Measurement Protocols for Exchange Biased Systems

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Abstract

A study of exchange biased systems has been made focused on the development of measurement protocols that yield measurement reproducibility. The materials studied were IrMn(t_{AF})/CoFe(10nm) thin films, with t_{AF} being 3nm and 5nm. These materials were selected due to their industrial applications. The thicknesses were chosen as they were the most appropriate for the purpose of this study.

Vibrating sample magnetometry has been used to investigate thermal activation properties in the set of samples. The measurement protocols developed allowed measurement reproducibility. They lead to a better understanding of the exchange biased phenomenon. It was found that the changes occurring in the exchange biased systems during measurement are due to thermal activation in the antiferromagnetic layer which reduced the degree of order within the antiferromagnet. It was observed that the peak in the coercivity with the temperature of thermal activation does not correlate with the median blocking temperature.

A study of training effects and their behaviour with temperature has led to the identification of three different contributions to exchange bias which are spin freezing, spin reorientation, and thermal activation. Each of these effects contribute to the shift of the hysteresis loop and the coercivity of exchange biased systems through different mechanisms.

The results obtained are consistent with a grain size dependence of the exchange bias phenomenon, and are in accordance with grain models of exchange bias. Also, the measurement protocols obtained have been used by other researchers in FeMn/NiFe exchange coupled bilayers. The results obtained were in accordance with the conclusions of this thesis.
## Abstract


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Declaration

I declare that the work presented in this thesis is based purely on my own research, unless otherwise stated, and has not been submitted for a degree in either this or any other university.

Signed

Luis Eugenio Fernández Outón
Chapter 1

Introduction

The phenomenon called exchange anisotropy or exchange bias was discovered almost fifty years ago, by William H. Meiklejohn and Charles P. Bean. They studied partially oxidised Co particles. This system exhibited a shifted, hysteresis loop when measured after cooling to a low temperature in a field. Also the loops were broader than those measured for pure Co particles. Their first paper was called "A New Magnetic Anisotropy" and was published in Conference on Magnetism and Magnetic Materials, Boston, MA, October 16-18, 1956: "We now have an additional mechanism involving an interaction between an antiferromagnetic material and a ferromagnetic material. Since the origin of this effect is postulated as being an interaction between the spins of the cobalt atoms in the metal and the cobalt ions in the antiferromagnetic cobaltous oxide, it might properly be termed an exchange anisotropy". Exchange anisotropy is responsible for an enhancement of the width and a shift of the hysteresis loop in the field axis by an amount known as exchange field \( H_{ex} \). Figure 1 shows a schematic of the hysteresis loop of an exchange biased system, showing the shift, defined as an exchange field, \( H_{ex} \), and the half width known as the coercivity \( H_C \). Full descriptions of the phenomenon and its effects have been extensively covered in review articles by Nogués and Schuller (1999), Berkowitz and Takano (1999), and Nogués et al., (2005).

![Figure 1 Schematic of the hysteresis loop of an exchange biased system. The exchange field, \( H_{ex} \), is given by the shift of the loop from zero field. The half-width of the loop is called coercivity, \( H_c \).](image-url)
Proposed applications of the exchange biased phenomenon include permanent magnets, magnetic recording media or magnetic domain stabilisers in recording heads. Since its discovery, several thousand of papers have been published on the topics of exchange bias or exchange anisotropy phenomena. This is mainly due to the fact that exchange bias is the principle of operation of read heads in magnetic hard drives used not only in computers but also other media and entertainment devices. Therefore the information storage industry is the epicentre of the investigation and development of exchange biased systems and applications.

Technological applications of exchange bias involve the use of thin film exchange coupled ferromagnets, antiferromagnets and in some cases ferrimagnets. The nature of the interfacial exchange interaction between the ferromagnet and antiferromagnet is of quantum origin. Hence, detailed atomic-level information about the structure and the magnetic state is needed to obtain reliable models. The development of measurement techniques facilitates the provision of data so that more accurate models of exchange biased can be designed. However, all state of the art models still contains fitting parameters to cope with this many body problem. In addition to this, to compare the predictions of exchange biased models with experimental data it is necessary to specify the experimental conditions prior to and during the measurement of the exchange biased system. However this is not often done in the literature, leading to measurement irreproducibility, and misleading modelling. Furthermore, it is not often appreciated that the state of the antiferromagnetic layer can change during measurements due to the exchange field from the ferromagnetic layer changing sign as the loop is measured. Also the state of the antiferromagnet can change due to thermal processes during measurement. Due to the application of exchange biased systems as read heads, it is of interest to know which are the best materials to be used, which offer the best performance at reduced thicknesses and stability at the elevated working temperatures. These factors are the motivation to obtain a deeper understanding of the exchange bias phenomenon.

It is the purpose of this thesis to develop and test measurement procedures that provide reproducible data. The resulting procedures need to be simple to use and should allow traceability of all stages of measurement to be able to check any detail of the process easily. That is why this thesis is not a survey of materials for exchange biased systems,
nor a study of the structural properties that give rise to the exchange coupling across the interface of a ferromagnet/antiferromagnet bilayer. This thesis is focused on the experimental conditions that give rise to measurement reproducibility so that measurement protocols can be established. Also, it is of interest that such protocols are developed to study exchange bias phenomena such as thermal activation and training effects, where the loop shift reduces with repeated measurement.

Most of the models published on exchange bias are based on the domain state of the interfacial layers between the ferromagnet and antiferromagnet [e.g. Mauri (1987), Malozemoff (1987,1988 (a,b), Stamps (2000), Stamps et al. (2000), Nowak et al. (2002)]. These models cannot be applied to systems of particles, such as the Co particles studied by Meiklejohn and Bean, and can only be applied directly to single crystal thin film systems. Systems such as those studied in this thesis, which are composed of IrMn/CoFe bilayers are granular in nature. Hence, models have been proposed which are based in the interpretation of both the ferromagnet and the antiferromagnet as ensembles of grains [e.g. Fulcomer and Charap, 1972(a,b), Nishioka et al., 1996, Takano et al. 1997, Stiles and McMichael, 1999(b), Tsunoda and Takahashi, 2002]. The grain models usually treat the ferromagnetic layer as a single grain. However changes in the ferromagnet due to its granular nature can lead to changes in the interactions between ferromagnet and antiferromagnet and consequently in the antiferromagnet itself with field cycling.

All the models proposed for exchange bias agree to some extent with experimental results. However there is no model that can predict all the features of exchange bias. The success of the various models is described in the reviews of exchange bias [e.g. Berkowitz and Takano (1999), Nogués and Schuller (1999), Stamps (2000), Nogués et al., (2005)]. In general granular models include the effect of thermal activation, whilst generally the domain models do not include temperature dependent effects.

