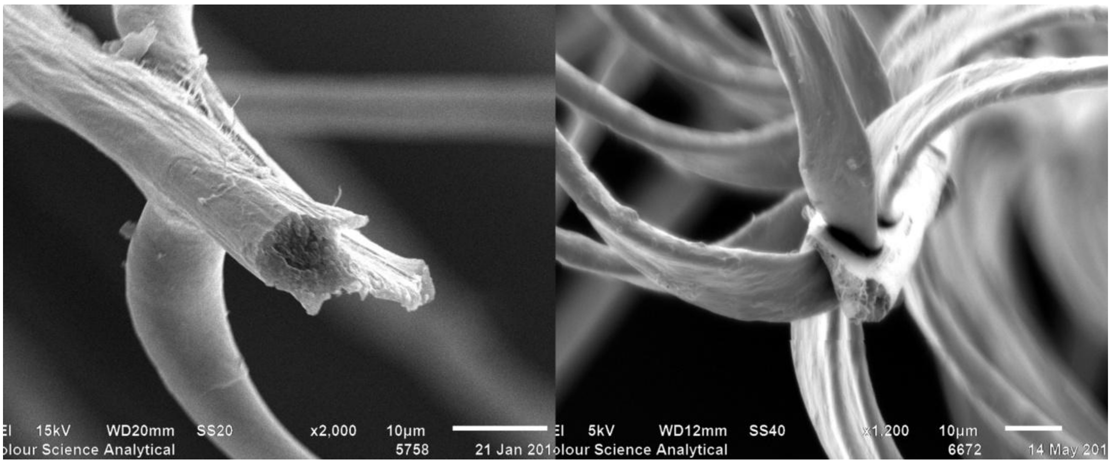


Figure 6-5 - SEM images of the fracture surface of two duck down barbs displaying granular fracture surfaces

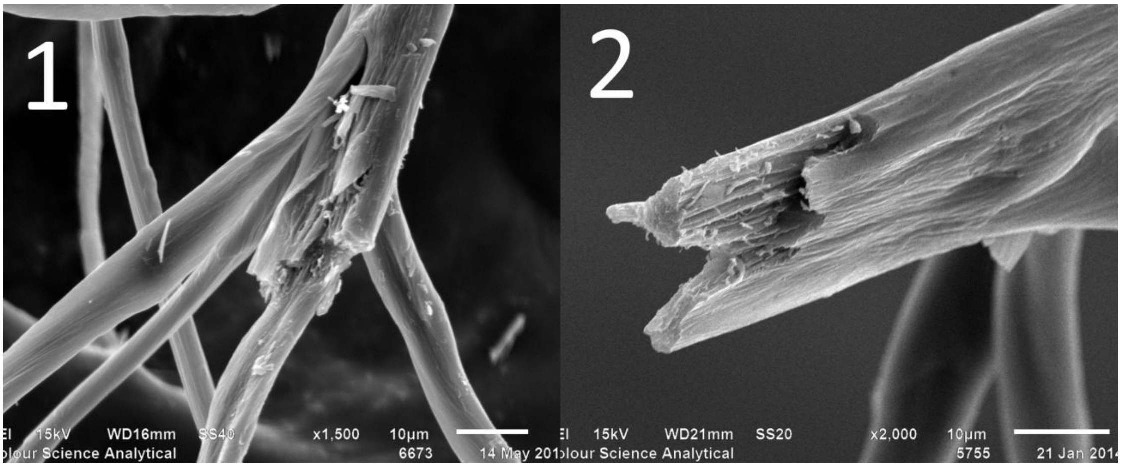


Figure 6-6 - SEM images of fracture surfaces with both axial and granular characteristics
1 – a goose down barb surface; 2 – a duck down barb surface. Greater detail is given in Figure 6-7

Both granular and axial fractures tend to occur when no obvious flaws are present in the fibre (Morton & Hearle 2008b) as flaws can create sharp breaks that emanate from them, but granular breaks imply the independent fracture of multiple fibrillar subunits.

The axial splits that are present in Figure 6-6 and shown in greater detail in Figure 6-7 and Figure 6-8 are characteristic of a fibre that has not broken due to a flaw but the initiation of multiple small fractures. The V-notch and separate cracks joining different granular regions, as shown in Figure 6-7's Picture 2, are particularly typical of axial splitting and are also seen in fibres such as acrylic (Morton & Hearle 2008b), another fibrillar fibre (Van Veld et al. 1968; Craig et al. 1962).

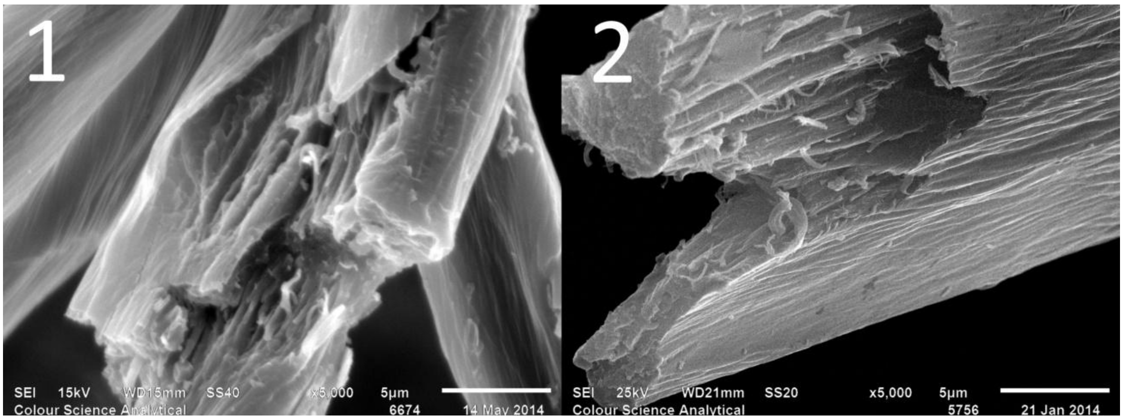


Figure 6-7 – higher magnification images of the axial and granular fracture surfaces shown in Figure 6-6
1 – a goose down barb surface; 2 – a duck down barb surface

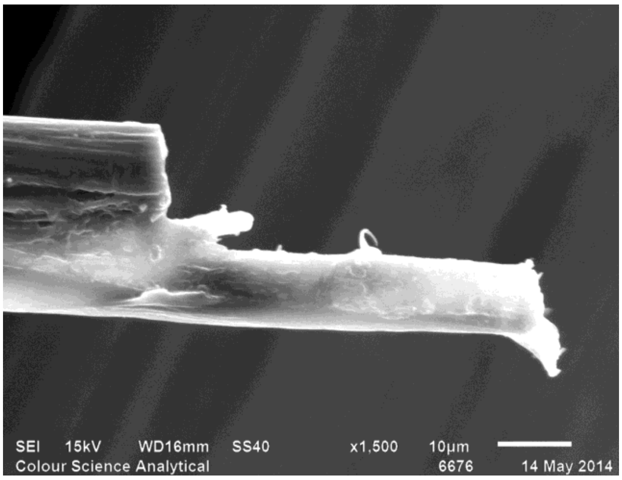


Figure 6-8 - SEM image of a fractured goose down barb showing axial splitting in a projection orthogonal to the fracture surface

The results of the fracture mechanical analyses further indicate that down's properties are similar to wool and hair's: the Chapman-Hearle model of wool (Hearle 2002b), which states that wool is a fibril-matrix composite, implies that the limiting factor in a fibre's strength is the break extension of the matrix, which is less than that of the fibrils. When the matrix fails the fibrils are subject to very high stresses and rupture to yield characteristic granular breaks (Hearle 2002b; Hearle 2002a). Though down is less extensible than wool or hair due to its avian, rather than α-keratinous protein structure, its mode of fracture is the same due to its fibril-matrix composition. These fracture mechanical analyses also highlighted how flawless the down barbs are, as cracks did not propagate from one area, but from multiple sites at once.

6.2 The compression resistance and recovery of down plumes and barbs

The compression testing of individual down plumes and individual down barbs was carried out. The plumes will be analysed in section 6.2.1, and the barbs in section 6.2.2.

6.2.1 The compression resistance and recovery of individual down plumes

Compression testing of individual down feather plumes was carried out to assess their compression resistance and immediate recovery. Though individual down feathers are never used in isolation, it is important to measure their properties as they clearly influence the properties of a bulk quantity of down. Also, the relationship between the bulk's properties and the individual feathers' properties reflects the interactions between the individual feathers, which are difficult to measure in isolation.

The method used to test the down plumes' compression resistance is described in section 2.4.2.2. 50 measurements of each of eider, goose and duck down plumes were made.

Examples of typical force/strain curves from the goose, duck and eider down plumes can be seen in Figure 6-9 and the individual shapes of the hystereses can be seen in greater detail in Figure 6-10:

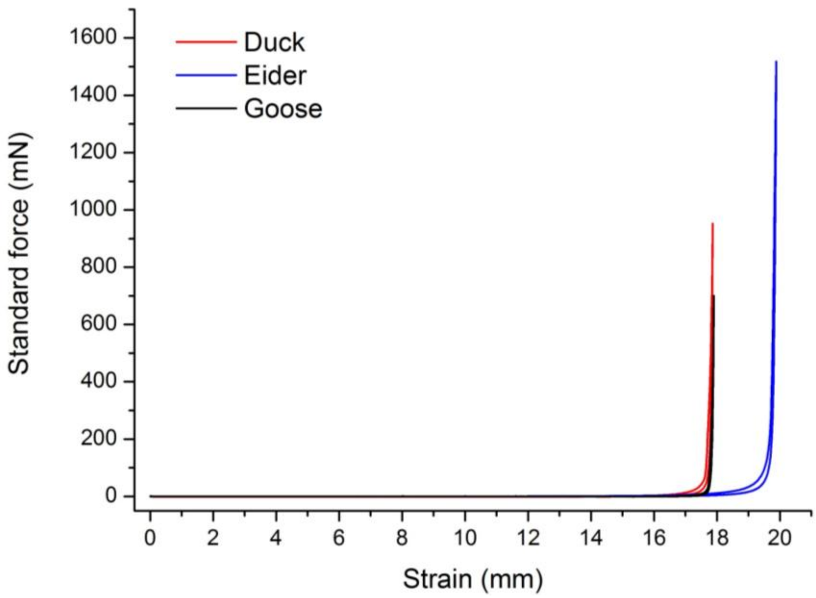


Figure 6-9 - force-strain curves from the compression of individual duck, goose and eider down feathers

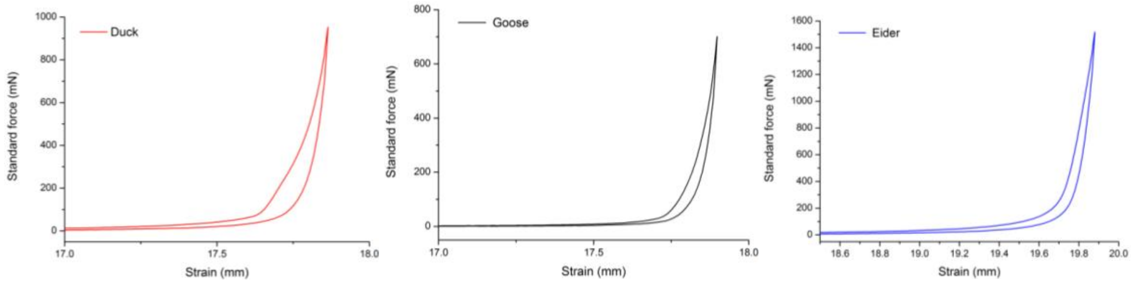


Figure 6-10 - force-strain curve of individual duck (left), goose (middle), and eider (right) plumes undergoing compression and recovery
(Expansions of Figure 6-9, highlighting the hystereses)

The mean and standard deviation results from the testing are shown in Table 6-6. Δ_{work} and the percentage Δ_{work} were calculated using Equations 6-25 and 6-26:

$$\Delta_{work} = work_{compression} - work_{recovery} \quad (6\text{-}25)$$

$$\% \, \Delta_{work} = \frac{work_{compression} - work_{recovery}}{work_{compression}} \times 100 \, \% \quad (6\text{-}26)$$

Table 6-6 - mean and standard deviation values from the compression of individual eider, goose, and duck down feathers

(Δ_{work} refers to the difference in work between the work in the compression phase and that in the recovery phase; % Δ_{work} refers to the difference in work as a percentage of the work in the compression phase)

Down	**Plume mass (mg)**		**Maximum force, Fmax (compression phase) (mN)**		**Work (compression phase) (N mm)**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**
Eider	2.02	1.17	1245.98	1014.50	0.208	0.210
Goose	0.77	0.62	1028.90	831.03	0.145	0.158
Duck	0.70	0.60	1022.98	1069.56	0.100	0.143

Down	**Δ_{work} (N mm)**		**% Δ_{work} (%)**	
	Mean	**SD**	**Mean**	**SD**
Eider	0.079	0.060	42.90	6.74
Goose	0.059	0.063	43.34	7.16
Duck	0.035	0.038	41.06	9.92

As shown in Table 6-6, the standard deviation figures of many of the down feathers' properties were quite large. This is a result of the down feathers' great variability in size, mass, and tertiary structures. The measurement of 50 down feathers of each type was approaching the maximum that was practicable in this experiment, and though in some circumstances this would be regarded as a large dataset, this remains a small sample size compared to the number of plumes that might be used in a garment or sleeping bag.

The force strain curves of the goose, duck, and eider plumes (Figure 6-10) were all very similar in shape, undergoing large displacement before the stress increased significantly. The contribution to thermal resistance from each plume is thought to be greatest when the feather is at its maximum size (therefore trapping the most air), so this low initial compressive modulus is not beneficial in providing maximal thermal resistance. However, it does mean that the down feathers can be compressed extremely easily to a small fraction of their original size, and this is beneficial when packing away equipment such as sleeping bags or insulated garments. The presence of other plumes around the test specimen would likely increase the initial compressive modulus by the inter-plume interactions of barbs, barbules, nodes, and prongs.

Box-and-whisker plots displaying the work in the compression phase from all the samples are shown in Figure 6-11:

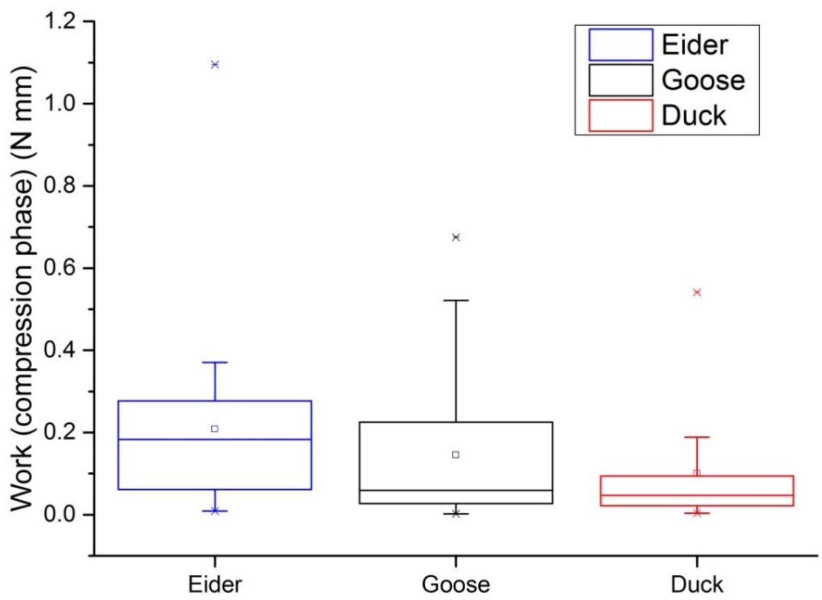


Figure 6-11 - box-and-whisker plots displaying the work in the compression of individual eider, goose and duck down feathers
x refers to the highest and lowest data values, the top whisker represents the 90th percentile, the top of the box represents the 75th percentile, the line inside the box the median value, the □ the mean value, the bottom of the box the 25th percentile, and the lower whisker represents the 10th percentile

As shown in Figure 6-11, the mean work of compression was marginally greater in goose down in than in duck down, though eider down had the greatest work of compression, being approximately double that of duck down and 30 % greater than goose down. The difference between eider down and goose down was not significant (p = 0.09), but the difference between eider down and duck down was highly significant (p = 0.006). The difference between goose and duck down was not significant (p = 0.16).

The maximum force in the application phase (Fmax), the difference in work in the compression and recovery phases, and the percentage difference in work as a percentage of work in the compression phase, are shown in Figure 6-12, Figure 6-13, and Figure 6-14, respectively:

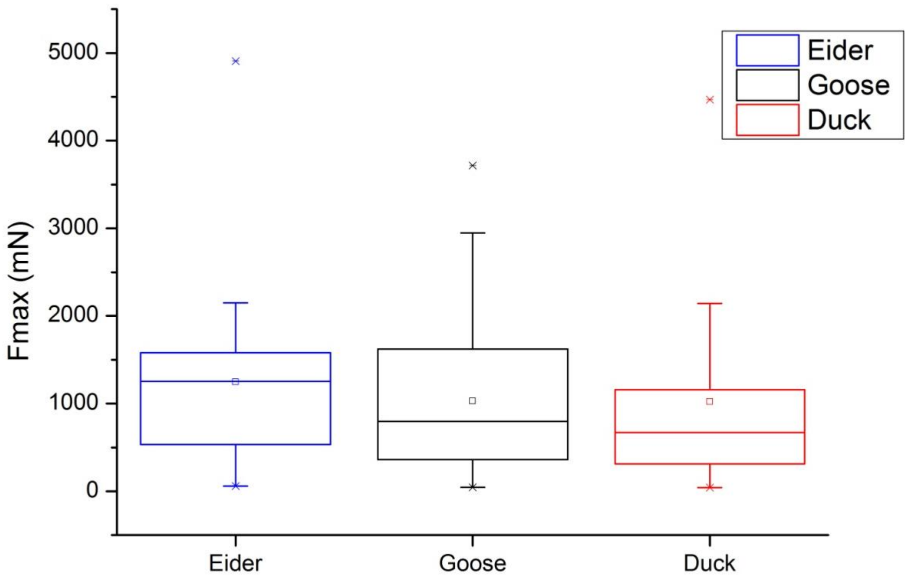


Figure 6-12 - box-and-whisker plots plotting the maximum force in the compression of individual eider, goose and duck down feathers

The mean maximum forces that the feathers resisted (Figure 6-12) were extremely similar in goose and duck down and though the eider down plumes' value was higher, this difference was not significant (eider to goose, p = 0.24; eider to duck, p = 0.30).

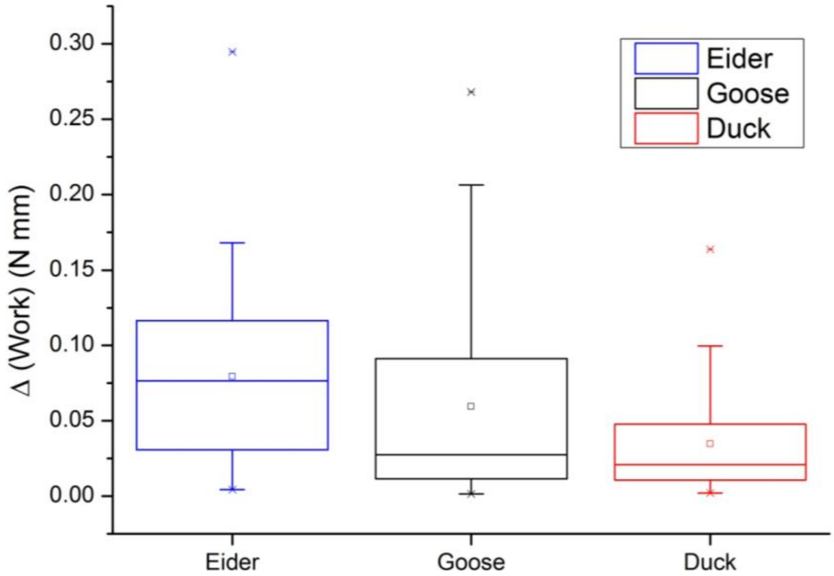


Figure 6-13 - box-and-whisker plots displaying the difference in work between the compression and recovery phases in the compression of individual eider, goose and duck down feathers

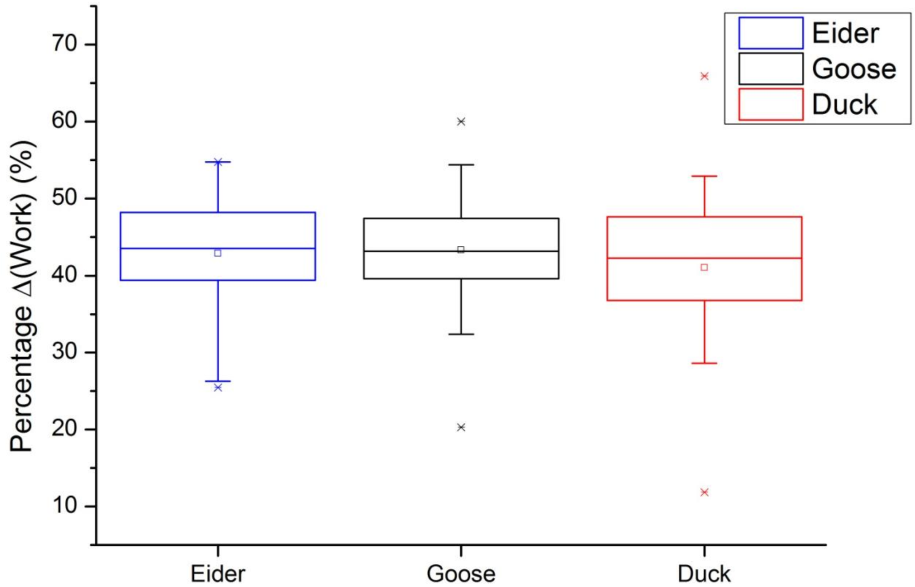


Figure 6-14 - box-and-whisker plots displaying the difference in work between the compression and recovery phases in the compression of individual eider, goose and duck down feathers

The difference in work between the compression and recovery phases was greater in the eider down plumes than in goose or duck down (Figure 6-13), which might indicate a greater hysteresis and therefore inferior recovery, but the difference in work as a percentage of the work in the compression phase (Figure 6-14) was extremely similar across the three different down feather types (all within 2 % of each other). This implied that despite the differences in their resistance to compression, their ability to recover from compression was identical. This shows that slippage of barbs and barbules across one another (contributing to unrecoverable energy in fibrous assemblies (Dunlop 1974)) was similar in each type of down, despite their differences in nodes and prongs. The effect of nodes and prongs may be more pronounced when multiple down feathers are present.

The findings in Figure 6-11 to Figure 6-14 regarding the work and maximum force in compression result from multiple factors: the individual bending moduli of the barbs and barbules, their diameter, their orientation, their number, and also their interactions with other neighbouring fibres through friction effects, sliding, and overlapping. The first attempt to model the interaction between fibres (Van Wyk 1946) ignored friction altogether (Dunlop 2008), and while this approach works well for some fibres (such as carbon fibres (Mousavi et al. 2014)), fibres with complex geometries, such as down barbs and barbules, possess significant frictional elements between them. The friction between barbs and barbules was anticipated to be greater in eider down than in either goose or duck down, owing to their greater number of nodes and prongs, and also the greater number of barbs and barbules themselves would contribute to greater overall compression resistance. The significantly greater work in compression for eider down versus duck down corroborates this. The non-significant differences in maximum force when eider down is compared to goose and duck down, and when comparing the work in compression to goose down, may partly be a result of the high variability of the materials. With a greater number of experimental repeats, these differences may become significant. Though not significant, the differences in mean work in compression between goose and duck down may be a result of goose down’s hollow cross section increasing bending resistance (Galileo 1914).

The great similarities between the maximum force values in goose, duck, and eider down were partly due to the design of the experiment: force was at its greatest when the minimum sample height of 1 mm was met, and the resistance at this point is thought to be a sum of the compression resistances of the flat barbs and barbules. Because the hardness of each type of down is very similar, as they are all keratinous, the maximum forces exerted are very similar. The influence of eider down feathers’ greater mass, and thus greater number of barbs and barbules, may be the reason for their marginally greater maximum force in compression. Because of the possible influence of mass on compression resistance, Figure 6-15 and Figure 6-16 were plotted using the mean feather masses reported in section 3.2.3 to hope to aid separation of the influence of mass from the other factors affecting compression resistance, particularly inter-fibre friction.