The Gaussian c.g.s system of units has been used in this work. The equations that appear in the following chapters are in the c.g.s. system. These units have been used since it is the system most widely used by the magnetic recording industry and the applied magnetism community. More information about the principal units used in magnetism can be found in e.g. Cullity (1972).
Chapter 2

Magnetism of Thin Films

2.1 Introduction to Exchange Coupled Films

An exchange bias system consists of an antiferromagnetic material, \( AF \), in intimate contact with a ferromagnetic material, \( F \). These systems exhibit some characteristic features when field cooled, from a temperature higher than the Néel temperature, \( T_N \), of the antiferromagnet, to below \( T_N \), under the influence of an applied magnetic field, high enough to saturate the ferromagnet. The effect was first discovered by W. H. Meiklejohn and C. P. Bean (1956) in a seminal paper “New Magnetic Anisotropy”.

During such a process the ferromagnet, \( F \), acquires uni-directional anisotropy. Its magnetisation \( M_F \) is biased in the direction of the applied field in which the \( F \) was cooled. The applied field necessary to reverse \( M_F \) is then higher than that needed to reverse the magnetisation of the same isolated \( F \). Hence, the hysteresis loop associated with the \( F-AF \) structure is centred about a non-zero magnetic field. This phenomenon is called exchange bias (EB). The shift in the loop from zero-field is known as the exchange bias field or bias field (\( H_{ex} \)). See figure 1.

The memory of this preferred direction for the magnetisation of the ferromagnet persists as long as the bias field is not modified by heating over \( T_N \) or over a blocking temperature \( T_B \), under the influence of a saturating magnetic field. The temperature \( T_B \), at which \( H_{ex} \) equals zero, is known as blocking temperature. There are other features characteristic of exchange bias such as an increase in the loop width, defined as \( 2H_C \), of the \( F \) material even when the \( AF \) is cooled in zero-field [Gökemeijer et al. (1999), Stiles and McMichael (2001), Kume et al. (2005)]. There is also a peak in the value of the coercivity, \( H_C \), in the region of \( T_B \) (Carey et al., 2001), and an asymmetric loop for the \( F \). An anomalous reduction in the value of \( H_{ex} \) is observed when the magnetisation is repeatedly cycled, known as training, and their uncharacteristic form of the magnetic viscosity in the ferromagnet [Fulcomer and Charap (1972 a,b), Goodman et al. (2000)].
This thesis describes a study of the features of the exchange bias phenomenon in thin film systems composed of a single ferromagnet-antiferromagnet bilayer and the development of protocols for their reproducible measurement.

### 2.2 Preparation of Thin Films

There are basically two types of nanogranular magnetic thin films. There are magnetic films that are composed of grains that are exchange coupled together which reverse by domain wall motion generally having low coercivity. On the other hand, there are magnetic thin films formed by grains that are segregated by nonmagnetic elements that have limited solubility in the magnetic host and as result of this segregation to grain boundaries causing exchange decoupling. The hysteresis is then dominated by the anisotropy and volume of the grains.

The importance of exchange-coupled films lies in the fact that they exhibit Giant Magneto-Resistance, GMR, (see section 3.4). To maximise the magneto-resistance effect it is necessary to induce an easy direction in the film, i.e. uniaxial anisotropy, and so preparation routes are important. Among the methods for thin film fabrication, electrodeposition, evaporation and sputtering are the most widely used.
Electrodeposition is used for the fabrication of sensors, especially recording heads, having the advantage that all components can be deposited in a single cell including the leads via a series of lithographic steps. Deposition is performed in a field to induce an easy axis. Often the films are annealed at 350°C in a field to improve their crystallography. The problem with electrodeposition is the production of anions, such as Cl⁻ or SO₄²⁻, inside the chamber, which lead to contamination and corrosion, leading to pinning. Thin films produced by electrodeposition have thicknesses larger than 100nm to obtain uniformity.

Thinner, less contaminated films can be made by evaporation in vacuum. In this process the use of a heated substrate removes the need for annealing and there is less contamination. Uniaxial anisotropy is generally obtained by applying an external field in the plane of the substrate. The quality of the substrate can have important effects on the magnetic properties of the thin film. Unfortunately the films are highly granular leading to pinning at grain boundaries. Lithography is more difficult as the process is on a batch basis and is not always reproducible.

Sputtering is capable of producing layers of extremely low thickness and high uniformity. Generally a magnetic field is applied to the substrate to promote the uniaxial anisotropy in the sputtered thin film. Of course in very thin films the effect of the demagnetising field forces the moment to lie in plane distorting the form of the domain walls. Figure 3 shows a schematic of a DC magnetron-sputtering system. DC sputtering applies a DC voltage between substrate and a target of the source material that is to be deposited. The chamber is filled with an inert gas of positively charged ions, generally Argon. These ions are accelerated towards the target, and knock off atoms from the source material, which on average are directed towards the substrate. The energy of the atoms that are deposited on the substrate will depend on the pressure of the gas.

To sputter insulating materials, such as MgO, a radio-frequency (RF) voltage is applied. By using an asymmetric geometry on the target, substrate holder and chamber shielding the deposition on the substrate is achieved (e.g. Mee and Daniel, 1996). This technique is used to produce thin films for the magnetic recording industry, such as magnetoresistive, GMR, and TMR materials (see section 3.4).
The crystallographic structure of the substrate plays an important role in the growth of the thin film, as it has a determining influence on its crystalline structure. The samples used in this work have been produced by DC-sputtering (Carey et al., 2001). The crystal structure is \( \gamma \)-fcc as shown in section 2.8. Growth in the (111) crystallographic direction is achieved by using Si (111) as seed layer. All samples produced by sputtering are polycrystalline.

![Diagram of a DC-magnetron-sputtering system](image)

**Figure 3** Schematic of a DC-magnetron-sputtering system.

### 2.3 Anisotropy in Ferromagnetic Thin Films

The definition of anisotropy is any physical quantity that exhibits different values depending on the direction of measurement. Magnetic anisotropy refers to the preference for the magnetisation to lie in a particular direction or directions. There are several factors that determine or give rise to magnetic anisotropy, which can be intrinsic to the material, magnetocrystalline, or dependent on the preparation of the sample. The main types of extrinsic anisotropy are: shape, stress, and exchange anisotropy.
2.3.1 Magnetocrystalline Anisotropy

Direct observations of the magnetisation of materials with different crystalline structures, such as bcc Fe, fcc Ni or hcp Co, showed that the material would magnetise more easily when the field is applied in certain directions. In the case of bcc Fe, \langle 100 \rangle are easy directions, and \langle 111 \rangle are hard directions. Removal of the applied field, after saturation is reached, will leave the sample with a higher remanence if the direction of the applied field is an easy direction. This effect is due to the magnetocrystalline anisotropy of the material. CoFe can be either bcc or fcc. In the case of the exchange coupled samples in this thesis, the ferromagnetic layer is composed of fcc-CoFe and the AF layer fcc-IrMn. The easy direction in an fcc structure is the \langle 111 \rangle, \langle 110 \rangle the medium, and \langle 100 \rangle the hard direction [e.g. Jiles, 1998].

Magnetocrystalline anisotropy is due to spin-orbit coupling and the crystalline electric field. In consequence, certain directions of the molecular orbitals will be energetically more favourable and hence preferred, depending on the crystalline field symmetries. When an external field is applied, it will cause a torque on $\mu_s$, the magnetic moment due to spin angular momentum. The response of this moment will depend on the strength of the coupling between the spin, $S$, and the orbital, $L$, angular momenta. Tsunoda and Takahashi (2002) suggested magnetocrystalline anisotropy to be the origin of the magnetic anisotropy of the AF in their study on NiFe/IrMn and NiFe/NiMn on the basis of the single spin model proposed by Meiklejohn and Bean (1956, 1957).

Exchange bias depends on the intrinsic properties of the AF, such as the magnetocrystalline anisotropy and the exchange coupling between $AF$ spins and between the $AF$ and $F$ spins. An increase in these quantities will lead to an increase in the exchange bias (Xi et al., 2003). Magnetocrystalline anisotropy plays a key role in the modelling of exchange bias systems, as it is a driving parameter for the availability of possible energy states of the magnetic moments in the $AF$. The symmetry of the magnetocrystalline anisotropy in the $AF$ depends on the crystalline structure of the material (Cai et al., 2001). The magnetocrystalline anisotropy is sensitive to the
environment, also being influenced by the direction of the magnetic moments at the interface between the $F$ and $AF$ (Nakamura et al., 2000).

### 2.3.2 Shape Anisotropy

L. Néel in 1947 explained the origin of this anisotropy due to the formation of uncompensated free poles at the surfaces of a magnetised material, giving rise to dipole interactions. It can be represented in terms of the external field applied to the sample as a demagnetising field. This field opposes to the state of magnetisation of the sample. It can be expressed as:

$$H_D = -N_D M_S \tag{2.1}$$

where $H_D$ is the demagnetising field, $M_S$ the saturation magnetisation, and $N_D$ the demagnetising factor, which is a tensor function of the shape of the sample. The values of its components vary between 0 and $4\pi$ (cgs). For thin film geometries, the demagnetising factor $N_D$ is 0 in the in-plane directions, and $4\pi$ in the direction perpendicular to the plane (Jiles, 1998). Hence the magnetisation of a magnetic thin film tends to lie in the plane. The use of exchange-biased systems in industry is based on thin film geometries [e.g. Childress et al., 2001].

Giant Magneto-Resistive (GMR) spin valve sensors take advantage of exchange bias phenomenon to set the magnetisation direction of one of its layers. The use of these GMR-spin-valves (Gregg et al., 2002) for read heads for the magnetic storage industry require an increasing miniaturisation of the elements used (See section 3.4). In a GMR-spin-valve the thickness of the AF is the dominant factor in the size of the spin valve. As described in section 3.4 the current used in a GMR device flows parallel to the interface plane (CIP) of the stack. For this configuration the magnetisation direction in the free and pinned layers in the spin valve lie in plane. Reduction of the size of the head is achieved if the configuration changes and the current flows perpendicular to the plane (CPP) of the spin valve. For this configuration the magnetisation also lies in plane.
The demagnetising field plays an important role when the magnetisation of the $F$ is required to stay out of plane as $N_D$ equals $4\pi$ in this direction. In some cases techniques such as adding Pt interlayers between $F$ and $AF$ are used to promote the orientation of the magnetisation to lie perpendicular to the plane [e.g. Sort et al., 2004].

### 2.3.3 Other types of anisotropy

Stress anisotropy is a direct consequence of the magnetocrystalline anisotropy and its influence in the change of the sample’s dimensions when a magnetic field is applied. This is due to orbital reorientation of the magnetic atoms in the direction of the applied field due to energy minimisation. In most of the cases, grain boundaries, or any type of defects, restrict this change to the direction of the orbitals. The energy stored in the system due to this effect is known as magnetoelastic energy.

Studies by Platt et al. (2000), on Si/Co$_{50}$Fe$_{50}$ suggested that the behaviour of coercivity with temperature for CoFe is influenced by small changes in strain due to the large magnetostriction of CoFe. Some anisotropic effects, which affect exchange bias, are those related to field annealing and the effect of deposition in a field. These two effects will be described in chapter 6.2.

Other mechanisms that introduce anisotropy in thin films are ion irradiation [e.g. Fassbender et al., 2004] and impurity addition; both methods can also be applied to tailor exchange bias properties.

### 2.3.4 Anisotropy in soft thin films

The use of exchange bias systems for the fabrication of read heads for magnetic recording systems add some requirements for the materials that form the exchange coupled bilayer. A read head must have low coercivity, low noise and extremely high permeability, $\mu_r$. Nickel-iron alloys and cobalt-iron alloys are the most widely used soft ferromagnets. Ni$_{78}$Fe$_{22}$ has almost zero magnetostriiction and magnetocrystalline anisotropy and has one of the highest permabilities (O’Handley, 2000).
CoFe alloys can have bcc or fcc crystalline structure. See figure 5. These alloys are characterised by a relatively low magnetic anisotropy and substantial magnetostriction. In these materials the grain size is the primary factor that determines the permeability and coercivity (Chin and Wernick, 1980). The value of \( \mu \) is proportional to \( M_s/K_{\text{eff}} \), where \( K_{\text{eff}} \) may include various sources of anisotropy: magnetocrystalline, magnetoelastic, heat treatment with or without an applied field (thermomagnetic anisotropy), cold working or plastic deformation, slip-induced anisotropy (English and Chien, 1967).

For very small thicknesses, much less than a domain wall width, the magnetostatic energy dominates over all other terms and the magnetisation tends to lie predominantly in plane. Canted spin configurations or perpendicular to plane magnetisation can also be found in CoFe thin films. These orientations may appear when there is inhomogeneous magnetisation in the film. In this case, the effective uniaxial anisotropy, \( K_{\text{eff}} \), can be described by a first order, uniaxial, positive term. In the case of \( K_{\text{eff}} \) being negative the magnetisation would lie in plane (O’Handley, 2000).

The nature of grain growth and inter-grain interactions plays an important role in the magnetisation reversal of thin films. Jung et al., (2002) studied Cu/Fe\(_{100-x}\)Co\(_x\), in the range 30 ≤ \( x \) ≤ 50, they concluded that the reduction of \( H_C \) was a result of the reduction in grain size, in accordance with the ripple theory (Hoffmann, 1973). These results are similar to those of Platt et al., (2000) for Fe\(_{50}\)Co\(_{50}\) grown on CoO. Controlling the grain size by change in the sputtering rate, Vopsaroiu et al., (2005) were able to tailor the coercivity in CoFe sputtered thin films. Figure 4 shows room temperature hysteresis loops of Co\(_{60}\)Fe\(_{40}\) samples with different grains sizes after Vopsaroiu et al. (2005).