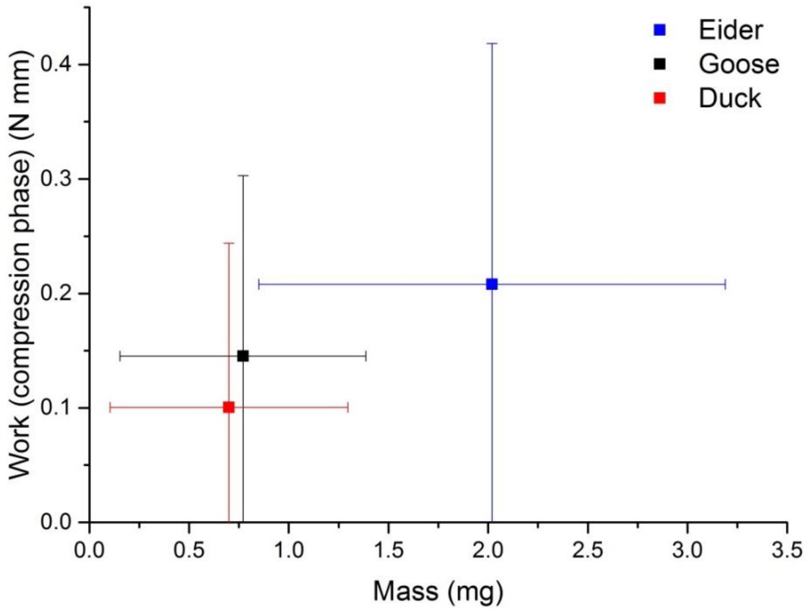


Figure 6-15 – scatter diagram displaying the mean work values in the compression phase as a function of mean plume masses (values from section 3.2.3) for individual eider, goose and duck down samples
The lines indicate standard deviations from the mean values

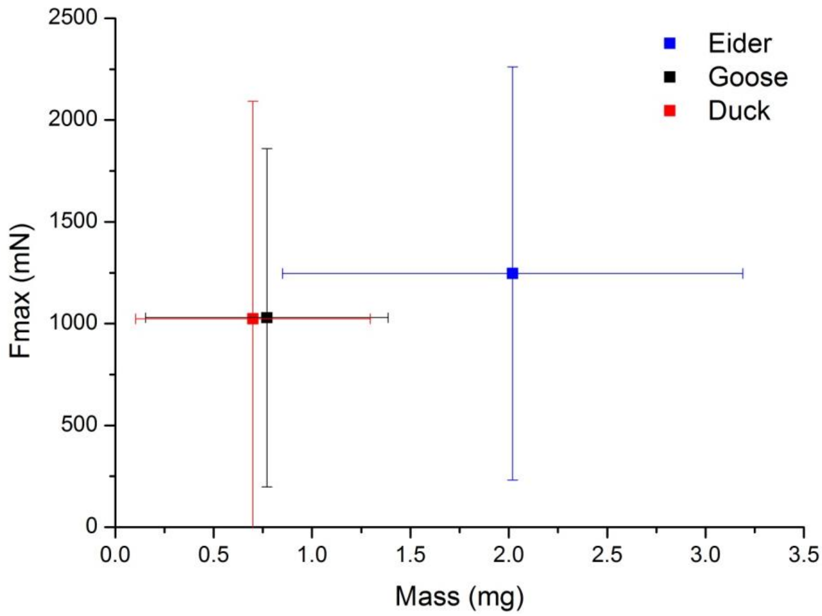


Figure 6-16 – scatter diagram displaying the maximum force values in the compression phase as a function of mean plume masses (values from section 3.2.3) for individual eider, goose and duck down samples
The lines indicate standard deviations from the mean values

The large standard deviation values in Figure 6-15 and Figure 6-16 made conclusions difficult to obtain from these graphs and it was not valid to relate work or maximum force to mass. An alternative experimental method, in which every down feather was weighed to five decimal places before being tested in compression, rather than using mean values, would allow for larger datasets to be compared to one another.

The compression of individual down feathers removes the interactions that they undergo with one another during use and provides an important bridge between the single-barb compression testing that will be carried out in section 6.2.2 and the testing of feather assemblies that will be discussed in Chapter 7. In this testing, goose and duck down were exceptionally similar in their performance, though eider down had a greater resistance to compression. This is thought to be due to its greater mass and subsequent number of barbs and barbules, creating more potential points of overlap and increasing the amount of material that must be compressed. The work of recovery of the three feather types was near-identical, suggesting that the slightly-different morphologies of the down feathers' barbs, barbules, and tertiary structures do not necessarily affect the compression recovery of the down feathers.

6.2.2 Uniaxial compression and bending of individual feather barbs

Compression testing of individual down feather barbs has previously been carried out by researchers (Gao et al. 2010), though their testing analysed only 2 mm barb lengths. While this gives a good impression of the compressive strength of the material, it does not relate particularly to applications in outdoor equipment, as down barbs are never this short nor compressed in this fixed way. In this experiment, a 15 mm fibre length was chosen as this enabled barbs from larger plumes to be mounted securely to card in a way analogous to the way used in tensile testing (method in section 2.4.1). This testing complements that of the plume testing from section 6.2.1 as it negated much of a down feather's geometry, disregarding the possible shape effects of a barb's cross section. The method used to analyse the barbs is described in section 2.4.2.3.

In engineering terms, a fibre with high aspect ratio held between two points is a strut. Struts may collapse due to compressive yield (demonstrated in Figure 6-17) or by buckling and bending, as described in Figure 6-18. Short struts (those with a relatively small aspect ratio) tend to fail by compressive yield, and those with high aspect ratio usually fail by buckling.

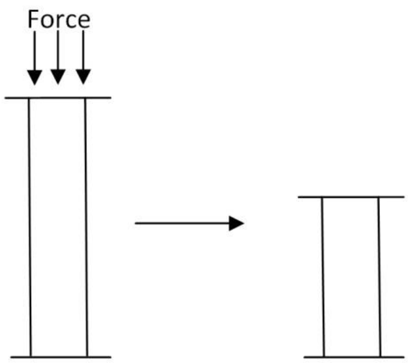


Figure 6-17 - pictogram representing compression of a short wide strut by compressive yield

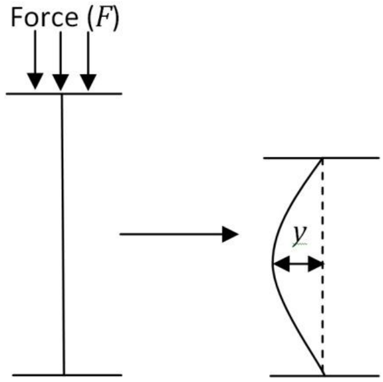


Figure 6-18 - pictogram representing bending of a thin strut
y is the deformation and the applied bending moment is equal to Fy

Theoretical considerations of compression were first made by Euler, who developed the formula in Equation 6-27, describing buckling:

$$F_{crit} = \frac{EI\pi^2}{L^2} \quad (6\text{-}27)$$

where F_{crit} is the critical force in N required to impart bending or buckling, E is the Young's modulus in Pa, I is the moment of inertia in m^4, π is a constant, and L is the strut's length in m

A fibre of circular cross section mounted exactly perpendicular to the jaws can be described as a point mass because the mass about the vertical axis is equal on all sides. The moment of inertia, I, of a point mass can be described as shown in Equation 6-28 (Clifford et al. 2009):

$$I = mr^2 \quad (6\text{-}28)$$

where m is the mass in kg and r is the radius from the axis in m

Substituting Equation 6-28 into 6-27 yields Equation 6-29, in which Euler's equation describing buckling has been expanded to include the terms used to describe the moment of inertia:

$$F_{crit} = \frac{Em(\pi r)^2}{L^2} \quad (6\text{-}29)$$

From Equation 6-29 it can be seen that the further the centre of mass is from the axis (i.e. the greater the value of r), the greater the moment of inertia and subsequent resistance to bending. This is why a hollow tube possesses greater bending resistance than a solid one of equal mass. However, the radius of the tube cannot be increased indefinitely without reducing thickness of the walls, or mass will increase (Meyers et al. 2013). With decreasing thickness of the walls a point will eventually be reached where local buckling increases in likelihood due to defects in the structure (Timoshenko & Gere 1989). In nature, multiple methods have been used to combat buckling (Meyers et al. 2013), such as the honey-comb-like core of a flight feather. The hollow core in goose down's barbs is thought to increase their F_{crit} value, owing to the greater distance between the centre of the columns and their mass increasing moment of inertia.

Thin struts tend to undergo bending rather than compressive yield because of the small F_{crit} – the force required to bend or buckle a strut – that derives from their small radius. No increase in the applied force is required for the bending moment to increase because an increase in deflection, y, is sufficient to bring-about an increase (as the applied bending moment = Fy), as shown in Figure 6-18. Thus, as soon as a strut begins to deform, its bending moment will increase without a change in the applied external force. This explains the rapid and sudden failure of columns that buckle or possess defects.

Euler explored different methods of attaching the strut to its mountings (the constraints) and found that they affected the F_{crit} value (Institute for Steel Development and Growth n.d.). The work by Gao et al. (2010) regarding the compression of down barbs used one pinned end and one fixed end. That is, one end was able to rotate but not translate, and one end was completely unable to move. In the current work, both ends were fixed and thus unable to move. Fixed ends tend to produce deformations as shown in Figure 6-19:

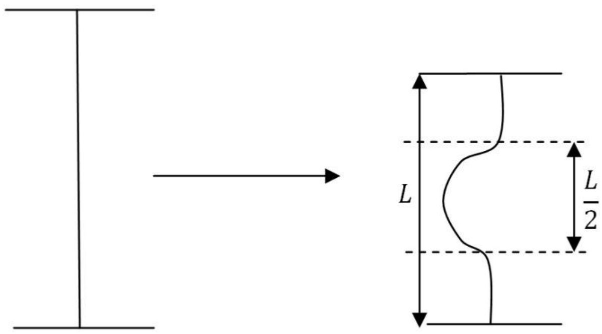


Figure 6-19 - representation of the compression of fixed-end struts

6.2.2.1 Results and discussion

No quantitative values could be obtained from this testing because the forces exerted during compression of the barbs were too small (peak forces of 0.2 mN were recorded during the compression phase and the work during the same period never exceeded 0.000 mN (to three decimal places, the limit of the load cell's resolution)). The small forces meant that the compression properties of the barbs were indistinguishable from the testing noise using the present equipment.

Despite the low peak forces measured, the testing proved useful in describing, in a qualitative sense, the bending that barbs undergo. There appeared to be no difference between the bending characteristics of goose, duck, or eider down barbs and as such they will be described together.

Most down barbs initially deformed as shown in Figure 6-19 but as compression continued they followed the pattern shown in Figure 6-20. Approximately 90 % of the down barbs deformed in this way; the remainder deformed as described in Figure 6-21:

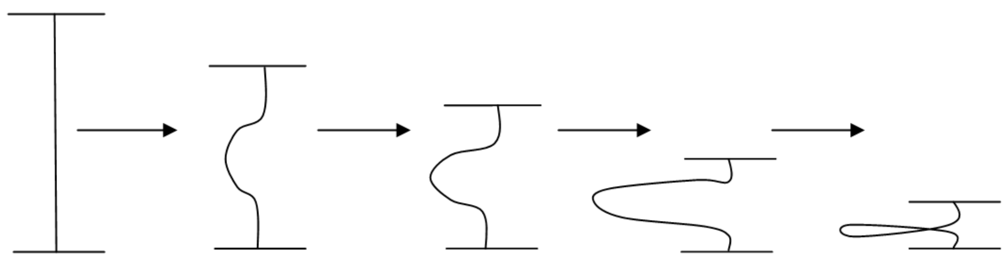


Figure 6-20 - pictograms representing the compression of a down barb undergoing bending
This was a more common bending mechanism than that shown in Figure 6-21

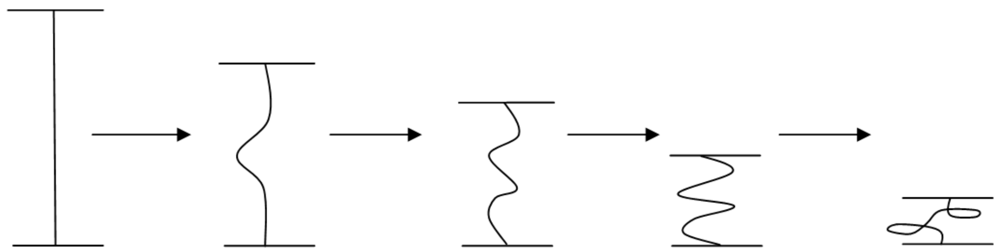


Figure 6-21 - pictograms representing the compression of a down barb undergoing bending
This mechanism occurred in far fewer samples than the sequence shown in Figure 6-20

The deformation shown in Figure 6-20 is the fundamental bending characteristic of a thin strut and can be described using Euler's buckling formulae, shown in Equation 6-30, a modified version of Equation 6-29:

$$F_{crit} = \frac{EI\pi^2}{{L_{eff}}^2} \qquad (6\text{-}30)$$

where L_{eff} is the effective length of the strut that undergoes bending

When the ends of the strut are fixed, the value of L_{eff} is $\frac{L}{2}$, as half of the fibre's length deforms (Institute for Steel Development and Growth n.d.), as shown in Figure 6-19. Euler's equation can thus be rearranged to give Equation 6-32:

$$F_{crit} = \frac{EI\pi^2}{\frac{L}{2}^2} \qquad (6\text{-}31)$$

$$F_{crit} = \frac{4EI\pi^2}{L^2} \qquad (6\text{-}32)$$

In general, buckling and the modes of bending can be described by Equation 6-33:

$$F_{crit} = EI\left(\frac{n\pi}{L}\right)^2 \quad (6\text{-}33)$$

where n = 1, 2, 3, ..., ∞.

From Equation 6-33, $n-1$ is equal to the number of nodes (points where displacement in relation to the axis = 0) present in the bending fibre. Thus, the fundamental mode (shown in Figure 6-20) has no nodes, and Figure 6-21 is the second overtone and has two nodes (n = 3 as L_{eff} = 1/3). That these theoretical modes were observed in these experiments implies that very few local imperfections exist in the barbs. It is also remarkable that even when down barbs undergo significant bending, no knotting or tangling occurs, despite the presence of barbules and nodes. The non-cylindrical shape of the goose, duck, and eider barbs was further verified by the character of their bending, which consistently occurred in the same direction in each individual sample, following the axis of least bending resistance.

The impact of barb diameter on bending resistance is shown in the radius squared term in Equation 6-29. The diameters of both goose and duck down barbs are smaller than those of eider down, as proven in section 3.3.1, and the large effect of radius on bending resistance helps explain the findings from section 6.2.1 that eider down plumes are more compression resistant than goose or duck down plumes. Thus, despite no quantitative results being generated in this testing, it is highly likely that eider down barbs would have a greater bending resistance than duck or goose down barbs, and due to their hollow core, goose down's barbs would be more bending resistant than duck down's. It has been stated (Skelton et al. 1985) that one of the keys to down's performance is its mixture of coarse and fine fibres: coarse fibres increase bending resistance and thus combat compression, whereas the finer fibres provide a barrier to radiative transfer. The work in this chapter supports the first part of this statement.

6.3 Conclusions

In this chapter, the tensile testing of individual goose, duck, and eider down barbs has been carried out, and the compression properties of individual goose, duck, and eider down plumes and barbs determined.

1) Tensile testing of individual down barbs was carried out using two different machines. The values from the tensile testing were comparable to previous literature results (Bonser & Farrent 2001; Bonser & Dawson 1999) when the barbs were modelled as cylinders, but when the goose, duck, and eider down barbs were modelled using shapes determined through TEM and SEM investigations, their moduli were higher than those from previous studies.

2) When modelled as cylinders, the mechanical properties of goose and duck down barbs were not significantly different to one another ($p > 0.05$).
3) Results from the eider down barbs showed significant differences ($p < 0.005$ in each case) in ultimate strength, strain at break, Young's modulus and fibre diameter to barbs from goose and duck down. This has been attributed to the greater stress resistance required of eider down, which must withstand great forces: eider are seabirds that have to counter harsh weather and sea conditions and the metabolic cost of replacing weak feathers is likely to exceed the metabolic cost of producing strong and more durable feathers.
4) Fracture mechanical analyses of duck and goose down barbs broken during tensile testing showed granular or axial splitting characteristics that are also seen in wool and hair fibres. These indicative barbs with no local imperfections, and instead cracks propagate from multiple sites simultaneously.
5) Individual eider down plumes were significantly more resistant to compression than those of either goose or duck.
6) Each of the down feather types recovered from compression to a very similar degree (within 2 %), indicating that the differing geometries of the feathers' barbs and differing nodes and prongs did not affect compression recovery when testing feathers in isolation of others.
7) Attempts to relate mean feather mass to compression resistance were unsuccessful owing to the large standard deviation values of both properties; a different method that measured the mass of each individual feather might be more fruitful.
8) Experiments into the bending and compression properties of individual down barbs did not generate quantitative results but showed that down barbs' bending can be modelled very effectively by the theoretical models of Euler, implying that they are fibres without many flaws or imperfections.
9) Euler's theories suggest that the hollow centre of goose down's barbs is expected to impart them with a greater bending resistance than the solid-cored duck down barbs.

This chapter provides an important assessment of the individual properties of eider, goose, and duck down feathers and forms an important link to the properties of bulk down feathers, which will be discussed in Chapter 7.

Chapter 7. The compression resistance and recovery, and the thermal properties, of down feather assemblies

In Chapters 3, 4, and 5 it was identified that goose and duck down differed in their structure, such as the presence of a hollow core in goose down, and different nodes and prongs. These differences were not particularly apparent in the properties of individual down feathers and barbs tested in Chapter 6, but the contributions from multiple feathers may make the dissimilar structures more influential in determining properties. In this chapter it will be determined whether the properties of down assemblies correlate with the mechanical properties of individual down feathers and barbs determined in Chapter 6, and with the structures identified in Chapters 3, 4, and 5.

The three most important attributes of an insulating material designed to be used in the outdoors are its thermal, compressive, and sorption properties. Here, the thermal and compressive properties of goose and duck down will be measured and compared to one another. Despite scant previous comparisons between the two materials, goose down commands a significantly higher market price (Bedard et al. 2008; Jacob et al. 2011; IDFL 2010a) than duck down and a common question amongst users of insulated equipment is "which is better; goose or duck down?" (UKClimbing 2014). This chapter aims to help answer this question in relation to the down properties identified in previous chapters.

The compression characteristics of down feather assemblies have been previously tested by Gao (2010) and Gibson (1990), using quite different approaches: Gao compressed masses of down at a lower pressure and agitated them to restore their bulk; Gibson measured the compression properties of down contained inside face fabrics and then compressed them using much higher pressures. In this chapter, both of these methods will be adopted to analyse the compression properties of down feathers in their raw state and inside down-filled test squares representative of real products. The relationship between the results of low-pressure fill power testing and compression resistance at higher forces will be scrutinised, and the effect of repeated compressions and agitation on down assemblies will also be discussed.

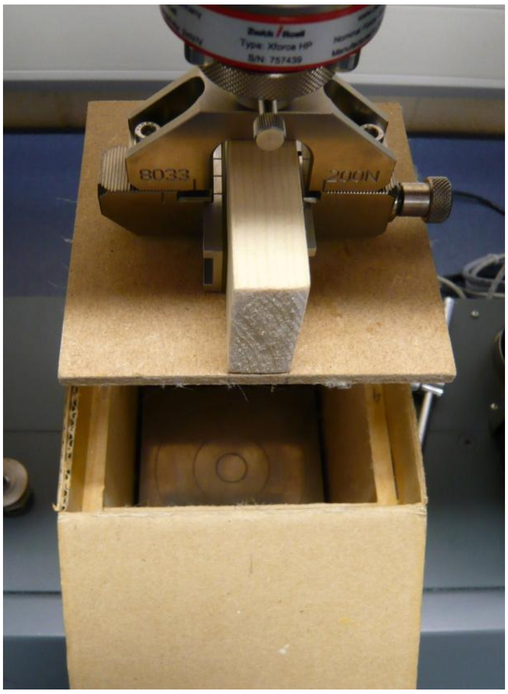
The quantity of down to put in a garment's or sleeping bag's baffles has traditionally been determined by trial-and-error without rigorous scientific justification. In this chapter, the relationship between the thermal insulation properties and the fill-weights of down-filled test squares will be studied to determine both their thermal resistance and thermal resistance per unit mass. This is of particular use to the development of high performance equipment used in the outdoor industry but is relevant to any manufacturer of down-filled goods.

7.1 The compression resistance and recovery properties of down feather assemblies in modified fill power tests

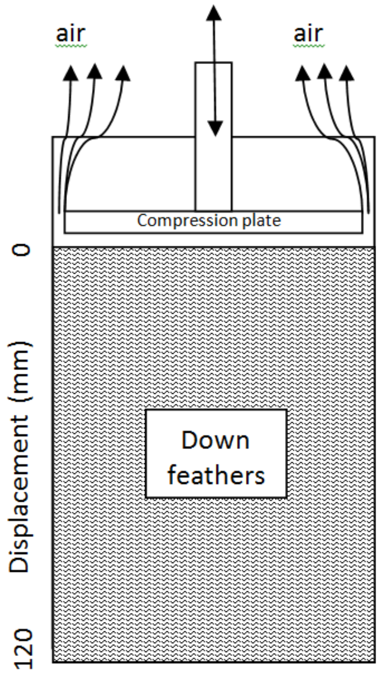
Down's compression resistance and recovery from compression are among its most important attributes. In industry, compression resistance at lower pressures is measured using fill power testing, which compresses down to one fixed pressure (14.80 Pa in the British Standard test (British Standards Institute 1998a)). In these experiments, a range of pressures will be used that will include the pressure exerted in a fill power test and much higher pressures, such as those that may be encountered in use, either in a sleeping bag or insulated garment.

7.1.1 Experimental method

The compression of goose and duck down feather assemblies were carried out using the Zwick Roell universal testing machine described in section 2.4.2. A novel test method was developed and used alongside the universal testing machine: a stiff-walled rectangular tube of internal dimensions 123 x 123 mm was constructed from smooth fibre board and then reinforced with 6 mm thick plywood. It was placed over the Zwick Roell lower compression plate (120 x 120 mm). The disparity in dimensions between the box and the compression plate meant that air could escape the bottom of the container, preventing false readings due to air pressure, while the gaps were too small to allow down feathers to escape the container. The top edge of the box was 125 mm above the lower compression plate. Due to the great required sensitivity, a lightweight compression plate of dimensions 120 mm x 120 mm was constructed from stiffened fibre board and then reinforced. This apparatus is shown in Figure 7-1:



1) Box and compression plate



2) Schematic of compression equipment

Figure 7-1 – photograph and schematic of the box and compression plate mounted on the Zwick Roell universal tester for use in the compression of down feather assemblies

The Zwick Roell testing machine was equipped with a 200 N load cell and 200 N jaws. The compression plate was clamped into the jaws and lowered under it made intimate contact with the lower compression plate and the plate-to-plate separation was then set to zero. The rectangular tube was placed over the compression plate and 1.800 ± 0.005 g of down weighed into it, which was the mass of down that approximately filled the box without risking it being disturbed by currents of air. From a starting height of 120 mm the plate was lowered onto the down sample at a rate of 200 mm min^{-1} until a preload of 0.03 N (2.08 Pa) was met. The test then began and continued at a speed of 30 mm min^{-1} until a plate-to-plate separation of 1.5 mm (a displacement of 118.5 mm) was reached. This was the minimum height that the plate could reach without causing a rapid increase in force with potentially damaging consequences for the load cell. The recovery was measured as the compression plate rose at the same speed as during compression.

Numerous tests were carried out on both goose and duck down, each described in Table 7-1:

Table 7-1 - experimental plan for the compression of goose and duck down assemblies

Test series	Compression resistance under which conditions?	Agitation applied?	Previous compression cyles
1	In agitated down	Yes, prior to experiment	None
2	In previously-compressed down	No	One
3	Of down undergoing multiple compressions	Yes, prior to first experiment and after 10th compression cycle	None, but 10 compression cycles carried out in this test

Agitation was given by gently stirring and shaking of the down inside the box by hand for approximately 30 seconds and waiting 60 seconds before beginning the next test. For each test series given in Table 7-1, three samples of goose and duck down were tested, and three repeats were made on each sample. Testing took place in a monitored atmosphere of 20 ± 1.5 °C and 55 ± 5 % relative humidity.

7.1.2 Comparisons between the compression resistance at high pressures and those experienced during the fill power test

Fill power is the industry-standard method of assessing a down sample's performance but information regarding the relationship between the resistance of a down sample at small compressive loads (those measured in fill power tests) to higher forces that may be exerted in situations such as under a rucksack's straps, in a sleeping bag under a supine person, or between other garment layers, is scant. The pressure applied to the down sample in the EN12130: 1998 filling power test is 14.80 Pa (British Standards 1998a) and though the pressures applied in other fill power testing standards vary slightly, they all use pressures of less than 20 Pa. In these experiments, down samples underwent compression to their minimum thicknesses, and the work and displacement were also measured at 14.80 Pa.

The displacements undergone by the down feather assemblies to the point where the pressure exerted in the fill power test was met, are shown in Figure 7-2:

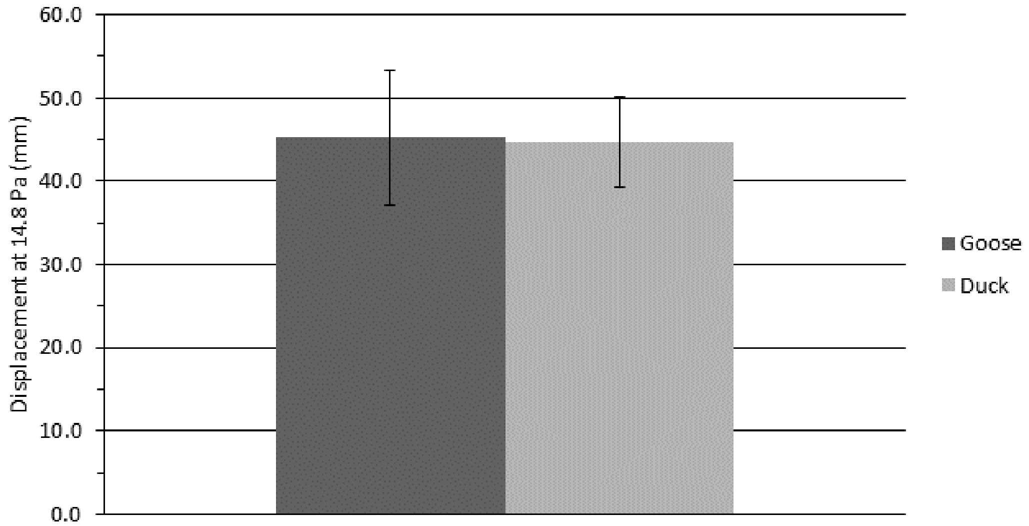


Figure 7-2 - displacement of goose and duck down up to 14.8 Pa (the pressure exerted in a fill power test)

There was no significant difference ($p > 0.05$) in displacements between the goose and duck down samples, indicating that at the pressure exerted in the fill power test, it was not possible to differentiate between the two types of down.