In the case of random local anisotropy in continuous films, exchange coupled grains reduce the anisotropy by averaging when the length scale, i.e. the grain size, is smaller than the exchange length. Another possible effect is that the magnetisation shows orientational coherence over a length that varies depending on the ratio of the exchange stiffness to the strength of local anisotropies. By comparison of the exchange length and the grain diameter the influence of the exchange and anisotropy energy on the
magnetisation reversal from grain to grain in polycrystalline films can be determined (Craik, 1995).

![Graph](image)

**Figure 4** Room temperature hysteresis loops for Co$_{60}$Fe$_{40}$ (Vopsaroiu et al., 2005)

The samples studied in this work contain a Co$_{90}$Fe$_{10}$ film 10 nm thick for the ferromagnetic layer. The crystalline structure of CoFe alloys is shown in figure 5. Hence Co$_{90}$Fe$_{10}$ has fcc ($\gamma$) structure up to 1000°C. CoFe alloys have a $T_C$ of the order of 1253K (Kaye and Laby, 1986). The exact value of $T_C$ for Co$_{90}$Fe$_{10}$ is not known and may vary when the material is in thin film form.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mean grain size D (nm)</th>
<th>Coercivity $H_C$ (Oe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>7.20</td>
<td>12</td>
</tr>
<tr>
<td>A2</td>
<td>14.1</td>
<td>20</td>
</tr>
<tr>
<td>A3</td>
<td>17.1</td>
<td>120</td>
</tr>
<tr>
<td>A4</td>
<td>20.0</td>
<td>126</td>
</tr>
<tr>
<td>A5</td>
<td>21.2</td>
<td>117</td>
</tr>
<tr>
<td>A6</td>
<td>24.1</td>
<td>108</td>
</tr>
<tr>
<td>A7</td>
<td>26.3</td>
<td>121</td>
</tr>
</tbody>
</table>

**Table 1** Data relative to the hysteresis loops for Co$_{60}$Fe$_{40}$ from figure 4 (Vopsaroiu et al., 2005)
2.4 Interactions in Ferromagnetic Thin Films

Nanoscale dimensions play an important role in composites of particles as well as thin films. At this scale, phenomena such as magnetostatic effects, exchange anisotropy, single domain behaviour, superparamagnetism, random anisotropy and exchange coupling play an important role in the understanding of the magnetic behaviour of these systems (Koch, 2002). In this section the different forms of interaction between the magnetic moments of atoms from different sites of ferromagnetic thin films and the influence these have on bulk magnetic behaviour in thin films are discussed.

2.4.1 Exchange Interaction. Direct Exchange

The tendency of electron spins to align parallel or antiparallel can be understood via the exchange interaction. This interaction can be quantified through the exchange strength, $J$, or exchange integral, which depends on the orbital overlap and is a result of the requirement for the electrons to be indistinguishable (Heisenberg, 1924). The values taken by $J$ depend on the interatomic distance related to the radii of the orbitals of the

Figure 5. Phase diagram of CoFe alloys (Bozorth, 1951)
interacting electrons. For $J > 0$ the spins of these electrons will align parallel while for $J < 0$ they will align antiparallel, resulting in ferromagnetism and antiferromagnetism respectively (e.g. Cullity, 1972). The exchange interaction is responsible for the magnetic state of the materials. For localised electrons on fixed ions, the Hamiltonian is

$$H = -2\sum_{i<j} J_{ij} \vec{S}_i \cdot \vec{S}_j \quad (2.2)$$

Depending on the value of the exchange integral, $J$, the spins will lie parallel, leading to ferromagnetism when $J > 0$, or will lie antiparallel when $J < 0$, leading to antiferromagnetism. The magnitude of the sign of $J_{ij}$ depends on the separation of the spins.

This form of exchange applies for systems with localised wave functions such as Mn and Cr, which are antiferromagnetic, or MnAs, MnSb, which are both ferromagnetic (Bozorth, 1951). However, Heisenberg exchange does not explain how a magnetic moment of an atom from one site couples to another from another site in the case of rare-earth metals. The magnetic moment of atoms of this type is due to electrons in the partially filled 4f orbitals, which are very localised and too small, typically 10% of the interatomic spacing, for electron hopping from one site to another. For these cases indirect coupling applies [e.g. O’Handley, 2000].

### 2.4.2 Indirect Exchange

This indirect exchange or RKKY interaction [e.g. Kittel, 1968] describes the coupling in rare-earth metals and alloys. For these materials the magnetic moments are given by partial filling of the highly localised 4f states. The radius of a 4f state is about 10% of an interatomic spacing. Hence, there is no 4f-orbital overlap between neighbouring atoms. The interaction between the magnetic moments at different sites is provided by mediation of conduction electrons. The 4f magnetic moment at one site polarises the conduction electrons in the 6s orbital, whose wavelength is quite extended and therefore these electrons communicate their spin polarisation state to other sites.
The conduction electron spin density is damped and oscillates with the distance from the local moment. Added to the spin polarisation of the conduction electrons is the fact that the amplitudes of the oscillations have different periods depending on the crystallographic directions. Hence, neighbours at different distances may experience different signs of exchange coupling. Hence, the RKKY interaction can give rise to ferromagnetism, antiferromagnetism, or helimagnetism but is weaker than interatomic exchange coupling in 3d metals and weaker than super-exchange in 3d magnetic oxides.

2.4.3 Super-exchange

Super-exchange is a variation of indirect exchange which applies mainly to 3d magnetic oxides with the rocksalt structures, spinel, garnet and perovskite oxides. The mechanism of super-exchange can be described as shown in figure 7. It shows two transition metal ions, e.g. Mn$^{2+}$, which are separated by an oxygen ion O$^{2-}$. The p orbital on the oxygen, filled in the ground state, can exchange an electron with each of the 3d orbitals of the Mn$^{2+}$. This gives divalent ionic bonding but some hopping is allowed. The positive and negative phase parts of the orbitals are shown in the figure.

To be in the ground state, the 3d and p orbitals must exchange electrons, so some excited states must be partially occupied. When one of the electrons is excited into one of the empty 3d states, the other electron, which is of opposite spin can exchange with a
3d orbital of the other neighbouring transition metal ion coupling to the p orbital. In the case of the two 3d orbitals having the same ground-state electronic configuration, this will result in antiferromagnetic spin coupling between the two M$^{2+}$ ions. Therefore, the basis of this antiferromagnetism is in the symmetry of the 3d and the p orbitals and the conservation of the spin polarisation for electron hopping [e.g. O’Handley, 2000].

The most important consequence of this type of exchange is that it is highly anisotropic. The bonding gives a direction for the coupling, which is very strong. Also because the moments are very small (atomic), and cancel, an applied field cannot couple to them so they are hard to reverse. In many materials the spin alignment is not perfectly co-linear. This gives rise to spin canting and therefore to a measurable susceptibility. In complex oxides, e.g. Fe$_3$O$_4$, FeO, Fe$_2$O$_3$, the cancellation is imperfect, resulting in ferrimagnetism. In the case of Fe$_2$O$_3$ the structural effect is dominant so different phases of Fe$_2$O$_3$ are antiferromagnetic or ferromagnetic. Due to direct exchange, the behaviour of alloys can give similar properties, e.g. Néel ferrimagnets.

![Figure 7](image)

**Figure 7** (a) Schematic of p and d orbitals. (b) Schematic of the electronic state of the p and 3d orbitals.

### 2.5 Magnetic Domains and Domain Walls

#### 2.5.1 Néel walls.

In bulk materials a domain wall consists of a region of spin rotation. The width of the wall is determined by the balance between exchange and anisotropy energies. However in thin films magnetostatic energy is also important, as high anisotropy is required to orient spins perpendicular to the plane. Thus the rotation of the spins must lie in the plane forming a special form of domain wall known as Néel wall (see figure 8). This
type of wall is characterised by the rotation of spins inside the walls in the plane of the surface. The magnetostatic energy would be smaller at the internal interface of the wall.

Figure 9 show the thickness dependence of the wall energy density for Bloch and Néel walls (a), and the domain wall width as a function of the film thickness for Bloch and Néel walls. Bloch walls can transform into the Néel walls near a surface in materials with film thickness larger than the thickness of a Néel domain wall (O’Handley, 2000). In general, domain wall widths change with film thickness.

![Schematic of a Néel wall. The magnetisation rotates across the domain wall 180° in the surface plane.](image)

**Figure 8** Schematic of a Néel wall. The magnetisation rotates across the domain wall 180° in the surface plane.

![Graph showing thickness dependence of the wall energy density for Bloch and Néel walls](image)

**Figure 9** (a) Thickness dependence of the wall energy density for Bloch and Néel wall, (b) domain wall width as a function of the film thickness for Bloch and Néel walls. (O’Handley, 2000)
For 30nm thick Co$_{50}$Fe$_{50}$, typical domain wall widths vary between 8 and 40 nm for 180° domain walls (Platt et al., 2000). As shown in the figure 9, the energy of Néel walls increase and the domain wall widths decrease with increasing film thickness.

2.5.2 Domain Formation

Cobalt-Iron, the ferromagnet used in the samples studied, is characterised for having a much higher magnetocrystalline anisotropy than NiFe, the other ferromagnet that is extensively used for exchange bias systems. Because of its high magnetocrystalline anisotropy, hysteresis loops of exchange-biased systems are characterised by a high saturation magnetisation and high coercivity. See figure 10.

![Bohr magneton numbers in CoFe alloys](image)

Figure 10 Bohr magneton numbers in CoFe alloys. (Bozorth, p.195, 1951)

As seen in chapter 2, section 3.4, the value of the coercivity of a CoFe layer depends on the grain size. As the magnetocrystalline anisotropy is high, the reversal of magnetisation of these alloys is characterised by domain nucleation leading to a high squareness in the hysteresis loops, as shown in figure 11.
The formation of multidomain structures is governed by energy minimisation, which involves different energy densities: the Zeeman energy, the exchange energy, and the magnetostatic energy. To describe coercivity and irreversibility it is necessary to take into account the spatial variation of $M$ and domain walls, each having different energy densities.

A single domain state has high magnetostatic energy due to the formation of poles at its ends. In the case of cubic anisotropy systems such as, Fe, the formation of 90° closure domains allows the flux to be contained within the sample reducing the magnetostatic energy. In the case of uniaxial anisotropy materials, the formation of closure domains will increase the anisotropy energy and cause elastic energy due to strain incompatibilities.

The materials studied in this work include Co$_{90}$Fe$_{10}$ as $F$ layer and Ir$_{30}$Mn$_{70}$ for the $AF$ layer. The formation of domains and domain walls in polycrystalline thin films such as CoFe has great importance in the study of magnetisation reversal processes on CoFe/IrMn systems. The fact that these films are composed of grains makes the study...
of the reversal properties complicated due to the nature of the grain growth and the interactions between grains (Platt et al., 2000).

Domain observation by Lorentz transmission electron microscopy (TEM) of magnetisation reversal on single-layer 30nm thick Co$_{50}$Fe$_{50}$, grown on 10nm thick SiO$_2$, shows the formation of ripple domain structures, which are almost stationary up to applied fields close to the coercive point as shown in figure 11 (Platt et al., 2000).

There is an increase in the contrast of the ripple structure with the reduction of the applied field towards negative values showing more pronounced spatial variation of magnetisation. At $H_C$, an 180° domain wall was observed to sweep across the film. Hence, low mobility domain walls and the ripple structure dominate the reversal of CoFe, and therefore higher coercivity values would be expected than for the case of higher mobility. When the CoFe is exchange coupled to IrMn the magnetisation reversal mechanisms change depending on several factors such as the thickness of the IrMn layer, the direction of the applied field, as shown in figure 12 (Gogol et al., 2002), and the crystallographic orientation of the bilayers (Sato et al., 2005). Magnetisation ripple can be induced in the CoFe layer by the IrMn as reported by Pokhil et al. (2002).

Sato et al. (2005) studied epitaxial (110) and (001) Ir$_{25}$Mn$_{75}$/Co$_{70}$Fe$_{30}$ bilayers. For (110) the reversal process is by movement of 180° domain walls separating large domains. In the case of (001), the magnetisation reversal proceeded by nucleation of small domains formed homogeneously across the whole film. Gogol et al. (2002) using Fresnel mode Lorentz microscopy, observed different domain features in the Co$_{90}$Fe$_{10}$ layer depending on the thickness ($t_{AF}$) of the Ir$_{25}$Mn$_{75}$ layer. For $t_{AF}$ = 2nm, ripple structures were observed to intensify as the applied field was swept to lower values. Domain wall nucleation during reversal of the applied field and formation of 360° wall loops were also observed. For $t_{AF} \geq 6$nm, the reversal of magnetisation was by nucleation events and a high density of walls separating submicron-sized domains. Magnetisation ripple was observed to behave similarly for lower $t_{AF}$ increasing, in contrast with decreasing applied field, with a high dispersion or a high density of low angle walls. The domain structure evolved to domain walls lying almost parallel to the applied field. During the return of the field cycle the magnetic structures were similar.
2.6 Magnetisation Ripple

This type of magnetisation state is found in polycrystalline films that are exchange coupled, e.g. Permalloy. In such materials domain walls can be wider than the grains (50nm). The reason for this to happen lies in the anisotropy being weak, so the rotation per spin is low. The walls can be as wide as 2 to 50µm, being almost visible to the naked eye.