When comparing the work of compression at the pressures exerted in a fill power test, the same trend was seen: there was no significant difference between goose and duck down. This is shown in Figure 7-3.

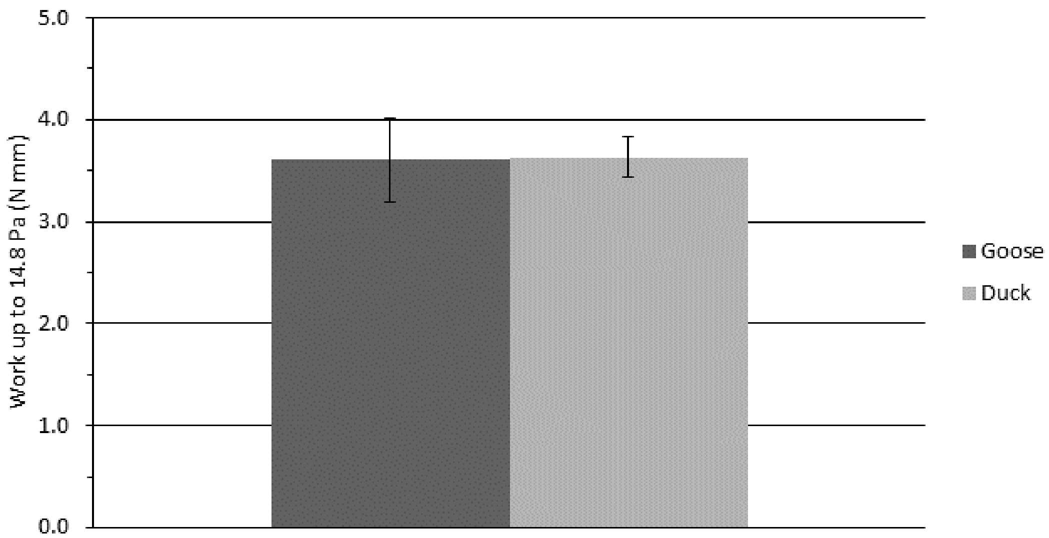


Figure 7-3 - work of compression of goose and duck down up to 14.8 Pa (the pressure exerted in a fill power test)

The relationship between the displacement up to an applied pressure of 14.8 Pa and the work up to the maximum pressures exerted in this testing is shown in goose down in Figure 7-4 and in duck down in Figure 7-5. These Figures plot the results of individual samples, rather than mean values.

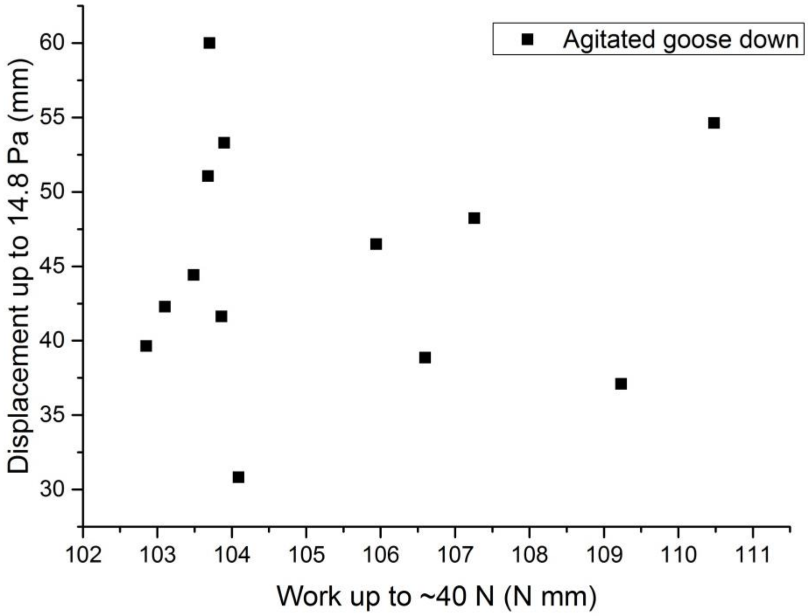


Figure 7-4 - relationship between the displacement up to the pressure exerted in fill power tests versus the work exerted in compression to a maximum force of approximately 40 N in goose down
Each data point represents an individual test of a goose down sample. There was no obvious trend relating the two terms

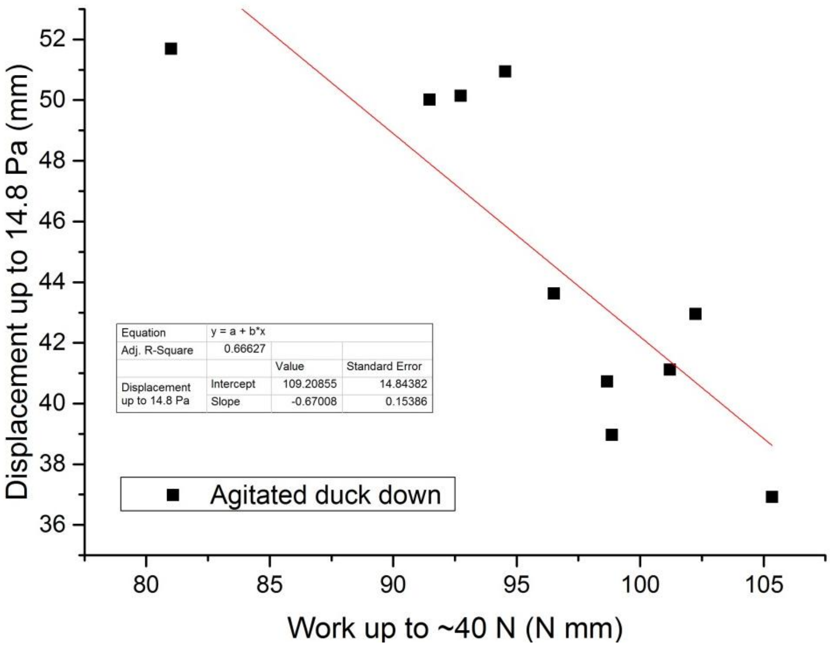


Figure 7-5 - relationship between displacement up to the pressure exerted in fill powers test versus the work exerted in the compression phase to a maximum force of approximately 40 N in duck down
Each data point represents an individual test of a duck down sample

There was no relationship between the displacement in a fill power test and the work up to 40 N in goose down but in duck down there was a quite weak inverse trend (R^2 = 0.67) which implied that materials that underwent greater displacement at low pressures (i.e. were less compression resistant) also contributed to a lower overall work when higher pressures were applied. This trend shows that the results of a fill power test may relate to the work of compressing duck down assemblies to higher pressures.

The work up to a maximum force of 14.8 Pa did not correlate with the work up to the maximal forces exerted in this test in goose down (Figure 7-6) but, as shown in Figure 7-7, the duck down samples possessed a weak negative relationship (R^2 = 0.44) between the two properties. However, it seems unlikely that a material that is highly compression resistant at low loads is poorly compression resistant at higher loads, especially when the same trend is not observed in goose down. Further testing is required to determine whether this trend is seen in other samples, and if it were, then this would show that fill power testing would be of limited use when assessing the resistance of compression of a duck down sample.

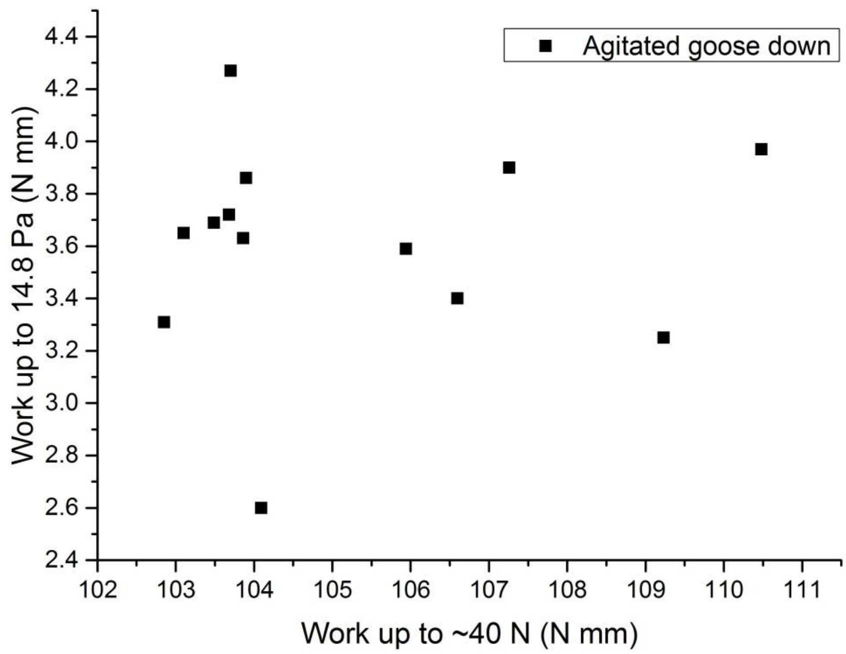


Figure 7-6 – the relationship in goose down between work up to the pressure exerted in fill power tests versus the work up to the maximum force exerted in this testing
Each data point represents an individual test of a goose down sample

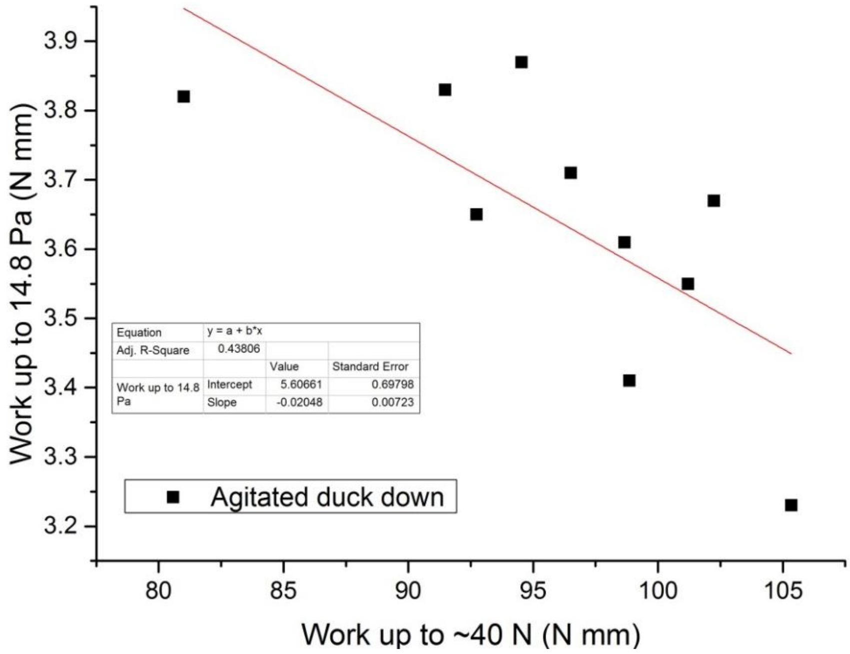


Figure 7-7 – the relationship in duck down between works up to the pressure exerted in fill power tests versus the work up to the maximum force exerted in this testing
Each data point represents an individual test of a duck down sample

Figure 7-4 to Figure 7-7 suggest that the results of fill power tests do not necessarily represent the compression resistance of a down sample under high pressures, such as those which may be exerted in a sleeping bag under a recumbent body, or between layers of clothing. This criticism of the test was also suggested by researchers in the US military (Loconti 1955), but this disapproval seems to have been ignored by the industry. Fill power is a very important test in representing the maximum loft that a down sample can achieve, and as such represents its greatest insulating ability, but it does not correlate effectively with the compression resistance of a sample under higher pressures. Further tests are required on numerous different down samples to determine whether there is a trend between the results of pressures representative of fill power tests and those at higher forces in other batches of down.

7.1.3 The compression and recovery properties of down assemblies

As shown in Figure 7-2 and Figure 7-3, the pressures of a fill power test could not differentiate between the compression resistant properties of the goose and duck down assemblies. In this section, testing of the compression properties of the down samples at higher pressures will help determine whether, in general, goose down has a higher resistance to compression than duck down. If this were the case then its significantly higher market price may be justified.

Typical compression resistance versus displacement in compression curves for goose and duck down assemblies that had been agitated before undergoing compression are shown in Figure 7-8:

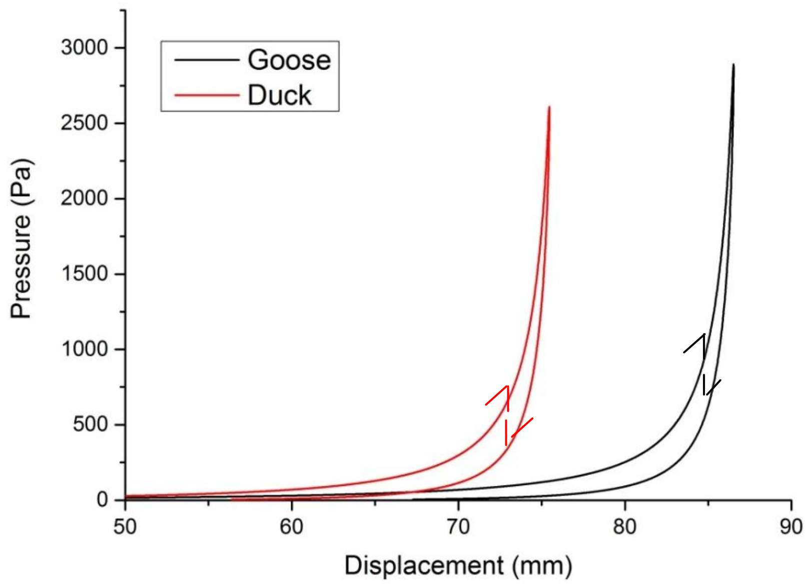


Figure 7-8 - pressure-displacement curves for goose and duck down assemblies undergoing compression and recovery
The pressures exerted at displacements below 50 mm were less than 20 Pa. The smaller displacement undergone by the duck down samples was not typical of all samples, but this sample is shown for clarity between the results of the goose and duck down

The hysteresis curves in Figure 7-8 were quite different to those recorded by Gao et al. (2010), shown in Figure 7-9, which display a greater difference between the compression and recovery phases:

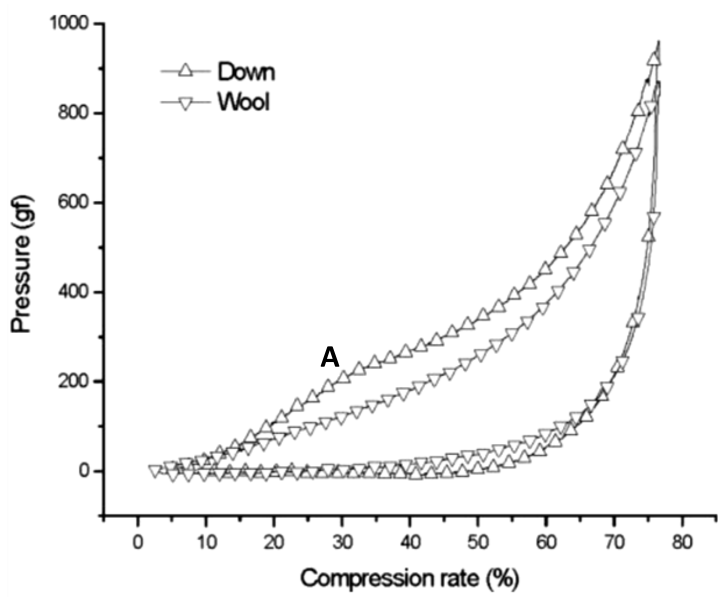


Figure 7-9 - pressure plotted against the percentage height of the sample for duck down and wool samples (modified from Gao et al. (2010))
Point 'A' refers to an increase in pressure that may coincide with an increase in air resistance

The difference in hysteresis between the results in Figure 7-8 and Figure 7-9 is either due to the effects of air resistance or test speed. Gao's testing took place at a speed of 100 mm min^{-1} compared to the 30 mm min^{-1} used here, and their test speed may be too rapid for down to recover and exert force on the load cell during the recovery phase, leading to the exaggerated hysteresis observed in their results. The effect of air pressure, which could not readily escape or re-enter their testing equipment, may also cause lesser compression recovery, and the effect of air resistance in the down sample may be observed at point 'A' in Figure 7-9, where there is a sudden increase in pressure.

Compared to the typical compression hysteresis curves of wool and other linear fibres (Dunlop 2008; Dunlop 1974), down is quite different: as shown in Figure 7-8, down undergoes very large displacements under only small pressures, while wool displaces more linearly. This low initial compression resistance has been missed by some previous reports on the compression resistance of down, such as that by Gibson (1990), which compared down to various synthetic insulations but did not consider compression under small pressures. Down's extremely low initial resistance to compression is a major factor in its success as an insulating material: it maintains extreme bulk and subsequent ability to trap air, but a small compressive force is sufficient to reduce its volume, which is useful when storing the material.

The difference between the goose and duck down samples shown in Figure 7-8 was approximately representative of the overall trend observed in the results: goose down was more compression resistant and so greater pressures were required to compress it to its minimum height. This is shown in Table 7-2, which also shows the mean value of work done in the compression phase, in the compression recovery phase, the difference in work between these two phases ($\Delta W_{(compression\text{-}recovery)}$), the percentage difference in ΔW over the work in the compression phase ($\frac{\Delta W_{(compression-recovery)}}{W_{compression}} \times 100\ \%$) , and the maximum pressure exerted during the compression phase:

Table 7-2 – summary of data from duck and goose down assemblies undergoing compression

('Agitated' refers to down that was agitated immediately before undergoing compression, and 'Un-agitated' refers to down that had previously undergone one compression cycle since last being agitated)

State of down	Source bird	Work (compression phase) (10^{-3} J)		Work (recovery phase) (10^{-3} J)		$\Delta W_{(compression-recovery)}$ (10^{-3} J)		Percentage $\Delta W_{(compression-recovery)}$ (%)		Maximum pressure (Pa)	
		Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Agitated	Goose	105.24	2.47	49.25	1.16	55.99	2.30	53.19	1.18	2847.22	64.58
	Duck	96.26	6.88	44.92	4.14	51.34	2.78	53.40	1.14	2481.94	337.50
Un-agitated	Goose	92.56	1.40	48.18	1.00	44.38	0.78	47.95	0.59	2770.14	49.31
	Duck	86.52	4.84	44.79	2.87	41.73	1.98	48.25	0.47	2523.61	174.31

The comparison between the mean works in the compression and recovery phases of the agitated goose and duck down samples are shown in Figure 7-10:

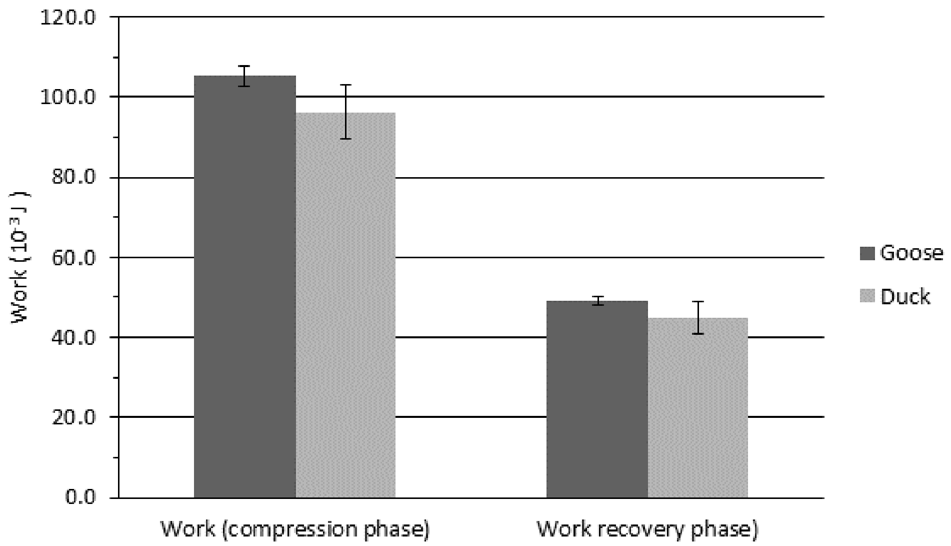


Figure 7-10 – work in the compression and recovery phases of goose and duck down that were agitated before testing
The errors bars display standard deviation values

As shown in Figure 7-10, the work to compress the down in the compression phase significantly exceeded the work in the recovery phase in both goose and duck down. This hysteresis is due to the slippage of barbs and barbules that occurs when a down assembly is compressed. It is assumed that as a down assembly is compressed the uppermost down feathers are first to bend and slide over one another (Wilde 2004). These feathers move until they become restricted by the position of other down feathers below them and their respective compression resistances increase beyond that of the feathers below them. At this point, these feathers then bend and translate to reduce the applied stress. Once all the barbs and barbules in the assembly have undergone this initial translation and bending process, they bend more drastically. It has been noted (Wilde 2004) that barbules will slide readily over one another until nodes or prongs meet, and then they become locked and the barbules may undergo extensive bending, even bending 360 ° on themselves. This stores significant elastic energy and is a contributing factor in down's excellent compression recovery. The low compression resistance of individual down plumes (as shown in section 6.2.1) means that they bend readily and this, coupled with their complex shape, stops excessive sliding of barbs and barbules that might otherwise result from compression. The sliding of barbs and barbules leads to a loss of recoverable energy and therefore hysteresis.

Single-factor ANOVA analyses proved that the difference between goose and duck down's work of compression, shown in Figure 7-10, was highly significant ($p < 0.0003$). The true density of the two down feather types is the same (as shown in section 3.2.2); their mass (and therefore presumably the number and size of barbs and barbules), as shown in section 3.2.3, is indistinguishable; and they possessed quite similar tertiary structures, as shown in section 3.3.3. As a result, the difference in compression resistance is probably due to the hollow core and non-circular cross section in goose down barbs identified in Chapters 4 and 5, which impart a greater resistance to bending due to an increased moment of inertia (as discussed in Chapter 6). There are no other major differences in internal structure and chemical composition between goose and duck down that have been found in this work which could influence compression resistance. The other possible reasons for the difference in goose and duck downs' compression resistances may alternatively be more subtle and something that needs to be identified through further investigation.

The percentage difference in ΔW divided by the work in the compression phase (i.e. the degree of unrecoverable deformation from compression) for goose and duck down samples is shown in Figure 7-11:

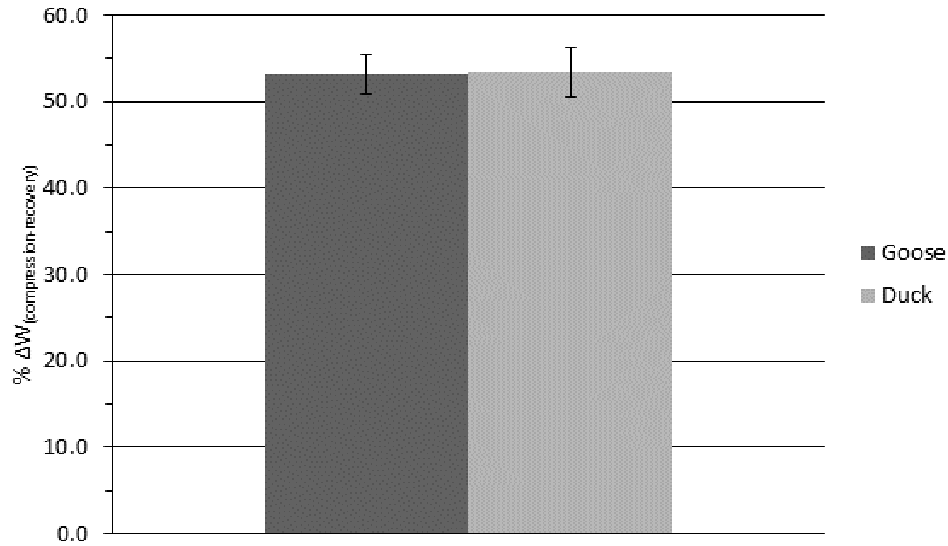


Figure 7-11 – the percentage difference in ΔW divided by the work in the compression phase (i.e. the degree of unrecoverable deformation from compression) for agitated goose and duck down

Figure 7-11 shows that the recovery from compression of the goose and duck down samples was almost identical, which implies that the energy loss to the relative movement of barbs and barbules, and friction and slippage between them, was extremely similar in goose and duck down. This corresponds to the similarity between the different types of down in the compression and recovery of individual feather plumes in section 6.2.1.