The anisotropy is never uniform. The direction of the anisotropy easy axes varies from one place to another in the film, or the magnitude of the anisotropy varies due to crystal effects and defects. This is called anisotropy dispersion. As a consequence of this, the direction of the local magnetisation varies from one site to another even within a domain (figure 13). The non-parallel magnetisation vectors add extra energy to the system. In addition, the demagnetising field at the ends of the sample, especially in small elements, leads to the divergence of the magnetisation just inside the sample. This has an effect in the creation of free poles even within a domain, creating stray fields and magnetostatic energy. As an example, a Permalloy thin film can be demagnetised by saturation in the hard direction. However, the anisotropy dispersion and the divergence of magnetisation lead to this ripple effect so the magnetisation is never uniform.
Chapter 2: Magnetism of Thin Films

Figure 13 Fresnel images of as deposited Ir$_{20}$Mn$_{80}$(2nm)/Co$_{90}$Fe$_{10}$(4nm) subjected to various magnetic fields. The applied field was parallel to the field applied during deposition. (After Gogol et al., 2002).

Magnetisation ripple reduces the magnetostatic energy for a marginal increase in exchange energy. This type of magnetisation is characterised by a wavelike variation of the magnetisation. This ripple effect is depicted in figure 14(a), where the film is in a single-domain remanent state after saturation in an easy direction. If a field is applied transverse to the easy direction, as shown in 14(b), the magnetisation ripple persists. Its relation to the local easy axis direction is indicated. When the hard-axis field is removed half of the magnetic moments rotate clockwise while the other half rotate counter clockwise giving rise to the domain structure shown in 14(c). The vectors indicated in (b) and (c) apply only to a narrow horizontal strip. The ripple persists in the vertical direction in each domain (e.g. Cullity, 1972).
Hoffmann (1968) provided a review of the theory of magnetisation ripple in thin films. The theory included measurable parameters such as grain size, stresses and magnetostriction. The linear magnetisation ripple described the cases where the stray fields are transverse to the direction of the magnetisation. This makes the coupling of the spins to be perpendicular to the mean magnetisation direction. When the stray field is along the direction of the mean magnetisation a non-linear theory is necessary. Longitudinal stray field leads to the blocking of the magnetisation ripple (Hoffmann, 1966), which defines a state of small angle ripple domain walls (5°-10°) which is the beginning of a metastable state. This blocking state can be removed with small ac fields.

![Figure 14 Schematic of domain formation in a thin film after magnetisation in the hard direction. (Prutton, M., 1964)](image)

However the Hoffmann’s ripple theory predicts a coercivity dependence on the median diameter of the grains proportional to $D^{0.5}$, where $D$ is the median grain diameter. This prediction does not agree with the results obtained by Vopsaroiu et al. (2005), which are shown in figure 15. Their results show a coercivity dependence on $D$ that has three different regimes. They explain their data on the basis of the random anisotropy model (Alben et al., 1978). For very small grains, smaller than the exchange length, $L_{ex}$, $H_C$ varies with $D^6$. For $D \leq L_{ex}$, $H_C$ is constant. And for $D$ larger than the domain wall width, $H_C$ varies with $D^4$. 
Figure 15: Coercivity dependence of grain size for CoFe (20nm). (Vopsaroiu et al., 2005)

Figure 16: Co₉₀Fe₁₀(4nm)/Ir₂₀M₈₀(14nm) Fresnel mode Lorentz microscopy image. The applied field is parallel to the direction of the field applied during deposition (Gogol et al., 2002)

For exchange coupled Co₉₀Fe₁₀(4nm)/Ir₂₀M₈₀(14nm) Fresnel mode Lorentz microscopy images are shown in figure 16. The sample was previously annealed for 20 s at 300 °C on a field of 500 Oe (Gogol et al., 2002). Inset images (a) to (e) are from the magnetising branch of the hysteresis loop. Images (f) to (g) correspond to the demagnetising branch. When the field is reduced there is an increase in the intensity of magnetisation ripple. In 16(e) the domain structure is more irregular with domains larger than those observed in (a). In fact the mean orientation of domain walls at 16(e), which corresponds to point 3 in the hysteresis loop in figure 17, and at 16(g) (point 7 in
the hysteresis loop) are very different. Gogol et al. interpreted this difference in terms of the “natural angle” arising from different energy landscapes for the $F$ to pass through in a magnetisation cycle (Stamps, 2000).

![Hysteresis Loop Diagram](image)

**Figure 17** Seven point model hysteresis loop.

### 2.7 Antiferromagnetism

The role played by the $AF$ is of critical importance in the understanding of the exchange bias phenomenon. Up until the 1980’s $AF$ materials were thought of as being of academic interest only in the study of the exchange interaction. Furthermore, there were no technical applications of $AF$s due to its zero net magnetisation. Even though the exchange bias phenomenon was discovered in 1956, in the quest for high coercivity particle magnets, it was not until the discovery of Giant Magneto-Resistance (GMR) in multilayers when $AF$ materials and exchange bias became of great technological importance.

The exchange interaction was briefly described in section 2.4.1. Depending on whether the exchange integral between atoms, $J$, is positive or negative, their magnetic moment would lay parallel or antiparallel, giving rise to ferromagnetism or antiferromagnetism.
Figure 18 Schematic of the Bethe-Slater curve (e.g. Cullity, 1972).

Figure 18 shows the variation of the exchange integral with the ratio \( r_a/r_{3d} \), where \( r_a \) is the atomic radius, and \( r_{3d} \) is the radius of its 3d shell of electrons. When this ratio is large the exchange integral is small and positive. When this ratio decreases, and the 3d electrons get closer, \( J \) is positive and gets stronger, leading to ferromagnetism, and then decreases to zero. If the spins are taken even closer, the exchange integral becomes negative and the spins have to lie antiparallel, giving rise to antiferromagnetism. Known the ratio \( r_a/r_{3d} \) this curve can be applied to different elements. A few pure metals are antiferromagnetic. Figure 18 shows that Mn is AF. Mn is antiferromagnetic below 100K, above this temperatures it is paramagnetic. The exchange integral also controls the Néel temperature. There are many oxides that are AF, having \( r_a/r_{3d} \) values much greater than these metals. The mechanism by which these oxides are AF is the super-exchange interaction. (See section 2.4.3).