The mean maximum pressures exerted in the compression of goose and duck down are shown in Figure 7-12; and the mean displacement undergone until this point was reached is shown in Figure 7-13:

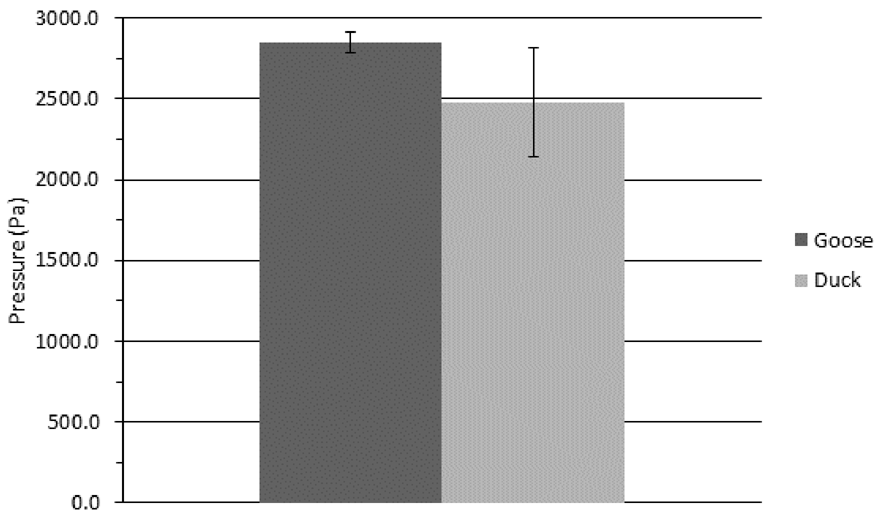


Figure 7-12 – maximum pressure exerted during the compression of goose and duck down samples to a minimum height of 1.5 mm

As shown in Figure 7-12, the maximum pressure that the testing exerted on goose down was significantly (single-factor ANOVA, $p < 0.001$) greater than on duck down when samples were compressed to a minimum thickness of 1.5 mm.

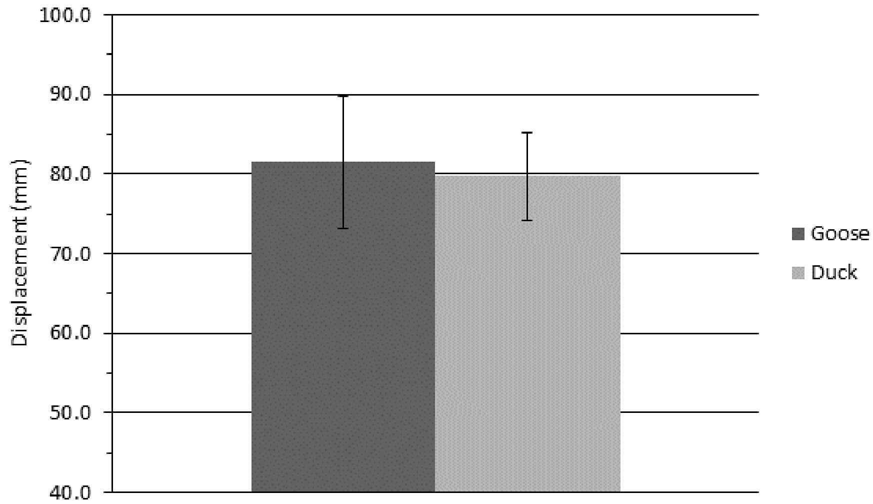


Figure 7-13 – the mean displacement to reach the maximum pressure exerted during the compression of goose and duck down samples to a height of 1.5 mm

As shown in Figure 7-13, the displacements of the duck and goose down assemblies to the point of application of maximum pressure were very similar, and they were indistinguishable by ANOVA analysis ($p > 0.05$), partly as a result of the large standard deviation values resulting from the testing. The large spread of data is because of the pre-load that had to be met before data recording took place: 2.08 Pa was used as a preload to ensure that significant compression of the sample did not occur before measurement began, but it was also small enough that contact with small local areas dense down feathers or large disturbances in air flow could overcome it. This meant that the standard deviation values in this measurement were far greater than for many of the other values from these tests. Gibson (1990) used a "touch density" of 0.002 psi (13.80 Pa) as a preload. This means that a large portion of the down's thickness would have been ignored by the test. Because the initial bulk thickness of down assemblies is one of their important characteristics, and down's initial compression resistance is small (i.e. it is highly compressible), their thickness under low pressure should be considered in these tests rather than being ignored by a high threshold preload. The reason for Gibson's high preload is that he intended to replicate the forces exerted under a recumbent male soldier (Gibson 1990), and so the down was compressed to relatively high pressures (a maximum of approximately 35 kPa).

7.1.4 The effects of agitation on the compression of goose and duck down

Agitation is known to affect the properties of goose and duck down assemblies under compression (Gao et al. 2010). Under compression, barbs and barbules lock together and agitation can disturb these interactions to allow a down assembly to loft more fully (Gao et al. 2010). In this section, the effects of agitation on goose and duck down will be determined, and whether differences exist in the effect of agitation on these two types of down.

Figures stating the compression and recovery properties of goose and duck down that had been agitated, and had not been agitated, are shown in Table 7-2 on page 178. The effect of agitation on both goose and duck down can also be seen in Figure 7-14, where agitated and un-agitated samples are compared to one-another.

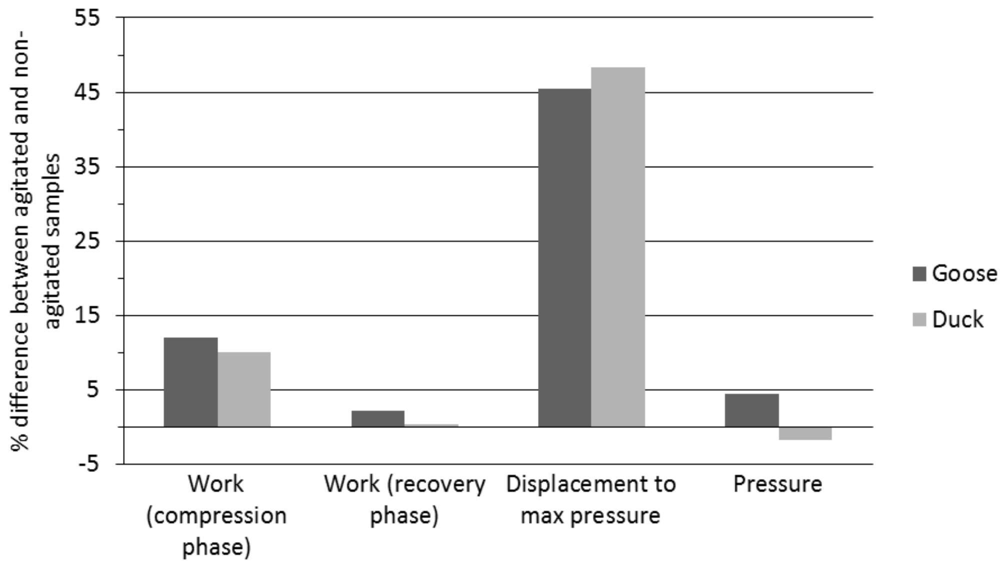


Figure 7-14 – graph showing the percentage difference between agitated and un-agitated duck and goose down assemblies undergoing compression

As shown in Figure 7-14, the effects of agitation were similar in both goose and duck down. The work in the compression phase was reduced by approximately 10-12 % when no agitation took place before compression, whereas the work in the recovery phase and the maximum pressure barely changed (less than 3 %). However, the largest difference between the agitated and non-agitated samples was in the displacement to the point of maximal pressure, which was approximately 45 % greater in the samples that had been agitated. This is a measurement of the distance travelled between the point where the preload pressure threshold of 2.08 Pa is met and the point of maximal pressure. This distance relates to the height (loft) of the sample. It is thought that during compression, down's barbs and barbules lock with one another and this causes a significant reduction in loft in the sample. External agitation supplies sufficient energy to overcome these intertwinements and allows the sample to loft to its previous maximum height. The maximum pressure during compression is not significantly changed by agitation because the applied load is sufficient to overcome the sum resistive forces in the down samples, thus flattening the sample. When the sample is very compressed, the resistance to compression is then no longer an inter-plume friction interaction, nor a measurement of the bending resistance of the barbs and barbules, but largely a compression resistance of the barbs and barbules themselves. This also explains the sharp increase in force that is observed in Figure 7-8. As $work = force \times distance$, the very small forces exerted in the initial part of a compression cycle do not contribute significantly to the overall work.

Agitation allows a down sample that has previously been compressed to increase significantly in loft and volume. It also affects the work required to compress a down sample. In use, down products that are taken from a state of compression (such as in a stuff-sack) and agitated will loft more fully and therefore insulate to a greater degree than those that are not agitated. Product manufacturers should consider telling this to consumers, who may be unaware of the effects of agitation on their down products. The effect of agitation is discussed further in section 7.1.5.

7.1.5 The effect of repeated compression cycles on down assemblies

Agitation of the down samples significantly increased both their resistance to compression and their loft, as shown in Figure 7-14, when compared to samples that had previously been compressed and not agitated. In this section, experiments were carried out to ascertain whether repeated compressions without agitation would lead to a continual reduction in compression resistance as a result of the additive effects of hystereses, or whether a plateau would be reached and further compression cycles would bring about no further change in compression resistance.

Figure 7-15 shows the mean maximum pressures in duck and goose down samples compressed to a minimum height of 1.5 mm plotted against the number of repeated compression cycles the samples had undergone since agitation.

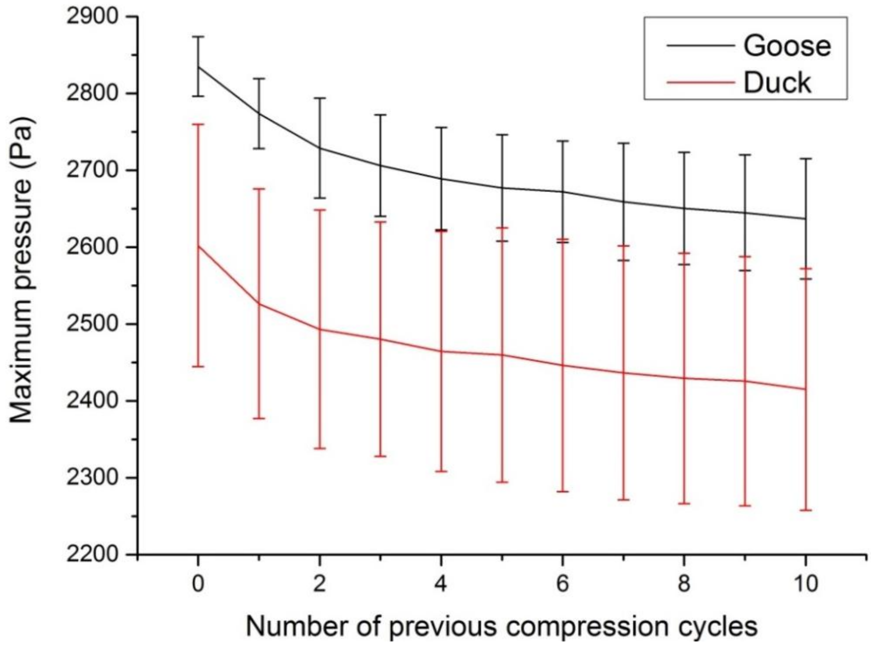


Figure 7-15 - mean maximum pressures of goose and duck down samples undergoing compression to a thickness of 1.5 mm plotted against the number of repeated compression cycles since agitation
Drop lines represent standard deviation values

A steady decrease in the maximum pressure was observed, as shown in Figure 7-15, believed to be a result of the repeated hystereses in compression. When the data from both goose and duck down was modelled using regression lines, Figure 7-16 was the result:

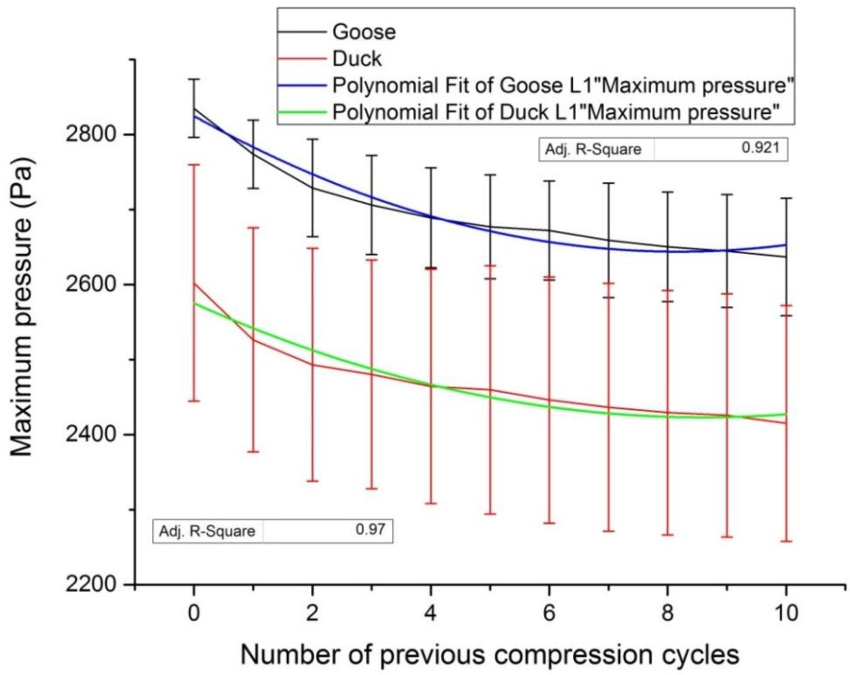


Figure 7-16 - mean maximum resistive pressures of goose and duck down samples when compressed to a thickness of 1.5 mm plotted against the number of repeated compression cycles since agitation
Regression lines are exponential (formulae shown in Equations 7-1 and 7-2)

The regression lines in Figure 7-16 were excellent fits to the data, with R^2 values of 0.921 (goose down) and 0.970 (duck down). The equation describing goose down is shown in Equation 7-1, and duck in Equation 7-2:

$$y = 2824 - 44.17x + 2.699x^2 \quad (7\text{-}1)$$

$$y = 2575 - 35.39x + 2.06x^2 \quad (7\text{-}2)$$

where y is the maximum pressure in compression in Pa and x is the number of previous compression cycles undergone by the sample.

Figure 7-17 shows the mean work in the compression and recovery phases in goose and duck down samples compressed to a thickness of 1.5 mm undergoing repeated compression cycles:

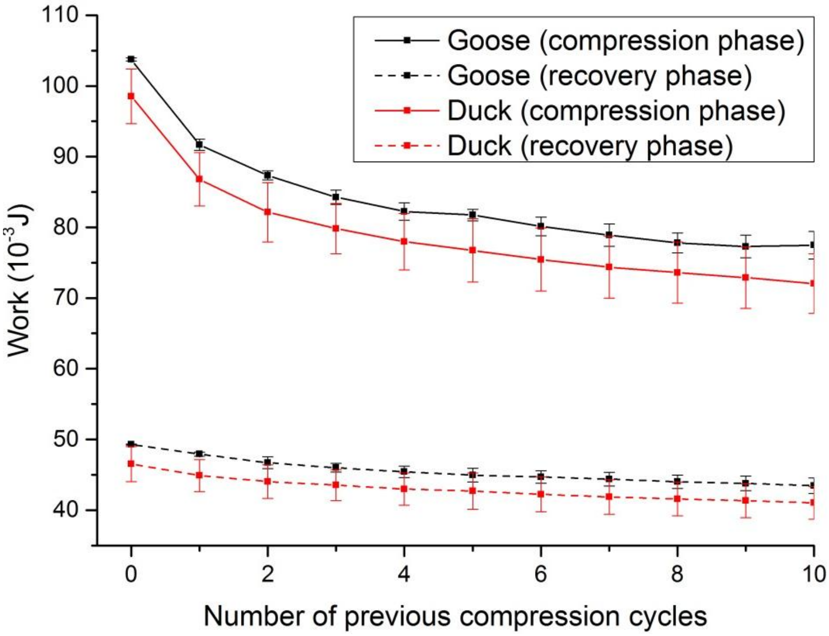


Figure 7-17 - the mean work in the compression and recovery phases of goose and duck down samples when compressed to a thickness of 1.5 mm plotted against the number of repeated compression cycles since agitation.

Modelling of the data in Figure 7-17 resulted in regression equations with excellent fit to the data (R^2 values of 0.995, 0.950, 0.999 and 0.988). The equations for work in the compression phase are shown in Equation 7-3 (goose down) and Equation 7-4 (duck down):

$$y = 24.69\, e^{\frac{-x}{1.80}} + 79.01 \quad (7\text{-}3)$$

$$y = 24.41\, e^{\frac{-x}{2.14}} + 73.21 \quad (7\text{-}4)$$

where y is the work done in compression in J and x is the number of compressions undergone by the sample.

By using Equation 7-3 and Equation 7-4 the effect of repeated compressions on the work to compress a down sample can be predicted for any number of cycles. This is shown in Figure

7-18, and suggests that after approximately 10 compression cycles, very little further change in the sample is expected:

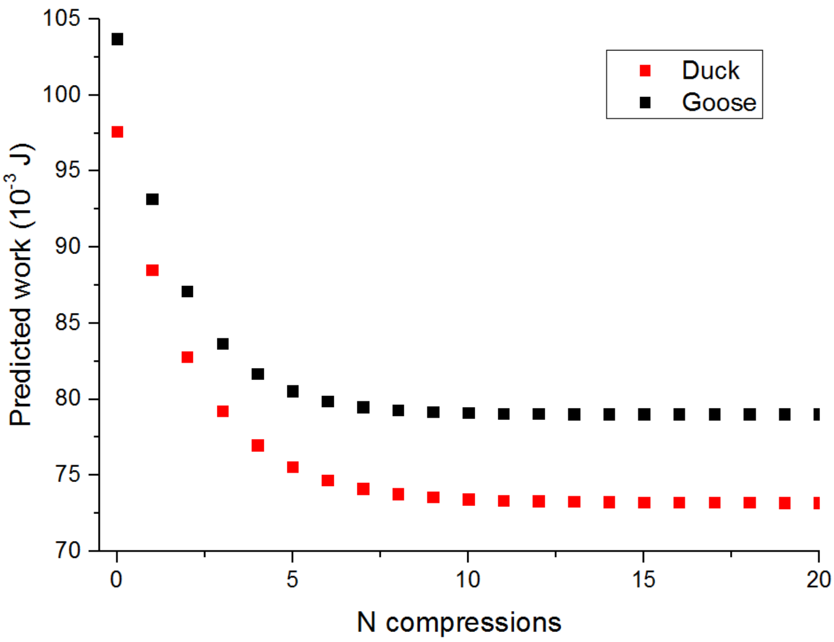


Figure 7-18 - modelled work in the compression phase of goose and duck down according to Equations 7-3 and 7-4

Following 10 compression cycles, agitation was applied to the goose and duck down samples and the samples were then compressed again. The samples required the same (single factor ANOVA, $p > 0.05$) amount of force and work to compress them as during their first compression cycle, indicating complete recovery from the previous compressions. This is shown in Figure 7-19 and Figure 7-20. As discussed in section 7.1.4, agitation is thought to untangle the barbs, nodes, and prongs and this enables the down samples to loft to their full potential, and also increases the forces that must be applied in order to compress them. These forces are thought to be influenced by the orientation of the down feathers, which are approximately randomised following agitation.

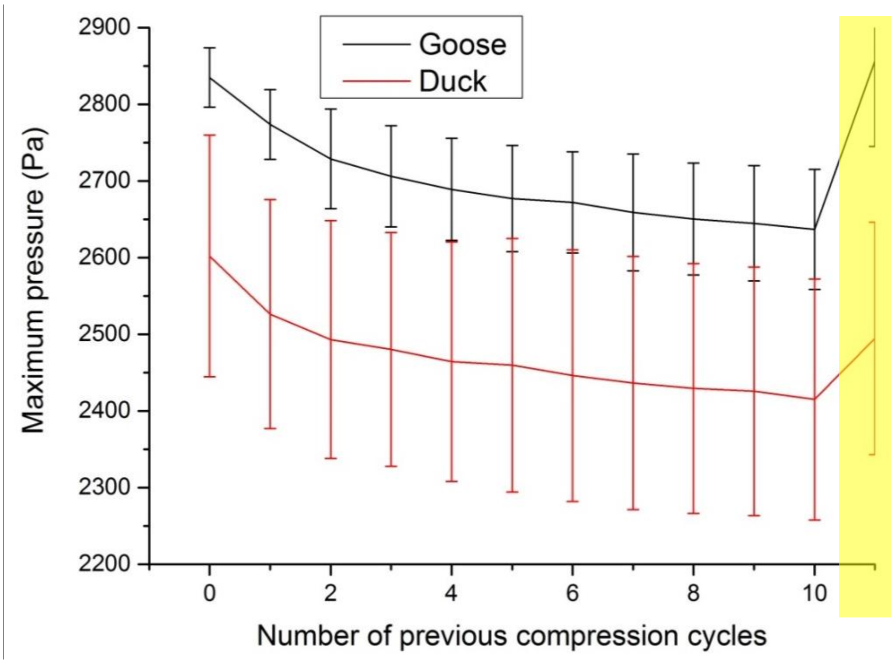


Figure 7-19 - maximum pressures in the compression of goose and duck down samples when compressed to a thickness of 1.5 mm plotted against the number of repeated compression cycles since agitation

After 10 compression cycles the samples were agitated to produce the data highlighted in yellow

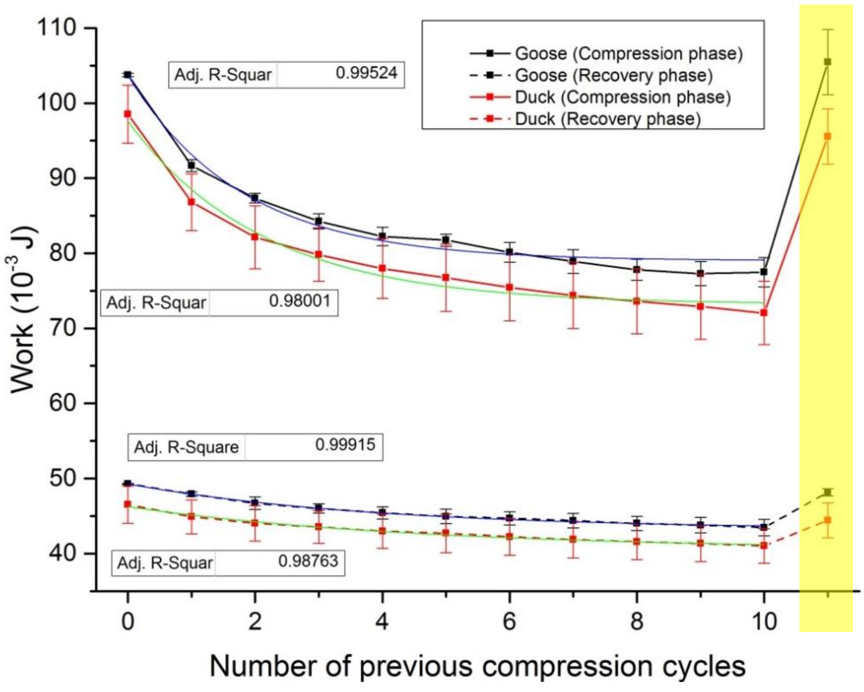


Figure 7-20 - work in the compression of goose and duck down samples when compressed to a thickness of 1.5 mm plotted against the number of repeated compression cycles since agitation

After 10 compression cycles the samples were agitated to produce the data highlighted in yellow

The effect of agitation on a down system can be considered akin to an energy transfer process where external energy from agitation is stored in the barbs and barbules. Under compression, the potential energy in the system decreases as the barbs and barbules slide and translate to positions from which recovery is not possible. Chemical reactions can be considered using the concept of activation energy (E_a): for a chemical reaction to occur, sufficient energy, signified by the activation energy, must be applied (Kotz et al. 2015). The concept of activation energy has been applied to a down assembly in Figure 7-21, and the size of the activation energy depends on the transition that is occurring. At Compression 1 in Figure 7-21, the energy required to compress the down assembly to a new state is applied. This energy is E_{a1}, and must overcome the energy barrier of barb and barbule bending and buckling, the friction between overlapping barbs and barbules, and factors such as air resistance. At Compression 2, the energy barrier again comprises barbs and barbules bending and overlapping and if activation energy 2, E_{a2}, is sufficient to overcome this energy barrier then compression can occur. To return the system to its original state, more energy must be supplied to the system; this is E_{a3}, which must exceed the total friction and interaction between and within the down. If E_{a3} is sufficient to overcome the energy barrier then the energy in the system will return to its pre-compression state.

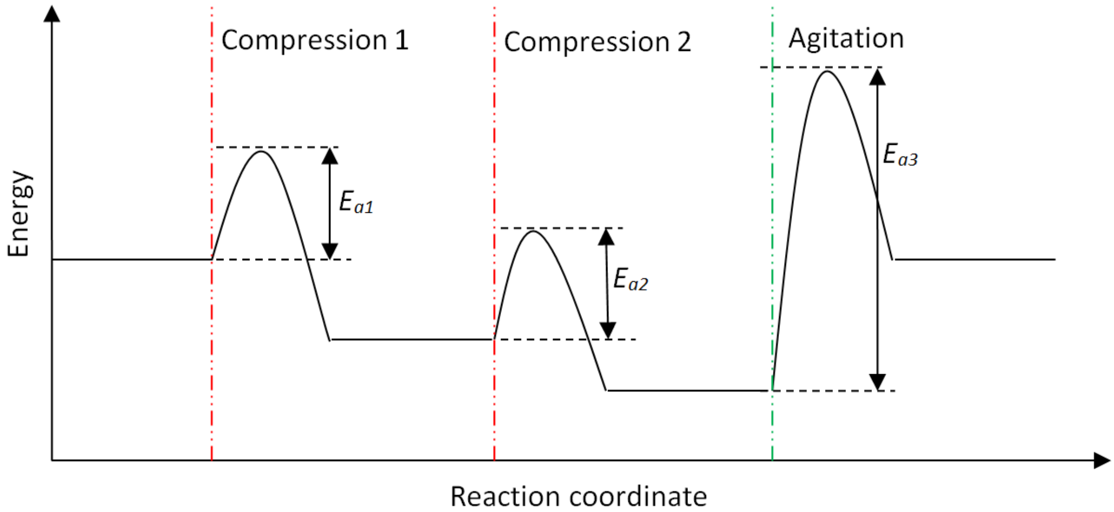


Figure 7-21 – representation of the energy changes that occur in a down system as compression cycles and agitation take place
E_{a1}, E_{a2} and E_{a3} are activation energies

7.2 Compression of down-filled squares

Down must be contained inside fabric for it to be an effective and practical insulator in outdoor equipment. These face fabrics may influence the compression of down due to their limited permeability reducing the rate at which air can be squeezed through them, and also their inherent compression and bending properties. In this section, down enclosed in face fabrics will be tested to determine whether the face fabrics influence the way that down compresses and recovers from compression. This testing also provides a way to estimate the compression characteristics of down-filled products.

Baffles impose a maximum volume that down inside them may occupy. By changing the mass of down inside them, baffles may appear under-filled (the baffle does not loft fully), optimally filled (the baffle appears full), or overfilled (the baffle looks as if it may burst). In this testing, identical test squares filled with different masses of goose and duck down were compressed and the differences in compression resistance of the two materials measured. This allowed for the effect of down's fill density (the mass of down inside a baffle of fixed maximum volume) on compression resistance to be determined, as well as any potential differences between the results of goose and duck down.

7.2.1 Experimental method

Test squares were prepared by Rab (Equip, UK) to provided specifications. They were 31 cm long and 31 cm wide and made using a box-wall construction with a strip height of 2 cm to create three separate baffles. The baffles at the side were 10 cm wide; the central baffle was 11 cm wide. A diagram of the test squares is shown in Figure 7-22. The baffles were made from 30 denier down-proof rip-stop nylon routinely used in the manufacture of high-quality sleeping bags and insulated clothing. The strips separating the insides of the baffles were made from down-proof woven nylon mesh. 18 test squares were made in total: 8 were filled with duck down, 8 with goose down, and 2 remained empty.

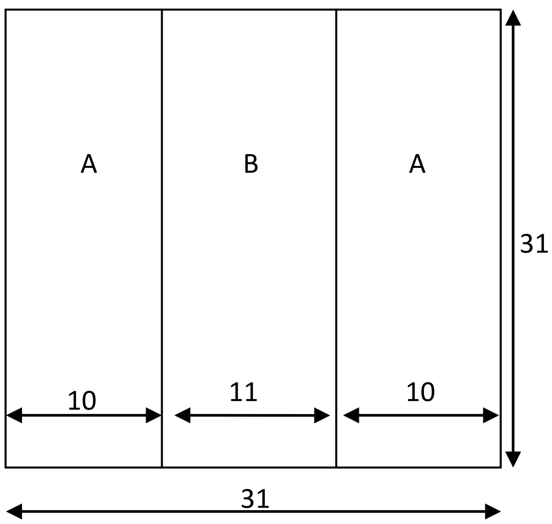


Figure 7-22 – Dimensions of the down-filled test squares
All measurements in cm

Down used to fill the baffles was supplied by Peter Kohl KG (Germany). The goose and duck downs were chosen because their fill powers (700 inches^3 oz^{-1} (equivalent to 404.6 cm^3 g^{-1}), measured following IDFB steam conditioning and according to the IDFB cylinder method) were equal, thus negating the possible effect of down's fill power on compression resistance. Each baffle in the test square was filled with down to the nearest 0.1 g and the range of filling masses was chosen to represent a large range, from an empty baffle to an overfilled baffle.

The ‘benchmark’ fill weight of a down baffle is defined by Equation 7-5 and was useful in determining the relative fill weight of different baffles:

$$'benchmark'\,fill\,weight = \frac{volume\ of\ baffle}{fill\ power} \tag{7-5}$$

For example, the volume of each ‘A’ baffle in Figure 7-22 was 620 cm^3; the volume of the ‘B’ baffle was 682 cm^3. Using these values and the fill power of 404.6 $cm^3\ g^{-1}$ allowed the ‘benchmark’ fill weight of down to fill the ‘B’ baffle to be calculated, as shown in Equation 7-6:

$$\frac{682\ \mathrm{cm}^3}{404.6\ \mathrm{cm}^3\ \mathrm{g}^{-1}} = 1.67\ \mathrm{g} \tag{7-6}$$

The same method was used in the determination of the benchmark fill weight for the ‘A’ baffles (1.53 g). The fill weights of the test squares used in this experiment were based on the benchmark fill weights of the whole test squares. The test squares’ fill weights are shown in Table 7-3 and Table 7-4:

Table 7-3 – fill weights of test squares fill with duck down and the fill weight as a percentage of the benchmark fill weight

Mean mass of down in ‘A’ baffles	Mass of down in ‘B’ baffle (g)	Total fill weight (g)	Percentage fill weight versus benchmark fill weight (%)
0	0	0	0
0.7	0.7	2.1	43
1.2	1.3	3.7	78
1.7	1.9	5.3	112
2.3	2.5	7.1	149
3.0	3.2	9.2	193
3.6	3.9	11.1	234
4.1	4.5	12.7	267
4.6	5.2	14.4	304

Table 7-4 –fill weights of test squares fill with goose down and the fill weight as a percentage of the benchmark fill weight

Mean mass of down in 'A' baffles	Mass of down in 'B' baffle (g)	Total fill weight (g)	Percentage fill weight versus benchmark fill weight (%)
0	0	0	0
0.4	0.6	1.4	30
1.2	1.4	3.8	79
1.7	1.8	5.2	110
2.2	2.5	6.9	146
3.0	3.3	9.3	195
3.6	4.0	11.3	238
4.2	4.7	13.1	275
4.8	5.4	15.0	315

The goose and duck down baffles described in Table 7-3 and Table 7-4 were tested using the Zwick Roell universal testing device described in section 2.4.2. Each sample was agitated before testing to ensure the down was distributed evenly and they were then placed on the flat steel compression plate of the Zwick Roell universal testing machine. Agitation was carried out following every test. Each sample was tested 3 times.

The 10 N load cell was equipped with 200 N jaws holding a 25 x 25 mm flat aluminium plate that compressed each sample in the centre of the central 'B' baffle (shown in Figure 7-23). Testing a small area of the baffle minimised the effects of the baffle's vertical stripping material and also reduced the forces that were measured during the test: testing a large area would have generated forces too large for the load cell. A starting separation of 40 mm between the compression plate and the base plate was used, and a 5 mN (8 Pa) preload was met before testing began. A 40 mm min^{-1} test speed was used and testing ceased once a tool separation of 1 mm or a force of 6 N (9.6 kPa) was met. These cut-off points were chosen to protect the load cell and because below 1 mm in height, very little thermal resistance would be provided by the test squares, regardless of the density of down inside them. In cases where the 6 N load cell limit was met, these tests were repeated using an identical method but making use of a 200 N load cell that was more suitable for the larger forces generated in these tests.

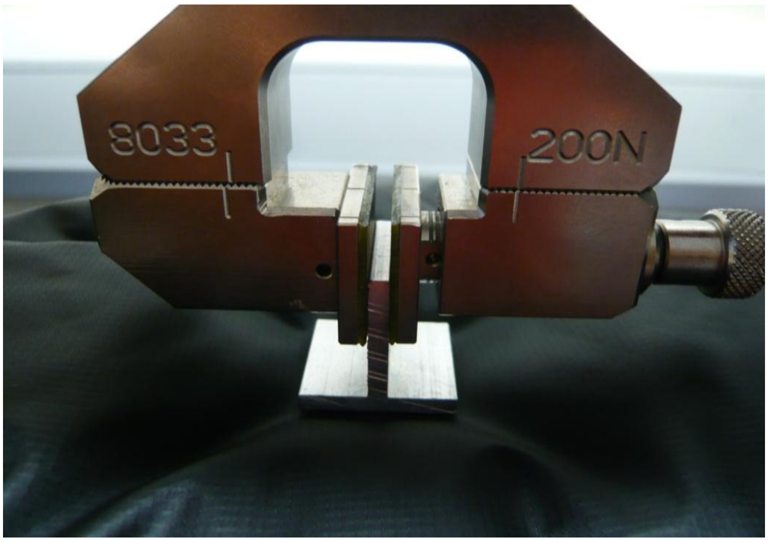


Figure 7-23 - compression of a down-filled test square using an aluminium plate held in 200 N jaws
Use of a small plate minimised the effect of the stripping between baffles on the compression of the down

7.2.2 Results and discussion

A typical pressure-compression graph for the compression of the goose and duck down baffles is shown in Figure 7-24:

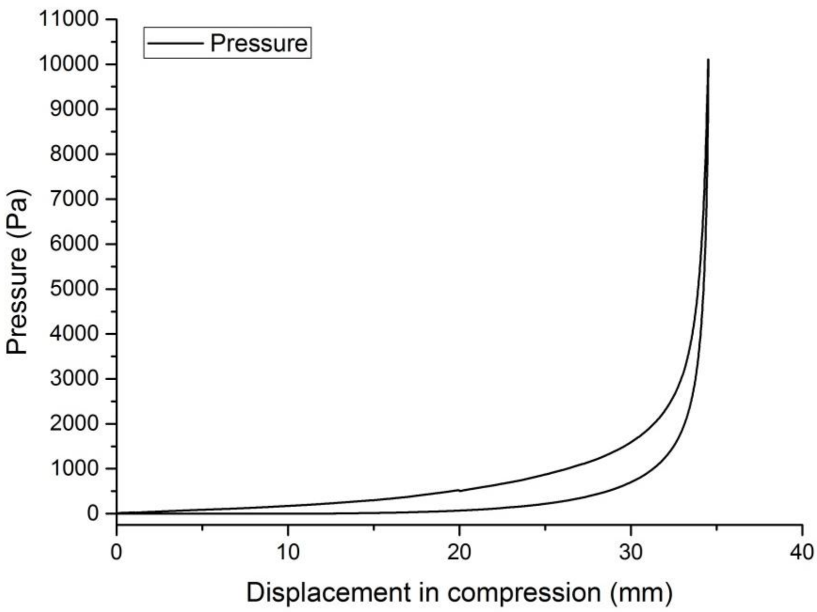


Figure 7-24 - typical pressure-displacement graph for a down-filled baffle undergoing compression

The pre-load of 8 Pa was met as soon as the aluminium plate made contact with the fabric of the sample, regardless of the density of the down inside the baffles. As shown in Figure 7-24, the pressure then increased very gradually before rising steeply as the minimum sample thickness of 1 mm was approached, resulting in a typical pressure-compression curve for the filled test squares that was very similar in shape to the equivalent curve resulting from compression of the down feather assemblies in section 7.1. The main difference between the results of the down-filled squares and those of the down feather assemblies was the reduced distance that the down-filled squares underwent during compression before the force began to rise significantly. This was a result of the baffles limiting the initial thickness of the down and the weight of the squares' fabrics keeping the down in a permanent state of slight compression. This signifies the importance of the weight of a fabric on the performance of a down baffle: a heavy face fabric will compress the down it contains and thus reduce its total loft; a lightweight fabric with excellent drape will not have such a noticeable effect.

A summary of the data from the squares containing the goose and duck down and undergoing compression is shown in Table 7-5 and Table 7-6, respectively:

Table 7-5 - results from test squares containing goose down undergoing compression

Total fill weight (goose down)	**Work (compression phase)**		**Work (recovery phase)**		**$\Delta W_{(application-removal)}$**		**Percentage $\Delta W_{(application-removal)}$**		**Maximum pressure**	
(g)	**(10^{-3} J)**		**(10^{-3} J)**		**(10^{-3} J)**		**(%)**		**(Pa)**	
	Mean	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**	**Mean**	**SD**
15.0	44.22	4.29	20.32	2.39	24.00	2.52	54.29	2.60	33640.0	7465.1
13.1	36.09	0.64	16.19	0.10	19.96	0.77	55.29	1.17	22932.8	184.8
11.3	28.77	4.06	12.83	1.80	15.98	2.27	55.53	0.80	17435.2	3182.9
9.3	19.20	0.64	8.28	0.33	10.97	0.30	57.13	0.47	11274.7	585.0
6.9	16.30	2.35	7.01	1.11	9.29	1.32	57.00	2.04	9253.8	1976.2
5.2	9.36	1.23	4.14	0.51	5.23	0.74	55.82	0.99	5222.2	632.5
3.8	4.33	0.16	1.63	0.04	2.71	0.17	62.52	1.76	2474.7	133.3
1.4	1.52	0.89	0.57	0.39	0.95	0.50	62.47	5.48	1146.9	661.4
0.0	0.07	0.01	0.02	0.00	0.05	0.01	75.08	8.63	57.8	19.0

Table 7-6 – results from test squares containing duck down undergoing compression

Total fill weight (duck down)	Work (compression phase)		Work (recovery phase)		$\Delta W_{(application-removal)}$		Percentage $\Delta W_{(application-removal)}$		Maximum pressure	
(g)	(10^{-3} J)		(10^{-3} J)		(10^{-3} J)		(%)		(Pa)	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
14.4	35.68	8.16	16.70	4.01	18.98	4.14	53.20	0.57	32280.0	5810.7
12.7	25.24	3.22	11.00	1.84	14.24	1.42	56.43	1.86	21051.2	5627.8
11.1	26.16	7.07	11.27	3.29	14.90	3.80	56.94	1.64	22608.0	6909.8
9.2	21.56	3.06	9.33	1.59	12.23	1.71	56.75	3.14	18632.0	4341.4
7.1	13.45	4.45	5.01	1.28	5.01	1.27	48.17	1.81	7427.0	1918.9
5.3	7.79	1.49	3.21	0.74	3.21	0.75	58.80	1.89	4355.7	1145.8
3.7	9.16	3.11	3.96	1.45	3.96	1.66	56.90	1.10	5794.1	2438.2
2.1	1.42	0.47	0.53	0.22	0.53	0.244	63.20	4.17	1075.8	251.4
0.0	0.07	0.01	0.02	0.00	0.05	0.01	75.08	8.63	57.8	19.0

The mean maximum pressures exerted in the tests are shown for goose and duck down in Figure 7-25 and Figure 7-26, respectively:

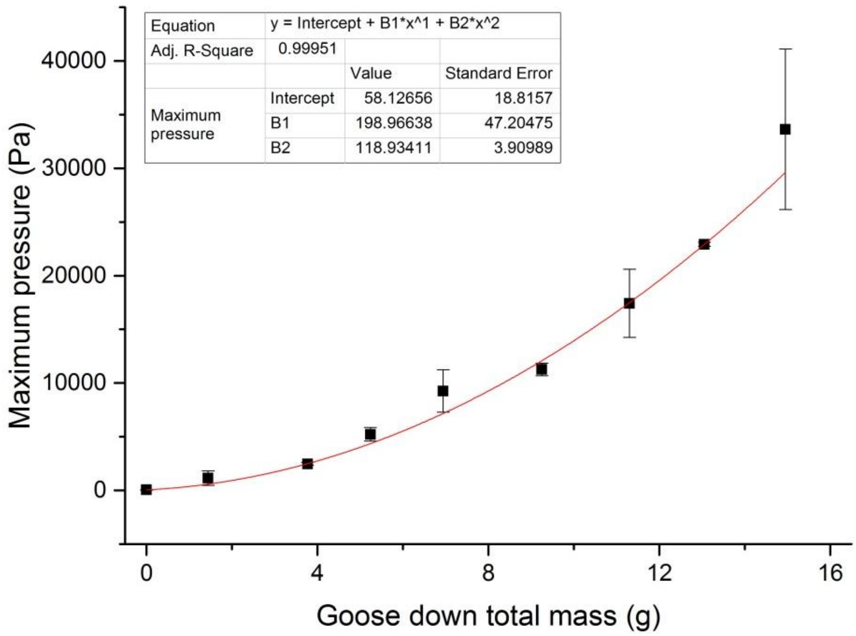


Figure 7-25 – the maximum pressures exerted in the compression of goose down test squares plotted against their total mass of down
The bars indicate SD values

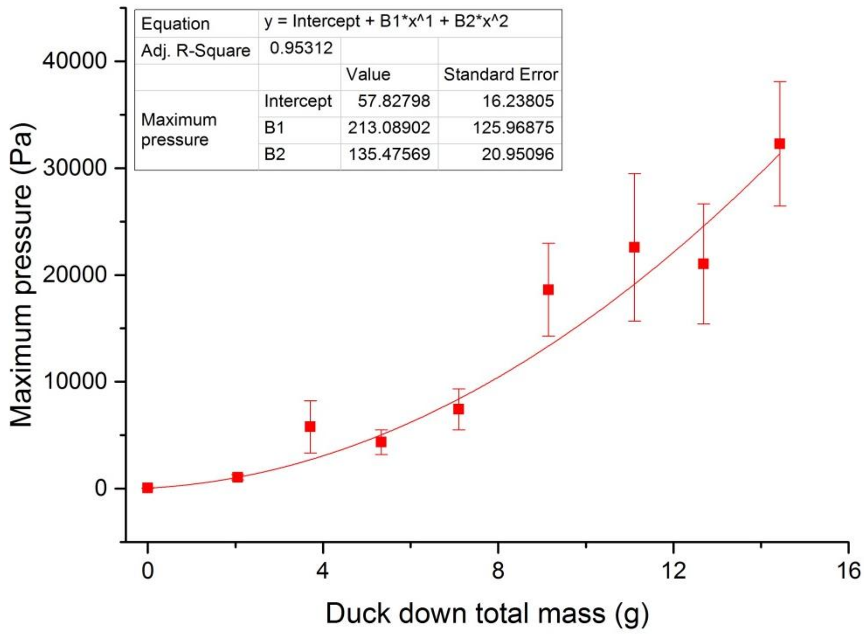


Figure 7-26 – the maximum pressures exerted in the compression of duck down test squares plotted against their total mass of down
SD values are also given

As shown in Figure 7-25 and Figure 7-26, the maximum pressures required to compress the goose and down baffles were each modelled effectively by polynomial curves (r^2 = 0.99 for goose down; r^2 = 0.95 for duck down). The standard deviation values were relatively small, particularly for the samples with lower fill weights. There was no significant difference in the maximum pressure during compression between the goose and duck down samples, at any of the different fill weights, which contradicts the result found in section 7.1 and shown in Figure 7-12 when down was tested in absence of face fabrics. This may be a result of using goose and duck down samples of the same fill powers in this test, or it may also be a result of the face fabrics negating any difference between the two down samples' compression resistances.

The mean work values in the compression phase for the goose and duck down-filled squares are shown in Figure 7-27 and Figure 7-28, respectively. Each was best-modelled by polynomial fits. Both sets of data, showing individual values rather than mean values, are also shown in Figure 7-29:

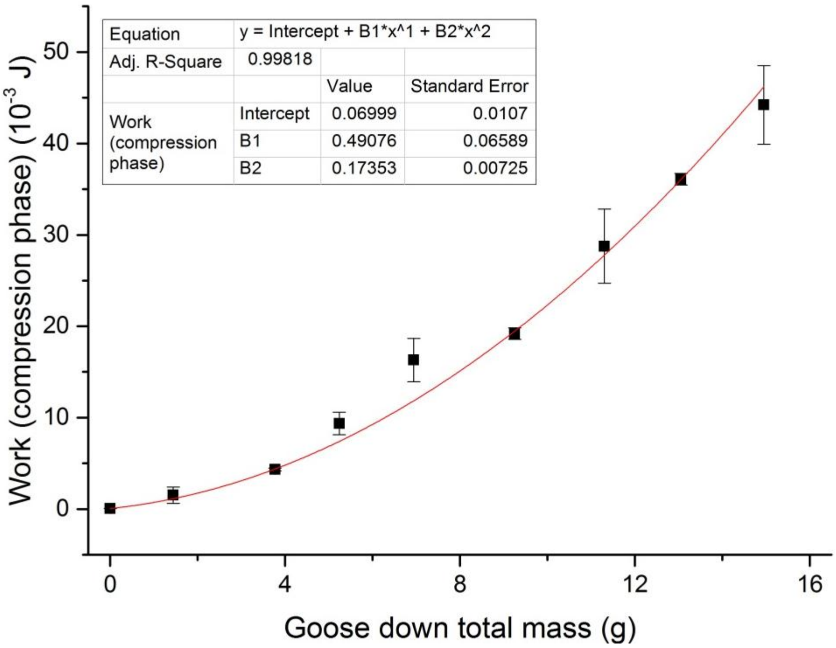


Figure 7-27 – the mean work in the compression phase when compressing goose-down-filled test squares plotted against their total mass of down

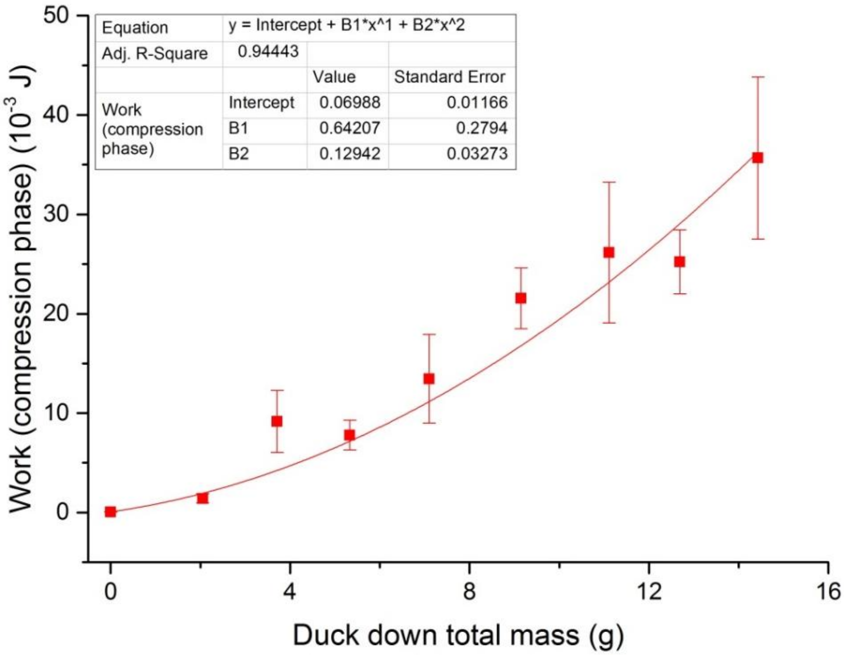


Figure 7-28 – the work in the compression phase when compressing duck-down-filled test squares plotted against their total mass of down

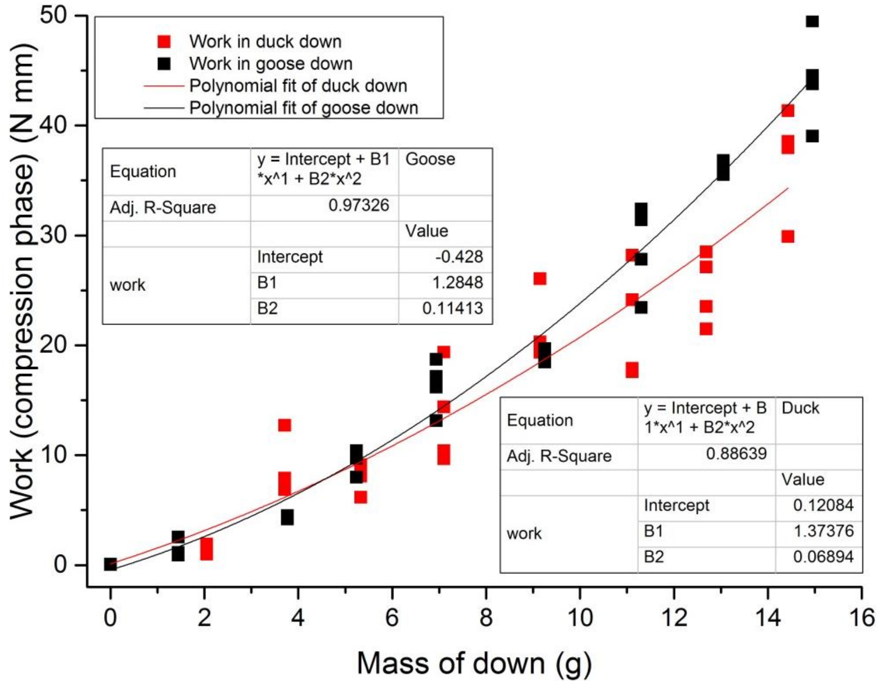


Figure 7-29 – the work in the compression phase when compressing duck and goose down test squares plotted against their total mass of down
Values shown are for individual tests, not mean values

Figure 7-29 shows that the goose down samples of higher fill weights had a greater resistance to compression than the duck down samples had, bolstering the findings from section 7.1.3. This is especially remarkable here, as the analysed down samples have identical fill powers and are therefore expected to be very similar in their compression resistances. This implies that even if the fill powers of two down samples are the same, the compression resistance at high pressures may be higher in goose down than duck down. This is a potentially crucial result for the industry and may further undermine the fill power test's ability to represent compression resistance at high pressures.