Most of the studies of exchange bias in thin films with AF oxides involve CoO or NiO. All these oxides are fcc above \( T_N \), with a slight distortion below \( T_N \) (Battle et al., 1979). The rocksalt structure shown in figure 19 is just two interweaved fcc structures with moments shown on the magnetic ions. Many oxides have this structure, e.g. NiO, CoO. The Néel temperature for these oxides decreases with decreasing film thickness (Wijn, 1991). Spins in any (111) plane are all parallel. All spins on adjacent planes are antiparallel to the spins in the reference plane, and parallel within the plane. These planes are too far apart for direct Heisenberg exchange. This arrangement is due to super-exchange interaction between nearest neighbouring ions along <100> via the intervening oxygen atom. The layer thickness dependence of \( T_N \) for CoO and NiO were obtained from heat capacity measurements (Abarra, et al. 1996). Both reach bulk \( T_N \) for
10nm thickness. Because of the variation of $J_{ij}$ with ionic spacing it is possible to produce many alloys that are antiferromagnetic. Changing the interatomic distance $r_{ij}$, especially in alloys of Mn, leads to AF behaviour. Examples of AF alloys are MnSe, FeRh, NiMn. Technologically important in Exchange Bias are FeMn, IrMn, and PtMn.

Figure 19 “Chemical and magnetic structure of Ni$_x$Co(1−$x$)O antiferromagnetic materials. The shaded spheres represent the magnetic metal atoms, and the unfilled spheres represent the oxygen atoms”. (After Berkowitz and Takano, 1999)

Metallic AFs are preferred to AF oxides for most applications due to their higher Néel temperatures and anisotropies. Despite this and that studies of exchange couples with metallic AFs are more numerous, it seems that there is less understanding of the exchange bias phenomenon involving metallic AFs. This is due to several factors, such as deviations of the crystal structures from the cubic symmetry, more complex spin structures than in the case of oxide AFs and higher $T_N$. This means that in some cases the field cooling procedure might damage the interface quality of the exchange coupled structure due to interdiffusion of the layers or grain growth (Berkowitz and Takano, 1999).
Table 2 Common antiferromagnets. (Nogués et al., 1999)

<table>
<thead>
<tr>
<th>Substance</th>
<th>Oxides and Metals</th>
<th>Structure</th>
<th>$T_N$ (K)</th>
<th>$\Theta T_N$ (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>MnO</td>
<td>fcc</td>
<td>122</td>
<td>610</td>
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<tr>
<td>Cr</td>
<td>bcc</td>
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2.8 Crystalline and Magnetic Structure of $\text{Ir}_30\text{Mn}_{70}$

The most common metallic AF used in industry for the fabrication of spin-valve sensors is IrMn. This is due to fabrication requirements such as high resistance, high GMR signal, low total thickness and high exchange bias (Childress et al., 2001). The samples studied in this work contain $\text{Ir}_30\text{Mn}_{70}$. The phase diagrams of IrMn alloys is shown in figure 20.

From figure 20, according to composition considerations, $\text{Ir}_30\text{Mn}_{70}$ should have a structure of the ordered AuCu$_3$-type IrMn$_3$ (L1$_2$-Pm3m) (Raub and Mahler, 1955). According to Yamaoka (1974) IrMn alloys in the composition range of 14 to 31 at% Ir, quenched from 1250-1350K are nominally disordered fcc. In the composition range of 8 to 13 at% Ir a disordered fct AFs with axial ratio $c/a>1$. The AuCu$_3$-type IrMn$_3$ structure is only found in alloys with approximately 25% Ir annealed below 1000K.

Yamaoka et al. (1971, 1974) extended the study of γ-phase Mn to IrMn alloys in both ordered and disordered states. Figure 21 shows the magnetic phase diagram of γ-phase $\text{Ir}_x\text{Mn}_{1-x}$ alloy obtained by magnetic susceptibility measurements. As shown by Umebayashi et al. (1966) there are three stable spin configurations which minimise the exchange interaction energy and the anisotropy energy of the fcc structures with identical spins. The three configurations are shown in figure 22.
Figure 20 Phase diagram of IrMn alloys (Binary Alloy Phase Diagram, American Society for Metals, p1421 (1986)).

Figure 21 Magnetic phase diagram of γ-phase Ir₃Mn_{1-x} alloy system. After Yamaoka (1974)
FeMn alloys have spin configuration (3), (Umebayashi et al. 1966). Neutron diffraction studies (Yamaoka et al., 1971 and 1974) on single crystals showed that the most probable spin structure for the γ-phase IrMn alloys is configuration (1) which is depicted in figure 22, showing the two sublattice spin structure.

According to these data our Ir30Mn70 layer is nominally disordered fcc (γ) phase. For alloys with this structure, with the percentage of Ir varying from 14 to 31, the $T_N$ varies from ~ 600 to 750K with increasing Ir (Yamaoka et al., 1974). The average spin on each (002) plane is aligned parallel along the c-axis, with alternating opposite sign in adjacent (002) planes. The average moment per atom is $-2.5 \mu_B$ up to $-20$ at% Mn, and falls to $-0.8 \mu_B$ at $-25.6$ at% Mn (van der Heijden et al., 1998).

Deposition temperature conditions may cause the presence of ordered IrMn3 within the disordered fcc phase, as reported by Imakita et al. (2004). They studied Ir$_{27}$Mn$_{73}$-Co$_{70}$Fe$_{30}$ bilayers. The Ir$_{27}$Mn$_{73}$ was deposited at substrate temperatures up to 200°C, the Co$_{70}$Fe$_{30}$ was deposited after cooling to room temperature and then the bilayer was annealed at 400°C for 1 hour in 1kOe. Their results showed superlattice lines in <111> textured Ir$_{27}$Mn$_{73}$ deposited at temperatures higher than 100°C, indicating the presence of ordered IrMn3. Changes in the temperature of deposition and annealing resulted in variations of the interfacial coupling energy being a maximum ($\Delta \sigma = 1.3$erg/cm$^2$) when the deposition of Ir$_{27}$Mn$_{73}$ was at 170°C the annealing at 320°C. They suggested that these superior properties were due to the increased presence of ordered IrMn3. The effects of annealing in exchange biased couples will be described in more in detail in section 6.2.4.
2.9 Superparamagnetism

One of the basic features of the way that exchange bias is conceived in this work is the description of the AF as an assembly of grains. This conception of exchange bias is based on a model proposed by Fulcomer and Charap (1972). This model is described in chapter 4. The AF grains are thought to be in a single domain state, each of which interacts with the F through exchange interaction. The grains are distributed in size, also the anisotropy easy axis directions of each grain are distributed. The grains are susceptible to thermal activation in the orientation of its moment at the interface of the AF-F bilayer above their blocking temperature, giving rise to superparamagnetic behaviour.