The percentage difference in work between the compression and recovery phases as a function of the work in the compression phase is shown in Figure 7-30 (goose down) and Figure 7-31 (duck down):

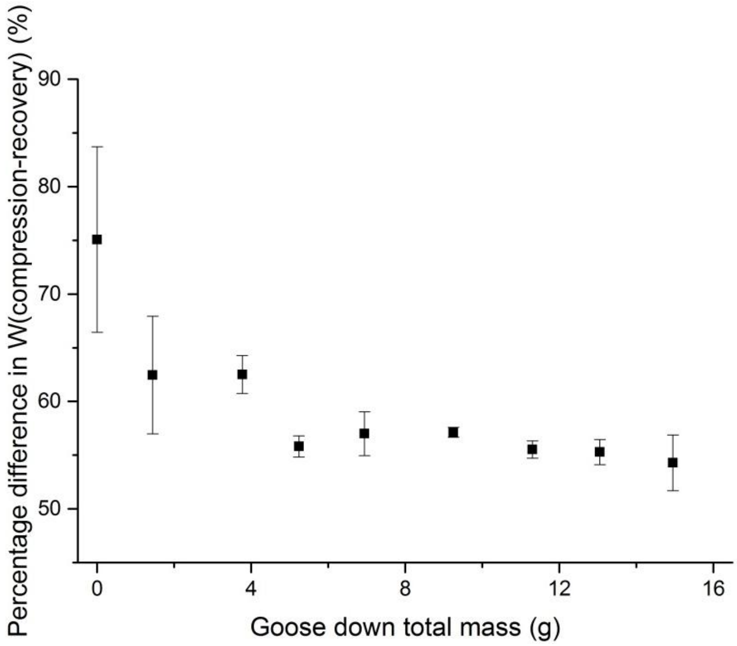


Figure 7-30 – the percentage difference in work between the work in the compression and recovery phases in the goose down test squares, plotted against their total mass of down

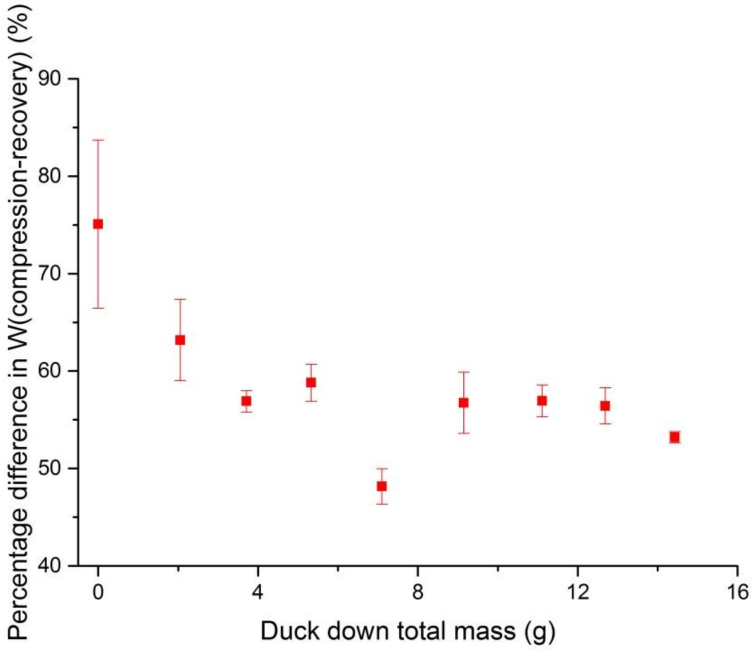


Figure 7-31 – the percentage difference in work between the work in the compression and recovery phases in the duck down test squares, plotted against their total mass of down

As had been seen in the compression testing of individual down plumes (section 6.2.1), and in the testing of the down assemblies (section 7.1), the percentage difference between the work in the compression and recovery phases was almost identical in each of the duck and goose down samples of equal fill weight. There was also a trend towards better recovery at higher fill weights in both goose and duck down. The baffles with low fill-weight had extremely poor compression recovery, but if the baffle was 'overfilled' (significantly more down in the baffle versus the amount suggested by the ‘benchmark’ values from fill power) as with the heaviest baffles in this test, their compression recovery improved. This is because a greater density of down feathers meant that barbs and barbules could barely slip over one another (resulting in non-recoverable compression) before coming into contact with another part of a down feather.

The compression resistance of the loose down assemblies tested in section 7.1 and the squares tested here have many remarkable similarities. Firstly, in both the squares and the loose down tests, the work to compress the goose down was greater than to compress the duck down. The percentage difference in work between the compression and recovery phases was also very similar in both types of test (53 % in the tests on loose down, and 54 ± 1 % in the squares of greatest fill weight). This shows the small effect of a face fabric on down's performance, as long as sufficient down is present to overcome the weight of the fabric.

When testing loose down in section 7.1, 1.8 g of down was used, and when directly comparing those results with the figures from the 'B' baffles filled with 1.8-1.9 g of down, differences in maximum pressure and in work are apparent, but these differences are largely because of the testing methods employed: when testing the down-filled squares, a small area was compressed to 1 mm of thickness (including the thickness of the face fabrics) whereas the loose down was compressed under a much larger area but to a minimum thickness of 1.5 mm. This meant that the down-filled squares underwent greater maximum pressures but lower work in compression and recovery.

The consistent differences in compression resistance between goose and duck down, regardless of fill power, can be attributed to their differences in morphology. Though their structures are very similar in regards to barb and barbule diameter, and overall feather mass, the differences in geometry identified in Chapters 3, 4, and 5 are very influential: goose down barbs' hollow cross sections increases their resistance to bending, as described in section 6.2.2, and this, allied to their approximately-triangular cross section which reduces chance of buckling, increases compression resistance.

7.3 The thermal resistance of down-filled squares

The thermal resistance of a down product is extremely important in determining its performance. However, testing of whole sleeping bags or jackets is expensive, complex, and may be impractical for companies in the outdoor industry given the time constraints of manufacturing deadlines. For example, the standard 'EN 13537: 2012' for testing sleeping bags (British Standards Institute 2012) requires the use of a heated, zoned manikin which is expensive and difficult to use correctly (McCullough 2009). Here, by using down-filled squares representative of larger products, results will be generated that may correlate with more complex systems such as sleeping bags, but without such difficult and expensive testing. The influence of down's density will also be related to thermal resistance, and the possibility of a difference between goose and duck down's thermal resistances will be investigated.

The relationship between thermal properties and down fill density has previously been investigated by researchers in the US military and in confidential commercial projects (Adams & Caffin 2012; Zarr 2000; Gibson et al. 2007; Gibson 1990; Fuller 2012). However, previous work in this area has rarely carried out multiple repeats of tests and at present, many manufacturers still use trial-and-error approaches to determine the best fill weights to use in their products. As a result, a more scientific mode of determining the relationship between down's fill density and thermal resistance, or thermal resistance per unit mass, may be very helpful in both maximising performance and reducing costs.

It is hypothesised that thermal resistance will increase linearly with fill weight until a baffle reaches its near-maximum thickness. This is because thermal resistance is linearly related to thickness (Rees 1941; Backer 1948). Once the baffle can increase only very marginally in thickness, thermal resistance will continue to rise as the resistance to radiative heat transfer increases. A plateau in thermal resistance will eventually be reached when the decrease in radiative heat loss is balanced by an increase in thermal conductivity: this will occur when the volume fraction of down feathers is sufficient to force air from the test square. As down (keratin) has a thermal conductivity of 0.19 W m^{-1} K^{-1} (Baxter 1946), and air has a thermal conductivity of 0.026 W m^{-1} K^{-1} at 300 °K and 1 atmosphere of pressure (Kadoya et al. 1985), replacing still air with keratinous down feathers will increase the overall thermal conductivity of the assembly. The expected relationship between thermal resistance and down density is summarised in Figure 7-32:

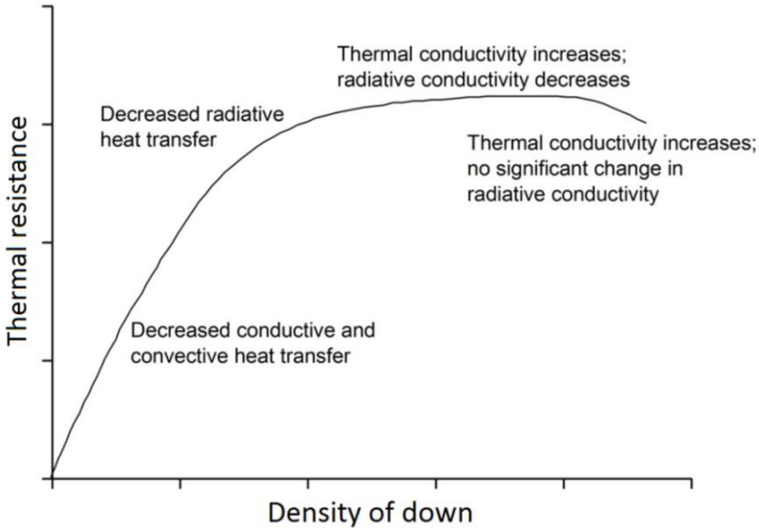


Figure 7-32 – idealised graph depicting the hypothesised relationship between thermal resistance and the density of down in a product or down-filled square

The method described in section 2.6 was used to analyse the down feather baffles described in section 7.2. Three measurements were made of each sample. The sweating guarded hotplate was used for this testing, rather than the more conventional tog test (BS 4745: 2005 (British Standards Institute 2005)) because round samples (as would conventionally be used in tog testing) were more difficult to manufacture with accurate fill densities and the sweating guarded hotplate test is much faster than tog testing, allowing more repeats to be carried out. In addition, the interface of a modern sweating guarded hotplate (Measurement Technology Northwest 2012) allows for more accurate data collection with a higher capture rate.

7.3.1 Results and discussion

The fill weights of the test squares and their relation to the 'benchmark' fill weights described in section 7.2.1 are shown in Table 7-7 (duck down) and Table 7-8 (goose down). The mean and standard deviation values of the resistance to conductive transfer (R_{CT}) are also shown:

Table 7-7 – mean and SD R_{CT} results of test squares filled with different masses of duck down

Total fill weight (g)	Percentage of fill weight versus benchmark fill weight (%)	Mean R_{CT} values (°C m^2 W^{-1})	SD R_{CT} values (°C m^2 W^{-1})
0	0	0.08	0.014
2.1	43	0.18	0.009
3.7	78	0.25	0.015
5.3	112	0.32	0.021
7.1	149	0.38	0.012
9.2	193	0.45	0.016
11.1	234	0.49	0.013
12.7	267	0.51	0.018
14.4	304	0.55	0.012

Table 7-8 - mean and SD R_{CT} results of test squares filled with different masses of goose down

Total fill weight (g)	Percentage of fill weight versus benchmark fill weight (%)	Mean R_{CT} values (°C m^2 W^{-1})	SD R_{CT} values (°C m^2 W^{-1})
0	0	0.08	0.014
1.4	30	0.21	0.002
3.8	79	0.27	0.025
5.2	110	0.33	0.006
6.9	146	0.41	0.005
9.3	195	0.46	0.005
11.3	238	0.50	0.009
13.1	275	0.51	0.031
15.0	315	0.57	0.003

The correlation between mean R_{CT} and the total duck down mass was modelled extremely well (R^2 = 0.999) by the binomial equation $y = -0.00149x^2 + 0.054x + 0.077$. The data is shown in Figure 7-33:

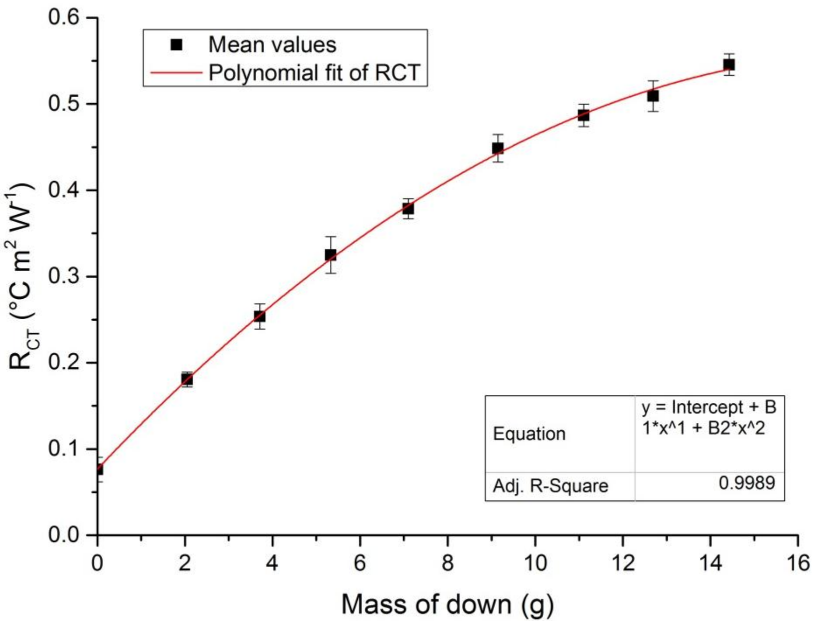


Figure 7-33 – mean values of R_{CT} plotted against the total mass of duck down in each test square
Error bars are standard deviation values. Equation for line is y = $-0.00149x^2 + 0.054x + 0.077$

The mean R_{CT} values plotted against the mass of goose down in each square were also modelled extremely well ($R^2 = 0.983$) by a binomial fit ($y = -0.00155x^2 + 0.054x + 0.1$), as shown in Figure 7-34:

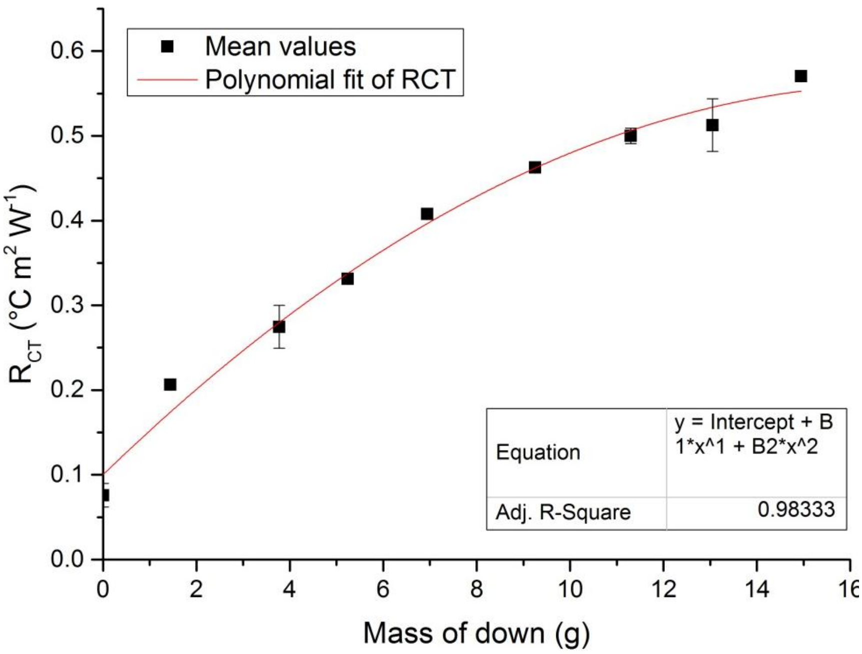


Figure 7-34 - mean values of R_{CT} plotted against the total mass of goose down in the test squares
Error bars are standard deviation values. Equation for line is y = $-0.00155x^2 + 0.054x + 0.1$

The highly reproducible results with low standard deviation values indicated an excellent test method: while the sweating guarded hotplate is not suitable for the testing of most sleeping bags, jackets, or quilts, by using test squares specifically sized to fit the testing apparatus the performance of these products can be simulated reproducibly and quickly. This is likely to be amongst the first times that the sweating guarded hotplate has been used to test a series of down-filled test squares representative of outdoor products.

Figure 7-33 and Figure 7-34 show that as the mass of down increases in the test square, so too does the resistance to conductive transfer. At low fill weights the increase is most apparent as the baffle increases in thickness and therefore the thickness of air that the down traps increases. Once the baffle is fully lofted (reached at approximately 10 g of down in the test square) the increase in thermal resistance lessens, though thermal resistance continues to rise. This is not likely to be due to changes in conduction, as the thickness of the baffle has already approached its maximum; or convection, as natural convection cannot take place inside a down assembly (Dawson et al. 1999). Therefore a decrease in radiative heat transfer is likely to be the cause of this increase in R_{CT}. The theoretical plateau and drop in R_{CT} depicted in Figure 7-32 did not occur in the measured samples, implying that even if baffles are highly 'overfilled' (300 % of their benchmark value), their thermal resistance continues to rise. One premium-quality down equipment manufacturer uses a fill weight 180 % of that derived from fill power (Manwaring 2014), and this is relatively typical of down equipment manufacturers: certainly, they are not at risk of 'overfilling' and additional down reducing the thermal performance of their products.

The R_{CT} values for each individual test square for both goose and duck down are plotted against the mass of each test square (including the fabric) are shown in Figure 7-35:

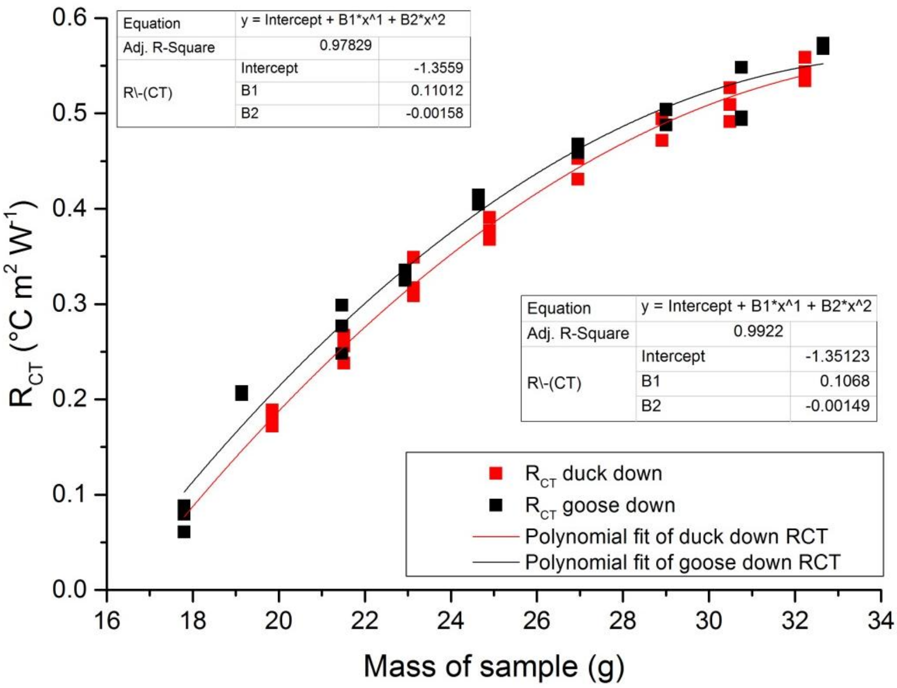


Figure 7-35 – R_{CT} results of all tests as a function of the masses of the down-filled squares

The data in Figure 7-35 shows the similarity between the performance of the goose and duck down samples. The goose down samples' R_{CT} values are very marginally higher than the duck down samples' but this difference was not significant (single-factor ANOVA, $p = 0.63$) when comparing each equivalent down-filled square.

From the R_{CT} results, one might conclude that increasing the amount of down in a product has no negative consequences and simply increases thermal resistance. This mindset could then be extrapolated into the manufacture of sleeping bags or insulated clothing where adding greater and greater quantities of down would increase their warmth. However, thermal resistance is arguably less important to a product than the thermal resistance per unit mass. This is because hikers, mountaineers, and those venturing into hostile environments must often carry their equipment, and transporting any extra mass, particularly uphill, increases fatigue. Thus, the R_{CT} per unit mass for the duck down samples was plotted against the total mass of each test square, including the fabric, in Figure 7-36. The equivalent graph for goose down is shown in Figure 7-37. The R_{CT} results of each individual measurement, rather than mean values, are plotted.

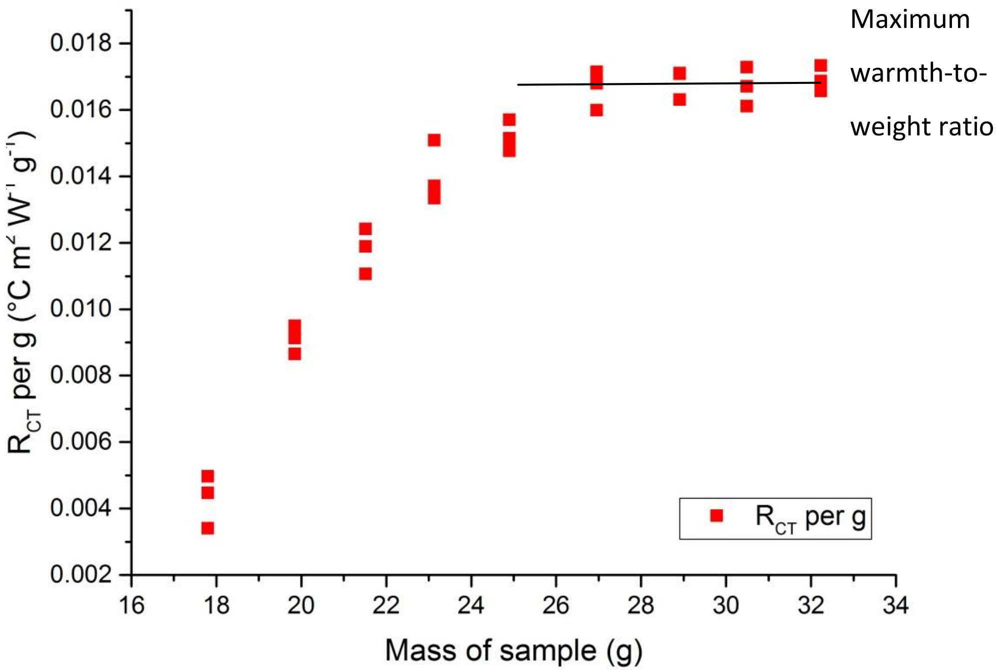


Figure 7-36 – R_{CT} per unit mass (warmth to weight ratio) plotted against the total mass of each test square filled with duck down
A line has been added to indicate the maximum warmth to weight ratio

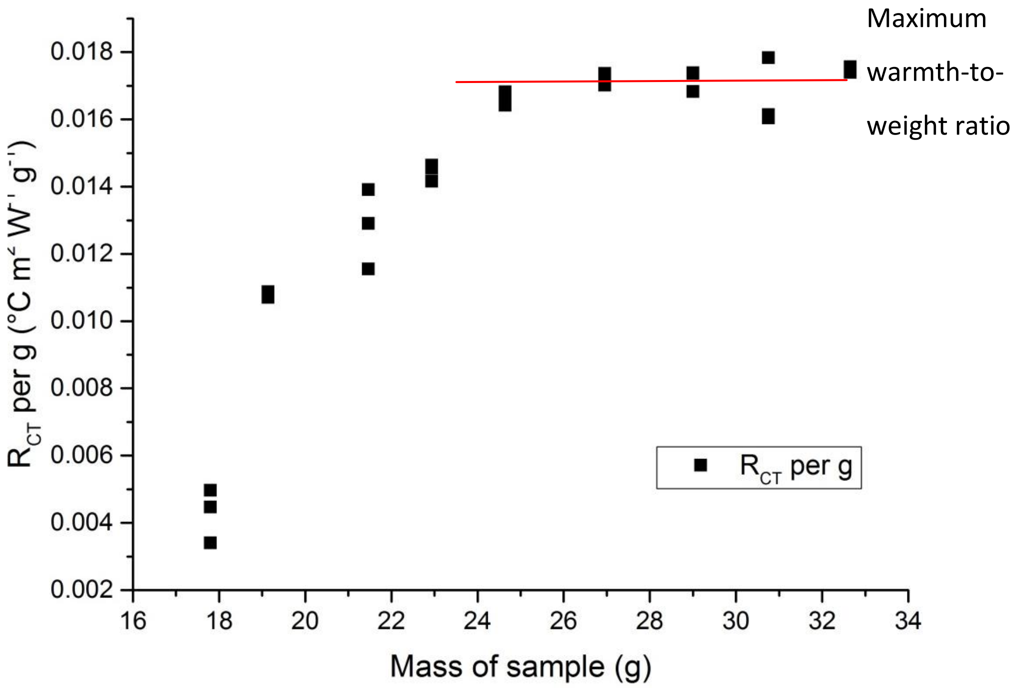


Figure 7-37 - R_{CT} per unit mass plotted against the mass of each sample filled with goose down A line has been added to indicate the maximum warmth to weight ratio

Figure 7-36 and Figure 7-37 show that thermal resistance per unit mass increases steeply until approximately 27 g total mass is reached, corresponding to approximately 190 % of the benchmark down fill weight. At this point the thermal resistance per unit mass remains relatively constant, and below this point the baffle could be regarded as under-filled as it fails to achieve maximal thermal resistance per unit mass. Knowing that an optimal thermal resistance per unit mass has been reached is important for all high-performance outdoor products. The limitation of this testing is that, while it demonstrates that an optimal R_{CT} per unit mass exists, it does not show exactly where this is for real products: their design and face fabrics both influence their total mass, and by changing these parameters, it affects the amount of down required to meet the optimum R_{CT} per unit mass. For example, had the fabrics and construction used in making these test squares been much heavier then the R_{CT} per gram would not have reached a plateau at the values observed here, and more down would have been required to reach this value. As a consequence, the results here regarding optimal R_{CT} per gram are not necessarily directly applicable to real products, though they demonstrate that a plateau of R_{CT} per units mass can be reached before any loss is observed. However, as the R_{CT} does not seem to decrease until the baffle is extremely overfilled (indeed if it decreases at all), it could be concluded that, if in doubt, more down should be added to a baffle as this will increase thermal resistance and will not reduce the thermal resistance per unit mass; alternatively, the baffles should be made smaller by reducing the stripping height or reducing their width in the case of stitch-through baffles to increase the density of down and reduce the weight of the equipment's construction.

The data from Figure 7-36 and Figure 7-37 is combined in Figure 7-38:

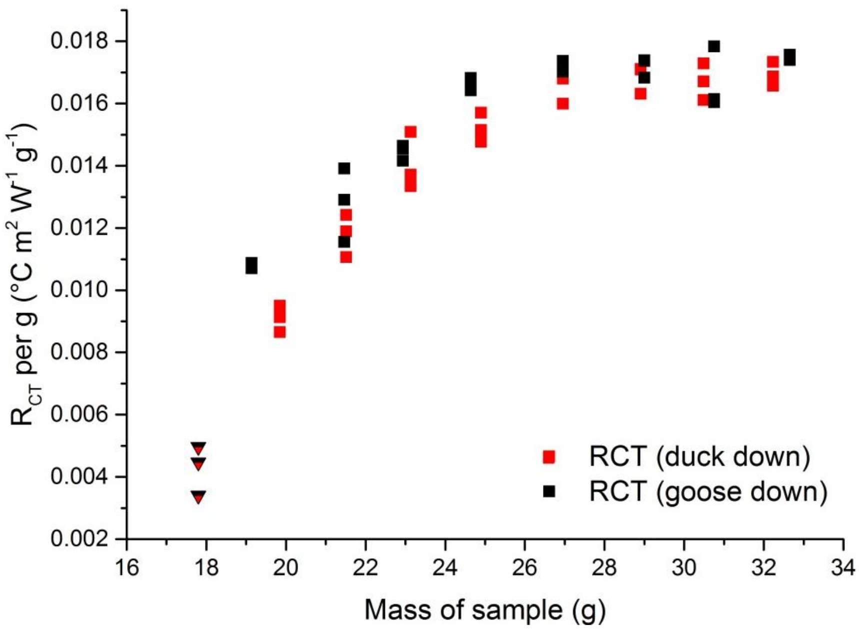


Figure 7-38 – R_{CT} per unit mass for all tests as a function of total test square mass
The triangular red /black data points are the empty baffles

There was no significant difference (single-factor ANOVA, $p = 0.34$) when comparing the R_{CT} per unit mass between the goose and duck down test squares (Figure 7-38). Goose down is significantly more expensive than duck down because of its reputation and relative rarity, and as shown earlier in this Chapter, its compression resistance is also superior. However, using duck down may be a valid way to reduce the costs associated with making high performance insulating products as, when uncompressed, its thermal resistance is equal to that of goose down.

In this testing the range of fill weights from which maximum warmth-to-weight ratio was obtained was quite broad, and despite very high fill densities being used in some samples, no decrease in thermal performance was observed as a result of too much down being in a baffle. This has an important relevance to the outdoor industry: two identical products with the same baffle sizes could be filled with different masses of down to create two products with the same warmth-to-weight ratio but different thermal resistances, making them suitable for different conditions. Though this would not allow a sleeping bag suitable for sleeping at 0 °C to be overstuffed to the point of it being useable in temperatures of -30 °C (due to possible damage of stitches under the force of extra down, possible long-term damage of the down due to compression, the difficulty of overstuffing a baffle to this extent, and the stiffness of the resultant baffle), it may allow small differences in thermal performance to be made without changing other aspects of construction. This is a much simpler protocol than the traditional method of increasing strip height (affecting baffle volume) while increasing down mass.

7.4 Summary

Two bulk properties of down feather assemblies have been analysed: their compression resistance and recovery, and their thermal resistance.

1) A novel apparatus for compressing down was developed and yielded reproducible results that showed that goose down had a higher compression resistance than duck down, generating significantly greater work in the compression phase and a higher maximum pressure during testing, possibly due to goose down's hollow barbs, or a result of subtle differences in the geometry of goose and duck down feathers that have not yet been determined.
2) The recovery of duck and goose down samples from compression was indistinguishable from one another.
3) Fill power testing subjects down samples to very small pressures which showed little or no correlation to the work in compression that occurred when down samples were compressed to pressures more representative of conditions they may encounter during use.
4) When the samples which had previously been compressed were compressed again, their work in compression reduced, as did their maximum measured pressure at a fixed degree of compression.
5) The greatest difference between agitated samples (that had not previously been compressed) and un-agitated samples (that had been compressed immediately before testing) was in the displacement to the point of maximum pressure, which was approximately 45 % lower following a previous compression cycle, showing that the down assemblies lock together during compression, preventing subsequent lofting to their full height.
6) By agitating the samples, the originally-recorded values for maximum force, work, and displacement were restored: the agitation 'unlocks' the tangled barbs, barbules and nodes, and restores maximum loft.
7) Repeated compressions of the goose and duck down samples showed that resistance to compression gradually decreased as the down feathers became intertwined.
8) Following 10 compression cycles, agitation could restore the pre-compression values of work, maximum pressure, and displacement, to both goose and duck down, which has implications for consumers, who should shake and agitate their down products following compression in order for them to loft fully.

9) Compression of test squares filled with goose and duck down of identical fill power generated similar results to those from the down assemblies: goose and duck down were very similar in their performance, but the compression resistance of goose down was greater than that of duck down. This further suggests that fill power is poorly-representative of compression resistance under high pressures.
10) As the mass of down increased in the test squares, so too did compression resistance. The compression recovery was very similar in goose and duck down and improved as fill weight increased.
11) The thermal resistances of down-filled test squares were measured with very high accuracy and repeatability using the sweating guarded hotplate.
12) The thermal resistance increased as fill weight increased, contributed to by increased thickness and reduced radiative heat transfer.
13) Despite significant overfilling of some baffles versus the mass suggested by fill power values, no decrease in thermal resistance was observed, indicating that baffles can be overfilled far beyond the ‘benchmark’ figure suggested by fill power.
14) The thermal resistance per unit weight increased with fill density until an optimum level was met and beyond this point, no further increase was measured.
15) The ‘optimum’ fill weight will vary for real products, but this testing showed that if a manufacturer is unsure how much down to put in a product, they should add more to ensure they reach the maximum warmth-to-weight ratio, or should reduce the size of the baffles that they are filling; neither of these are likely to result in decreased insulating performance.

Chapter 8. Manufacture of a down-feather-based nonwoven insulating material

A limitation of using down feathers in outdoor clothing and equipment is that it must be contained in baffles, and this makes construction complicated, adding cost, weight, and increasing the time of manufacture. Many synthetic insulations are not limited in the same way as they can be sewn into place in continuous sheets, reducing expense and negating the need for specialist down-filling facilities. Leading synthetic insulations such as Primaloft (Donovan 1986), Thinsulate (3M 2012), and Polarguard (Harding 1979; Frankosky 1983) are available in different weights and thicknesses with corresponding thermal performances. Because these materials are battings they also offer advantages in use: if the fabric containing the insulation is ripped, then, unlike with down, the battings are not liable to disperse. Also, because synthetic materials tend to be hydrophobic, they absorb very little water, making them preferred to down in very wet conditions. The greatest limitations of synthetic fibrous insulations are their warmth to weight ratio, and their diminishing performance under repeated compressions (REI 2014).

In this chapter, a down-based insulation that is simple to sew into garments like a synthetic insulation, but has the insulating performance of down, will be made. In addition, this down-based nonwoven provides a unique opportunity to compare the thermal properties of down in baffles, as tested in Chapter 7, and down-based nonwovens.

Prototype insulating nonwovens have been developed using the method described in section 2.5 and tested using the methods described in sections 2.5.1 and 2.6. During sweating guarded hotplate testing, an ultrafine cellulose sheet was placed over the samples so as to stop the potential blowing of un-bonded down plumes inside the environment chamber. This sheet provided no measurable difference to R_{CT}.

8.1 The structure of the manufactured down-based nonwovens

To investigate the effects of construction technique on the down nonwovens and to determine the extent of mixing between the down and the bicomponent fibres, two types of sample were made: blended samples, in which duck down and bicomponent ('bico' from this point) fibres were mixed together and airlaid at the same time; and sandwich samples, in which bico fibres were airlaid then a blend of down/bico, then another layer of bico. This is illustrated in Figure 8-1:

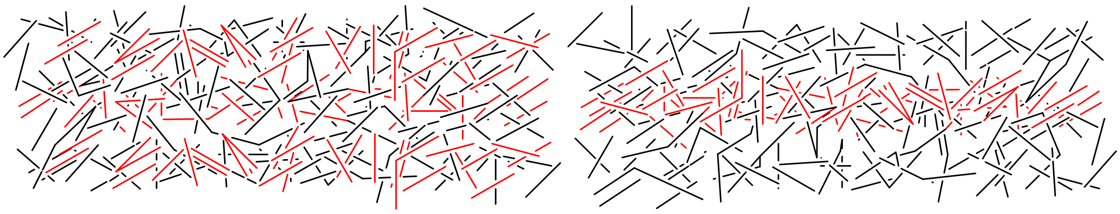


Figure 8-1- schematic of blend (left hand side) and sandwich (right hand side) nonwoven constructions
Black lines represent bico fibres, red lines indicate down plumes. Not drawn to scale

The nonwovens constructed using the sandwich method were far more likely to remain integrated, both in the plane and through the plane of the fabric, than the blended samples, which had a greater propensity to shed down feathers. This was partly because of the Kroyer airlayer's mode of operation: when blended bico and down were fed into the distributor box at the same time they were affected by the air currents to different degrees. Down feathers, being composed of many barbs and barbules designed to trap air as effectively as possible, tended to remain in the distributor box for some time, circulating under the force of the impellors. The bicomponent fibres, however, were not as affected by the air currents as the down and tended to leave the distributor box through the screen far more quickly. The greater size of the down feathers versus the bicomponent fibres exacerbated this problem as the bicomponent fibres fitted easily through the screen whereas some of the down feathers would not fit through. This meant that the intimate blend of bico and down portrayed in Figure 8-1 did not materialise in the blend samples, and instead there tended to be a large concentration of bico at the bottom of the web and the bico concentration gradually decreased as the web increased in thickness. When the sample was heated the bico fibres on the bottom of the web melted but they did not adhere to many of the down feathers above them, meaning that the down at the top of the sample bonded quite poorly and was likely to leave the web. The use of a calender that heats and presses the sample would likely lead to better bonding through the structure but at the detriment to loft, which is essential to thermal performance. With modifications to the airlaying process such as a much coarser screen or greater vacuum suction through it, it may be possible to create more even blending, but this was outside the scope of this work.

In the sandwich construction nonwovens, the formation of a complete layer of bico on the bottom and top of the blended components compensated for any stratification of the bico and down blend in the centre of the nonwoven. The bico on the bottom of the sample formed a porous mesh through which down could not easily escape and the top layer melted to bind some of the feathers towards the centre of the web while providing the same barrier to down migration that the bottom layer provided. The blend of bico fibres in the centre of the material held the centre-most down feathers together.

In use, the greater strength and resilience of the sandwich structure fabrics would be vital, but when compared to commercial insulating materials such as Primaloft (a nonwoven formed of polyester microfibres of different thicknesses (Donovan 1986) and generally regarded as the highest-performing synthetic insulation frequently used in outdoor clothing and equipment (Albany International 2015; REI 2014)), the down-based nonwoven materials lacked flexibility and resilience. Whereas Primaloft had no loose fibres, the sandwich nonwovens could still shed some feathers when under stress. This would require improvement if these fabrics were to be used commercially in products that demand durability.

An SEM image of the nonwoven composed of 100 % bicomponent fibres (Figure 8-2) was somewhat similar in appearance to the commercial Primaloft sample (Figure 8-3).

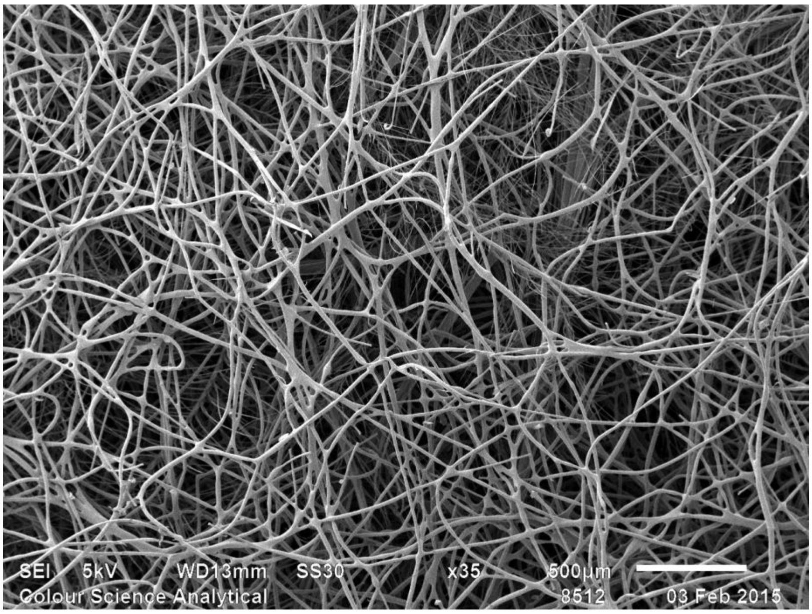


Figure 8-2 – 100 % bicomponent fibre nonwoven sample observed using SEM

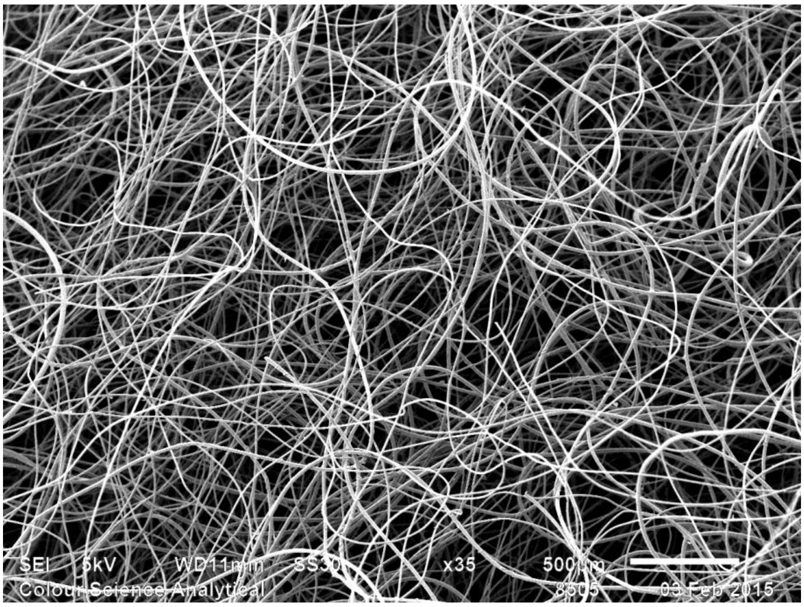


Figure 8-3 – 4 oz. m^{-2} commercial Primaloft sample observed using SEM

Despite the similarities to the 100 % bicomponent fibre nonwoven, Primaloft was very different in morphology to the down-based nonwovens (see Figure 8-4). In the down nonwovens, the air spaces between barbs and barbules were much more varied in size: some gaps were very small, whereas other much larger spaces were present between barbs.

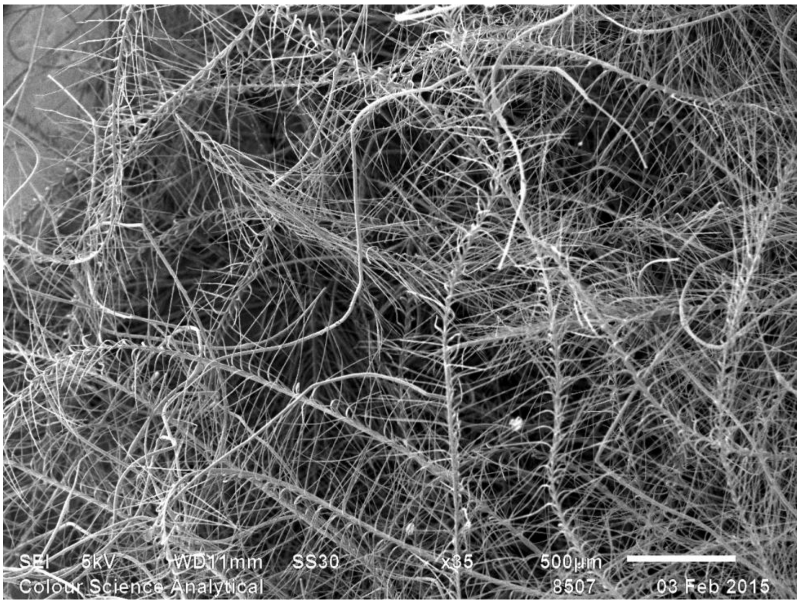


Figure 8-4 – a blended bico and duck down airlaid nonwoven observed using scanning electron microscopy

Adhesion between different bicomponent fibres (for example, see Figure 8-5), were visible in all of the samples and bonding between the down and bicomponent fibres were observed in some (see Figure 8-6):

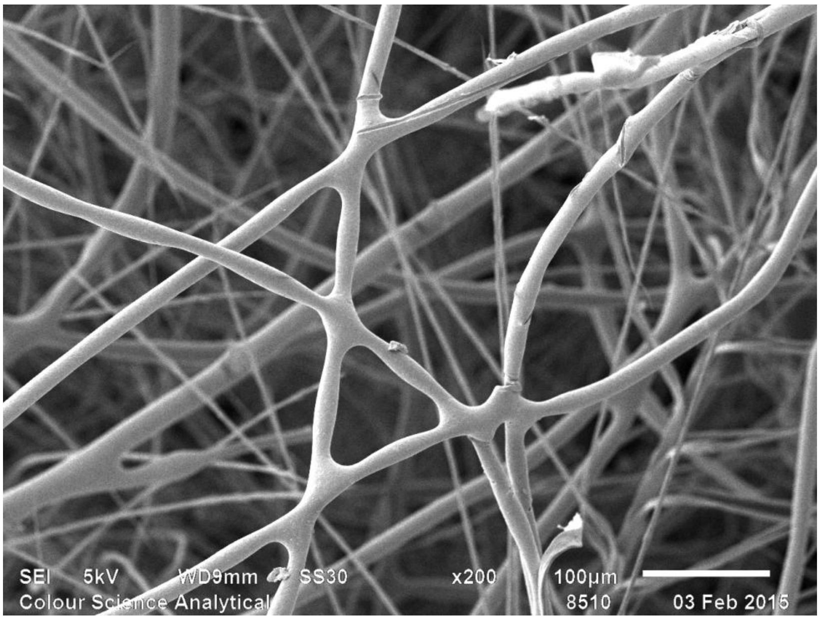


Figure 8-5 – sandwich-construction bico and duck down airlaid nonwoven observed using scanning electron microscopy
Note the pull-out marks in the fibres at the top of the image

Figure 8-6 – blended bico and duck down airlaid nonwoven observed using scanning electron microscopy with duck down barbs bonded to bico fibres

It is likely that the bicomponent fibres were more adherent to one another rather than to down feathers as both surfaces could melt during thermal bonding. The relative lack of direct bonding between the down and bicomponent fibres was not as problematic in the sandwich construction samples as in the blended samples as the network of bicomponent outer fibres in the composite held the down feathers inside the structure.

Possible degradation of the down-based nonwovens was indicated in some SEM images as lines across the bicomponent fibres (as shown in Figure 8-5) that implied bonds between fibres had since debonded. It was not possible to determine whether these bonds failed during solidification of the composite, or whether they failed under subsequent mechanical stress. Interestingly, the same markings were observed in the Primaloft fibres (Figure 8-7):

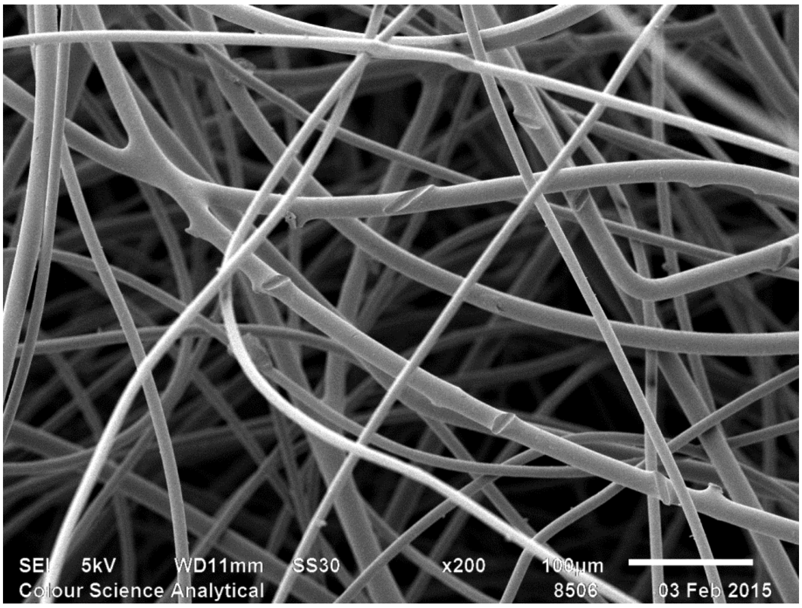


Figure 8-7 – SEM micrograph of commercial 4 oz. m^{-2} Primaloft sample highlighting fibre pull-out

The debonding shown in Figure 8-7 may be a cause of the degradation in thermal properties (REI 2014) that occurs in synthetic insulating materials with time: the fibre crossing-points are broken and the fibres are then able to slide over one another and so compression recovery is worsened, as discussed in Chapter 7. The down-based nonwovens' performance has not been tested over a prolonged period, but it is anticipated that it would undergo similar performance decreases as commercial synthetic nonwoven products due to the observation of these same debonding characteristics.

8.2 Physical properties of the manufactured down-based nonwovens

Figures regarding the physical properties of the down-based nonwovens are shown in Table 8-1, overleaf:

Table 8-1 – composition of different down and bico blends and their masses, basis weights, and R_{CT} values

(Bico refers to bicomponent fibre; the Primaloft sample was 4 oz. m^{-2} Primaloft One, supplied by Albany International (USA) (Albany International 2015))

Composition	**Approximate % down feather**	**Sample mass (g)**	**Basis weight ($g\ m^{-2}$)**	**Mean thickness (mm)**	**R_{CT} (°C $m^2\ W^{-1}$)**	**% CV R_{CT}**	**R_{CT} /mass (°C $m^2\ W^{-1}\ g^{-1}$)**
100 % bico	0.00	5.98	62.23	2.38	0.15	1.65	0.0244
1:1 down: bico blend	50.00	4.85	50.47	5.40	0.20	2.04	0.0412
7:6 down: bico blend	53.85	5.82	60.56	6.50	0.21	0.54	0.0361
4:3 down: bico blend	57.14	5.71	59.42	5.68	0.25	1.62	0.0438
2:1 down: bico blend	66.67	4.81	50.05	8.68	0.27	1.55	0.0561
4:1 down: bico blend	80.07	4.35	45.27	6.48	0.27	0.67	0.0621
5:7 down: bico sandwich	41.67	5.67	59.00	4.90	0.24	0.34	0.0419
5:6 down: bico sandwich	45.45	4.48	46.62	4.08	0.20	1.64	0.0443
6:7 down: bico sandwich	46.15	5.51	57.34	5.28	0.21	1.79	0.0377
1:1 down: bico sandwich	50.00	4.93	51.30	5.83	0.23	1.35	0.0467
7:4 down: bico sandwich	63.64	5.42	56.40	5.85	0.24	1.37	0.0449
Primaloft	0.00	11.91	123.93	6.18	0.33	2.17	0.0277

The basis weight of the down-based nonwovens is plotted against their thickness in Figure 8-8:

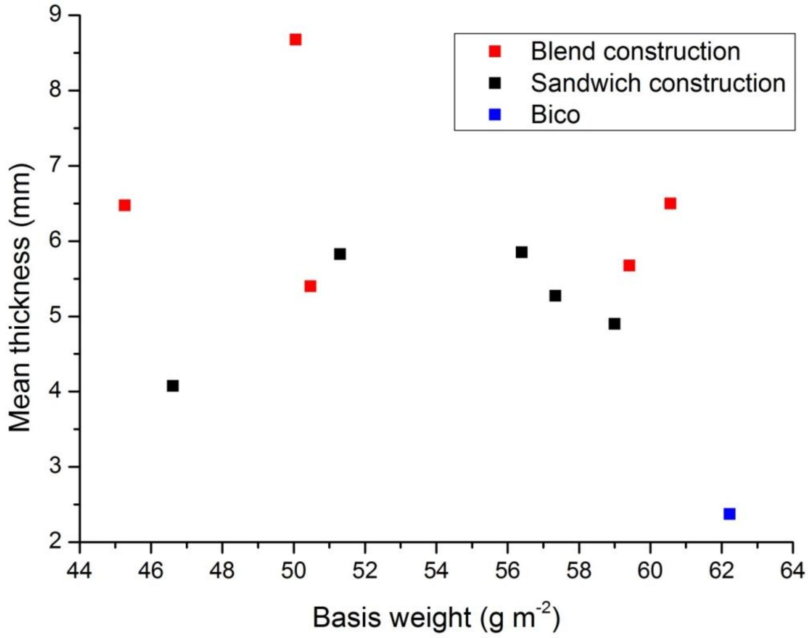


Figure 8-8 – the mean thickness of the down-based nonwovens plotted against their basis weight measured using standard method BS: EN 5084 (British Standards Institute 1997)

There was no obvious relationship between the basis weight of the fabrics and their mean thicknesses, as shown in Figure 8-8. This is an indication that the dominant factor that determines their thickness was the basis weight of down in their construction, not simply the mass of fibres. This is shown to be true in Figure 8-9, where both the sandwich and blend constructions increase in thickness as the basis weight of down increases (the basis weight of the nonwovens multiplied by the fraction of down):

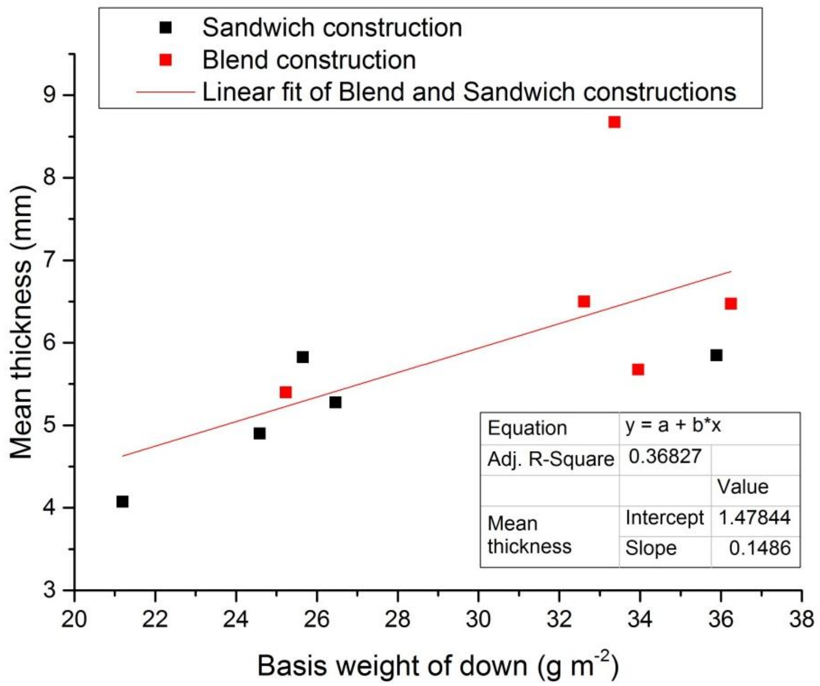


Figure 8-9 – the thickness of the nonwoven samples plotted as a function of their basis weight of down (the basis weight of the nonwoven multiplied by the fraction of down)
Thickness measured using standard method BS: EN 5084 (British Standards Institute 1997)

The well-known relationship between thickness and thermal resistance (Fletcher 1945; Schiefer 1944; Pierce & Reese 1946) was adhered to by the down-based nonwovens, as shown in Figure 8-10:

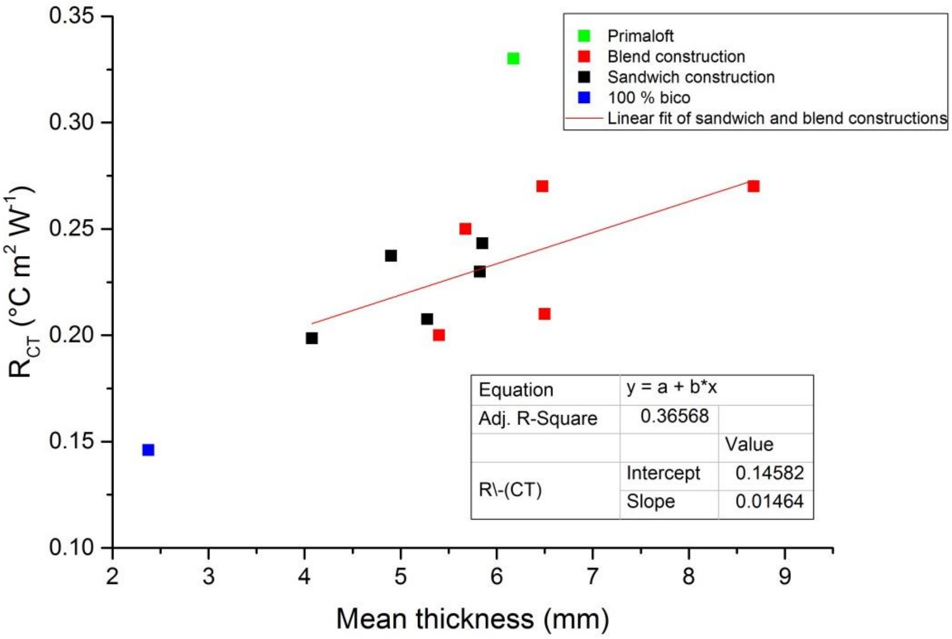


Figure 8-10 - R_{CT} of down and bicomponent nonwoven fabrics plotted as a function of their thickness
Thickness measured using standard method BS: EN 5084 (British Standards Institute 1997)

R_{CT} increased with thickness in both the sandwich and blend nonwovens. Interestingly, as shown in Figure 8-10, the Primaloft sample had a greater R_{CT} as a function of its thickness than the down nonwoven samples did. Previous researchers (Gibson 1990) have shown that down is inferior to some nonwovens when compared on a thermal-resistance per unit of thickness relationship, and this was also found in these down-based nonwoven samples. Dry thermal resistance is a function of the radiative, conductive, and convective heat transfer mechanisms in an insulating material, so if comparing the Primaloft sample to down-nonwovens of identical thickness, differences in conduction can effectively be ignored, as they are likely to be comparable due to the similar amounts of air trapped in each fabric, and air is the largest contributor to the conductive transfer of a fabric (Baxter 1946). The radiative resistance of down's barbs and barbules are thought to be near-perfect (Wan et al. 2009), but the greater mass of fibres, and thus greater surface area, offered by the approximately 100 g m^{-2} Primaloft, which itself has a mixture of fine and ultrafine fibres (Donovan 1986), probably offers a greater resistance to radiative transfer than the much lighter-weight down-based nonwovens. The properties of the down-based nonwovens and the Primaloft are also likely to differ in their resistance to convection: though a very thin cellulose scrim was placed over the samples during testing to prevent loose down becoming airborne, this did not negate all forced air flow in the test, which operated under an air speed of 1 m s^{-1} ((British Standards Institute 1993), sufficient to disturb still air in the fabrics. The greater mass of fibres in Primaloft versus the down nonwovens better prevents the loss of heat by convection, and this also contributes to its greater thermal resistance per unit thickness. The relatively poor performance of the down nonwovens in relation to thickness makes them perhaps poorly suited to applications where dexterity is vital, such as in gloves (Hartland 2010). Thickness also influences handle and the perceived thermal properties of the fabric: many consumers associate warm clothing with thick layers, perhaps due to the use of down jackets and thick woollen knits in cold weather. Thus, these relatively thick down nonwovens may actually be favoured by consumers over thinner synthetic nonwovens.

The R_{CT} per unit mass for each nonwoven samples is plotted against the percentage of down in the sandwich and blend constructions in Figure 8-11. Trend lines have also been added:

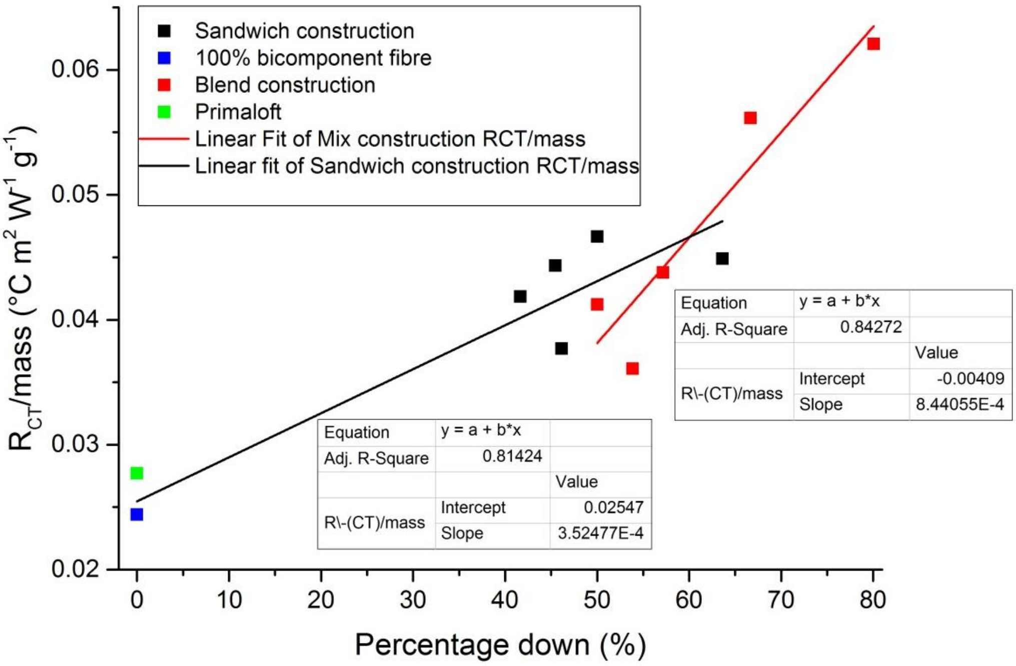


Figure 8-11 - R_{CT} per unit mass of down for down and bicomponent nonwoven fabrics plotted as a function of their percentage of down

The relationship between R_{CT} per unit mass and the percentage of down shown in Figure 8-11 in the sandwich and blend constructions were similar: as the percentage of down increased, so too did the warmth-to-weight ratio (R_{CT} per unit mass). This is because down has a greater thermal resistance per unit mass than the bicomponent fibres, and as down feathers are incorporated, these aid the confinement of still air and thus the R_{CT} per unit mass increases. The melting of part of the bicomponent fibre likely exacerbates its low thermal performance, as the glue-like binder between fibres barely aids the trapping of air. The R_{CT} per unit mass of the sandwich construction nonwovens increased approximately linearly with the percentage of down, but the rate of increase was greater in the nonwovens with a blend construction. This is likely to be because, in the sandwich constructions, the mass of bicomponent on top of the down caused it to be limited in thickness and unable to loft as fully as the down that was bound internally, rather than externally.

Comparisons to Primaloft showed that the down-based nonwoven insulations had superior warmth-to-weight ratios, providing very similar R_{CT} values for approximately 50 % of the mass. This is an excellent result, as Primaloft is very well regarded in the outdoor industry (Albany International 2015; REI 2014), and if a down-based nonwoven such as this could provide similar warmth for half the weight then this would be beneficial to many users.

The down-based nonwovens offered an excellent opportunity to investigate the effects of construction techniques on R_{CT}. In Figure 8-12 the thermal resistance of the down-based and Primaloft nonwovens are plotted alongside the thermal resistance of the goose and duck down samples tested in Chapter 7 which were enclosed in face fabrics:

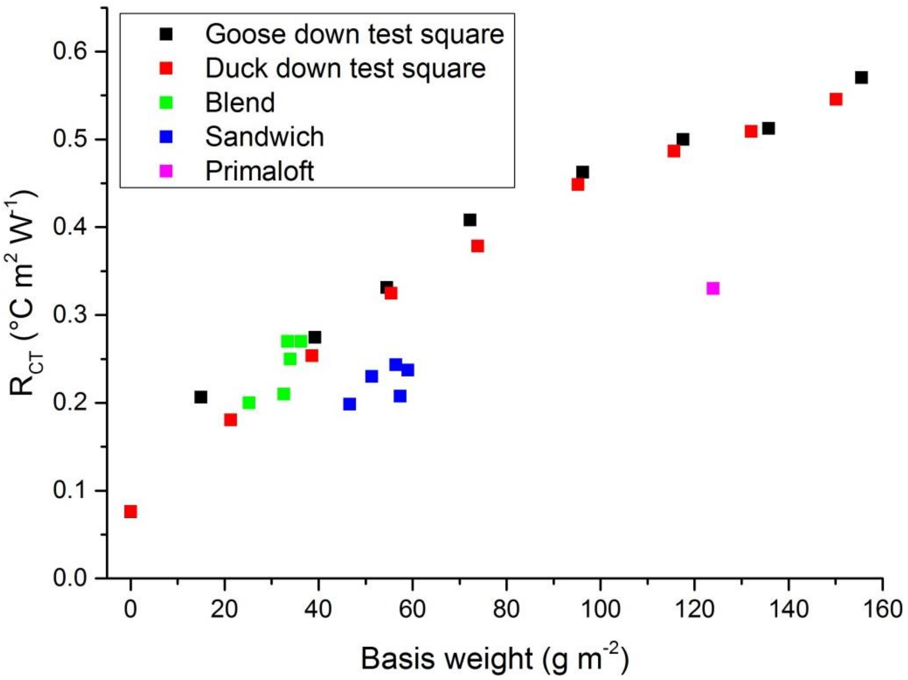


Figure 8-12 - R_{CT} plotted against basis weight for goose and duck down samples enclosed in baffles, for down and bicomponent nonwovens, and for 4 oz. m^{-2} Primaloft synthetic insulation

In Figure 8-12, the thermal resistance of the goose and duck down samples enclosed in face fabrics present a formidable 'benchmark' for their basis weight. In the experiments in this Chapter, the down feather nonwovens were not made in a wide range of basis weights, but between 30 and 40 g m^{-2} it is clear that the blend nonwovens offer very similar thermal resistance values to the more conventional down-filled constructions used in the test squares. The sandwich construction nonwovens were less warm for their basis weight. The Primaloft sample was significantly less insulating for its weight than the baffled goose and duck down samples, and as seen in Figure 8-11, the down nonwovens achieved similar R_{CT} values to it, despite being much lower basis weights. Figure 8-12 shows the exceptional performance of down enclosed in face fabrics and its superiority over synthetic insulations, but it also shows the potential for down-based nonwovens to bridge the gap between synthetic nonwovens and down.

The manufacture of these down-based nonwovens has concentrated on making materials suitable for use in the outdoor industry, but they could also be used in numerous other applications where warmth-to-weight ratio is needed, such as in automotive insulation. The techniques could also be beneficial in reducing the complexity of manufacturing down-filled bedding and the potential to use down of lower quality than that used here could be a valuable way to reduce costs while still providing excellent insulation. The process of blending bicomponent fibres with feathers could also be extended beyond down feathers as a way of making cheap insulating materials, but the geometric differences between, for example, chicken feathers and down feathers, means that these materials may offer limited thermal resistance.

8.3 Summary

In this chapter, down-feather-based nonwovens have been made using air-laying and thermal bonding.

1) Two different construction techniques were used: sandwich, and blend constructions. Of these, the sandwich construction offered greater structural integrity due to it forming dense webs at the top and bottom of the nonwovens as the bico fibres melted and trapped any down feathers that were not formally bonded to other bico fibres.
2) The great advantage of these materials over conventional down feathers is their ability to be sewn rather than being blown or hand-stuffed into garments and equipment, reducing manufacturing cost and complexity.
3) The warmth-to-weight ratio of the down-based nonwovens was higher than that of Primaloft, an industry-leading synthetic nonwoven.
4) The excellent warmth-to-weight ratio of the down-based nonwovens was contributed to by their high thickness, enabling them to trap a large volume of still air.
5) When compared to down enclosed in baffles, the blended nonwovens had similar warmth-to-weight ratios, while the sandwich nonwovens were marginally less insulating.
6) The exceptional warmth-to-weight ratio of the down nonwovens means they are of potential use in garments and equipment, though issues relating to durability and structural integrity need to be resolved.
7) The method used in this chapter might be applicable to other filling materials, and may be particularly useful as a way of utilising low-quality down in outdoor equipment.

Chapter 9. Conclusions and future work

9.1 Conclusions

The purpose of this work is to examine the structure of down feathers and evaluate how this impacts on their mechanical and thermal properties. Here, conclusions drawn are summarised in two parts: those relating to down feather structure, and those relating to properties.

9.1.1 Structure

1) The mean masses of individual goose, duck, and eider down plumes were determined as 0.77 mg, 0.70 mg, and 2.02 mg, respectively. The differences between eider down plumes' masses and those of goose and duck down were statistically significant.
2) The mean diameter of eider down barbs was significantly greater than those of goose and duck down.
3) The barbules of goose, duck, and eider down each have a planar cross section where they intersect with the barbs. It is believed that this structure aids compression resistance and helps down feathers recover from compression.
4) The lengths of barbules are adapted to their positioning on the barbs to maximise their space-filling potential while minimising potential tangling and leverage causing collapse.
5) Each type of down had nodes that were distinct in shape and size.
6) Eider's down feathers are larger, their barbs thicker, and they have more tertiary structures (nodes and prongs) than either goose or duck down feathers.
7) Components common to wool and hair were also observed in duck and goose down's cross sections when observed using TEM.
8) Goose and duck down both had a sulfur-rich cuticle, a cortex made of orthocortical and paracortical cells, cell membrane complexes, and nuclear remnants. These characteristics show that down feathers are, like wool fibres, fibril-matrix composites.
9) Immature macrofibrils were observed with excellent clarity using AFM. AFM had never-before been used to study down feathers.
10) The barbules of goose and duck down were very similar in shape, but their barbs were different: goose down's barbs were approximately triangular, whereas duck down's were more circular.
11) The examined goose down barbs had a hollow core, as shown using SEM, TEM and EDX, while the examined duck down barbs exhibited solid cross sections.

12) Osmiophilic granules observed in TEM analyses were shown, using EDX, to be melanin granules due to their shape, and high potassium and oxygen contents. Melanin granules give keratins their colour and may affect modulus and toughness.
13) A novel method was developed for preparing complex samples for X-ray diffraction and was used to prepare goose and duck down feathers for X-ray analysis of their crystal structures. By using airlaying and hydroentanglement, the diffraction patterns were realised with much greater resolution.
14) Analysis of goose and duck down showed near-identical X-ray diffraction patterns that were modelled accurately by using six diffraction peaks, including a diffraction spacing of 3.15 Å that is indicative of avian keratin.
14) Close similarities in thermal properties between goose and duck down and wool were shown in DSC results. Down required heating to 150 °C for complete drying to take place. It is of note that though the T_g of the down samples could not be determined, values from wool suggest that a change in T_g may explain down's change in fill power upon steam treatment or tumble drying.

9.1.2 Properties

1) The true densities of goose and duck down were statistically indistinguishable (duck: 1.44 g cm^{-3} when undried, 1.27 g cm^{-3} when dried; goose: 1.42 g cm^{-3} when undried; 1.25 g cm^{-3} when dried), meaning that differences in their properties were not merely due to differences in their densities.
2) The properties of goose and duck down's surfaces under ATR-FTIR analyses were extremely similar, indicative of near-identical surface chemistries in these commercial feathers.
3) Individual down barbs from goose, duck, and eider down were tensile tested. Those from goose and duck down were tested using two different machines, those from eider using one. Each was modelled as cylinders. Their tensile properties were very close to those reported by previous researchers (Bonser & Dawson 1999; Bonser & Farrent 2001): goose down barbs had a Young's modulus of 1.94 and 2.76 GPa, duck down barbs a Young's modulus of 2.41 and 2.12 GPa, and eider down barbs a Young's modulus of 3.20 GPa.
4) Cylinders did not accurately represent the cross sections of the down barbs, and when they were modelled according to the cross sections observed using TEM and SEM, their Young's moduli were much higher. The ultimate strength, strain at break, and Young's modulus of eider down's barbs were significantly greater than those of goose or duck down; goose and duck down were not significantly different to one another.

5) The compression of individual goose, duck, and eider down plumes showed that goose and duck down feathers were very similar in the maximum force and work required to compress them. Eider down plumes, however, were significantly more resistant to compression than either goose or duck down.
6) When compressing individual down feather plumes, the three types of down recovered from compression to very similar degrees – within 2 % of one another – indicating that differences in geometries and tertiary structures made almost no difference to compression recovery when feathers were tested in isolation.
7) The compression of individual down barbs showed that they could be modelled very accurately by Euler's theories regarding the compression of struts. This implies structures with very few flaws and excellent consistency along their length and that the hollow structure of goose down barbs is expected to impart them with greater bending resistance than the solid duck down barbs.
8) A novel device for measuring the compression resistance and recovery of down assemblies was developed that yielded reproducible results.
9) Goose down assemblies were more compression resistant than duck down assemblies, requiring greater work to compress them. This might be attributed to the cross-sectional shape and hollow cross section that were observed in goose down and which are anticipated to provide it with an increased bending resistance. The recovery of the goose and duck down assemblies, as had been observed in the testing of the individual feathers, was identical to one another.
10) The compression of goose and duck down samples to the pressures exerted in the fill power test was not sufficient to distinguish between the goose and duck downs, yet their properties differed when compressed with greater force. There was little or no correlation between the compression resistance of a sample under the small pressures exerted in fill power testing and the higher pressures that might be encountered in use, indicating that fill power is not necessarily a good way to assess a down sample's resistance to high pressures.
11) With repeated compressions of the goose and duck down samples their resistance to compression gradually decreased as the down became intertwined. After ten compression cycles, agitation could restore the pre-compression values of work, maximum pressure, and displacement, to both goose and duck down.
12) Down-filled test squares were made specifically to fit a sweating-guarded hotplate and to measure their thermal resistance. This allowed for designs representative of real-life products to tested relatively quickly and with excellent accuracy.

13) The down-filled test squares increased in both compression resistance and thermal resistance with an increasing density of down inside them. Despite filling some baffles in excess of 3 times the benchmark figure calculated by using fill power, no decrease in thermal performance was observed. If manufacturers are unsure how much down to use in their product, they should add more, so as to achieve maximum warmth-to-weight ratio; there seems to be little risk to overfilling the product and losing thermal performance.
14) Complex construction techniques are required to use down feathers in outdoor equipment, so nonwoven fabrics made from down and bicomponent fibres were developed using two different construction techniques. Nonwovens made using a blended construction were insufficiently durable, but sandwich construction composites were much more stable.
15) The thermal resistance per unit mass of the down-based nonwovens developed in this project was greater than that of Primaloft, an industry-leading synthetic insulation, and the blended samples had very similar warmth-to-weight ratios to down-filled test squares.
16) The method used to manufacture the nonwovens may be commercially viable if the limitations in durability can be negated and may also be an excellent way to utilise lower-quality down feathers in insulated products.

9.2 Future work

Down insulation remains a formidable benchmark as an insulating material and this is influenced primarily by its unique hierarchical structure and cross-sectional morphology, but there remains great potential for further work into the structure and properties of down feathers. Future work regarding the morphology of down should largely concentrate on the testing of further batches of down. In this work, goose and duck down of premium quality were tested, but analysing goose and duck down that is used in cheaper products would be beneficial to determine whether there was a difference in structure between these materials. The other major area of research into structure could focus on the growth of the feathers during the life cycle of the bird. This would require very closely-monitored live geese and ducks, where samples were either plucked from the birds at specific intervals, or birds were slaughtered at different ages and the feathers subsequently removed. This would be a very large project, requiring significant preparation regarding housing and care of the birds, and the seeking of potentially-difficult ethical approval. However, research with this level of control over the source of the down would likely reduce its variability.

The internal structure of goose and duck down was assessed here using TEM, and further analyses of this type would show whether the differences between the birds' down feathers are consistent across a greater range of samples. Eider down was not studied using TEM during this project, and it is anticipated that it would possess similar internal characteristics to both goose and duck down, though it would likely contain more melanin than either material, owing to its darker colour.

This work has established that AFM is a very powerful tool for studying down feathers' surface structure, but further work in this area could attempt to detect the avian microfibrils that are close to the resolution-limit of TEM. Detailed images taken using AFM of these materials may be able to verify the structure of avian keratin proposed by Fraser and Parry (2008). Use of synchrotron radiation for further X-ray analysis could also be beneficial in determining the location of amorphous, crystalline, and semi-crystalline regions in down. The exceptional X-ray flux of synchrotron radiation makes high resolution analysis possible without experiments lasting many hours.

The tensile properties of down barbs are now quite well-understood, but the compression resistance of individual down plumes is something that could be further investigated. In this work, limitations in equipment meant that the weighing and subsequent analysis of individual plumes could not easily be undertaken, but further work should attempt to carry this out. The uniaxial compression of individual down barbs could also be undertaken using the Agilent T150 that was used for some of the tensile testing. This is thought to be the only commercially-available machine sensitive enough to test the very small forces exerted in the compression of these very fine materials.

Further testing of bulk quantities of down feathers should concentrate on analysing eider down and goose and duck down of significantly lower quality than those measured here. The compression resistance of lower-quality duck and goose down is thought to be lower than that of high quality down, though it may be that the compression resistance is actually higher due to the presence of numerous stiff flight feathers, and the compression recovery worse due to these flight feathers tangling with the down plumes. The thermal resistance of down of lower fill power is almost certainly lower than that of high fill power down due to it trapping less air, but to the author's knowledge this has not previously been determined. It is expected that an approximately- linear relationship between fill power and thermal resistance would exist for most samples, but down of exceptionally high fill power may be unable to maintain its bulk under the mass of some face fabrics, and this has not been determined.

The down-based nonwoven prototypes developed here are promising, but further work is required to ascertain whether their durability and resilience could be improved. If these characteristics could be made comparable to existing synthetic insulations then this material would doubtless be of great benefit to the outdoor industry.

The largest area of research that has not been addressed in this thesis is the water repellence of down. This is a very large topic and one where the existing work carried out on wool could be very influential. The prevalence of commercially-available water-repellent treatments for down in recent years has led the IDFL (2012) to recently develop a method for testing down's water repellence and water absorption, and a British Standard (British Standards Institute 2001) exists to measure the mass of water absorbed by down feathers, but preliminary testing showed these methods to offer low repeatability (Fuller 2012). The measurement of contact angles on down feathers is also extremely problematic owing to their geometry, as contact angle measurements rely on the presence of a flat surface. Despite the relatively poor performance of wet down, down's performance when dry means that these remarkable materials will continue to be used by the outdoor industry for many years.

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