2.9.1 Superparamagnetism in single domain particles

When atoms assemble to form nanoparticles these can be in a single domain or multidomain state. The former state will prevail if its energy is less than that required to form a domain wall. The size of particle that forms a single domain state depends on the anisotropy (Frenkel and Dorfman, 1930). Equation 2.3 relates the anisotropy to the critical radius, \( r_c \), of a ferromagnetic particle to be in a single domain state, where \( A \) is...
the exchange stiffness, \( K \) the unidirectional anisotropy, and \( M_S \) the saturation magnetisation (O’Handley, 2000).

\[
r_c \approx 9 \frac{(AK)^{1/2}}{\mu_0 M_S^2}
\]  

(2.3)

Single domain magnetic particles are of great importance for magnetic recording media. Magnetic nanoparticles with uniaxial anisotropy behave as a two state system. They are susceptible to magnetisation reversal under the influence of temperature in a given time of observation at zero applied field. This occurs when the ambient thermal energy is large enough to overcome the energy barrier to magnetisation reversal in the time of measurement, \( \Delta E \), which in zero applied field is given by the anisotropy energy, \( KV \) (Néel, 1949). In this case the particles are said to be in a superparamagnetic state (Bean and Livingston, 1959). The critical volume below which this behaviour is observed depends on the time of measurement relative to the relaxation time, \( \tau \), which is the time it takes for the magnetisation to decay to 1/e or 37% of its initial value (Néel, 1949),

\[
\tau^{-1} = f_0 \exp \left( \frac{-\Delta E}{k_B T} \right)
\]

(2.4)

where

\[
\Delta E = KV \left( 1 - \frac{H}{H_K} \right)^2
\]

(2.5)

\( f_0 \) is the attempt frequency which is taken to be \( 10^9 \)Hz (Kneller et al, 1966), \( V \) the particle volume, \( k_B \) Boltzman’s constant, \( K \) the anisotropy energy density and \( T \) the temperature. \( H_K \) is the anisotropy field which for aligned easy axes is given by

\[
H_K = \frac{2K}{M_S}
\]

(2.6)
The size at which a particle becomes superparamagnetic is found when $KV$ is less than a multiple of $k_B T$, for a given attempt frequency $f_0$. For $f_0 = 10^9$ Hz (e.g. Kneller, 1969), and an observation time of 100s, the superparamagnetic criterion

$$KV = 25k_B T.$$  \hspace{1cm} (2.7)

This means that when an applied field reduces the energy barrier $KV$ to less than or equivalent than $25k_B T$ the magnetisation of the nanoparticle will reverse in 100 seconds. This condition is applicable for the determination of the superparamagnetic limit on the particle size of recording media systems. For these systems the observation time is considered to be $\tau = 10$ years leading to a proportionality constant of 40. Particles with volume bigger than the critical volume $V_p$ exhibit stable magnetic behaviour and are termed "blocked" particles,

$$V_p = \frac{25k_B T}{K}. \hspace{1cm} (2.8)$$

Accordingly, given a particle volume, it will become "blocked", when its temperature is less than the blocking temperature, $T_B$, given by

$$T_B = \frac{KV_p}{25k_B}. \hspace{1cm} (2.9)$$

Superparamagnetic systems exhibit no hysteresis and saturate in modest applied fields and their magnetisation can be described by the Langevin function $L(\alpha)$

$$L(\alpha) = \coth \alpha - \frac{1}{\alpha}, \text{ where } \alpha = \frac{\mu H}{k_B T}. \hspace{1cm} (2.10)$$

Hence such systems exhibit superimposition of loops measured at different temperatures when they are plotted as a function of $H/T$. These phenomena have been observed by Bean and Jacobs (1956). Ferromagnetic systems of nanoparticles of these characteristics will behave according to the Langevin function when their volume is less
than $V_p$. In the case of "blocked" particles, those with volume greater than $V_p$, will behave as ferromagnetic systems. Particles with a volume equal to $V_p$ will experience time dependence according to (Néel, 1949)

$$\frac{M(t)}{M(0)} = \exp\left(-\frac{t}{\tau}\right).$$

(2.11)

Accordingly, systems of nanoparticles with distributed sizes, or volumes, or distributed anisotropies will show a distribution of blocking temperatures. Substitution of equation 2.4 in equation 2.11 gives

$$M(t) = M(0) \exp\left(-t \frac{KV}{k_B T}\right).$$

(2.12)

Street and Woolley (1949) obtained analytical solutions for equation 2.12 for the special case of $t_0 >> 1$, and $f_0$ independent of the energy barrier, which to a first approximation is the case of the systems of nanoparticles: $\Delta E = KV$, and $f_0$ is independent of $K$ and $V$. Hence, at first approximation the change of magnetisation, $M$, in time $t$ is given by

$$\Delta M = S \ln t + \text{const}.$$  \hspace{1cm} (2.13)

The variation of $S$ with applied field for ferromagnets was considered by Gaunt (1976). Using a phenomenological theory, Gaunt predicted the maximum value of $S$ to be at $H = H_C$ as observed by Street and Woolley (1949). O'Grady et al., (1981) showed that eq.2.13 is generally true and gave a general analytic solution for eq.2.12 for systems containing a distribution of particle sizes. Uren et al., (1988) showed that the maximum value of $S$ occurs at $H_n$, the remanent coercivity, obtained from DC demagnetisation remanence measurements. According to eq. 2.13, the magnetisation decay is given by

$$M(H,t) = M(H,0) \pm S(H) \ln t.$$  \hspace{1cm} (2.14)

Hence,

$$S(H) = \frac{dM(H)}{d \ln t},$$  \hspace{1cm} (2.15)
the magnetic viscosity coefficient, which is maximum when the applied field is close to the coercivity. This law has been shown to be a first order approximation of a series expansion (el-Hilo et al.1993). Only if the distribution of energy barriers $f(\Delta E)$ remains constant over the time of measurement the $\ln t$ law is followed. Otherwise, non-linear variations of magnetisation with $\ln t$ are observed. The variation will be convex when $f(\Delta E(t))$ is increasing with time and concave when $f(\Delta E(t))$ is decreasing with time.

![Energy barrier distribution](image)

**Figure 24** (a) Schematic of a symmetric energy barrier distribution showing three different values of energy barriers to reversal. (b) Non-linear time dependence of the magnetic viscosity coefficient shown at three different stages in the energy barrier distribution.

The magnitude of the non-linearity was found to increase when the energy barrier distribution becomes narrow. This is a result of $S(H)$ being a function of $f(\Delta E)$ (Gaunt, 1986). At a critical energy barrier $\Delta E_C$ activated by a field $H$,

$$S(H) = 2M_s k_B T f[\Delta E_C(H)]$$

This explains the relation between the magnetisation time dependence and the energy barrier distribution.
2.9.2 Superparamagnetism and antiferromagnetism in exchange biased systems.

As shown in section 2.9.1, Néel’s study (1949) of the reversal of ferromagnetic particles by thermal activation showed that the relaxation time of a system of particles with their easy axis aligned parallel to the direction of the applied field which reverse coherently under thermal activation is given by the Arrhenius-Néel law, obtained by substitution of eq. 2.5 in eq. 2.4,

\[ \tau^{-1} = f_0 \exp\left(\frac{-\Delta E}{k_BT}\right) = f_0 \exp\left(\frac{KV(1 - H/H_K)^2}{k_BT}\right). \]  

(2.17)

where \(\Delta E\) is the height of the energy barrier to reversal, and \(f_0\) is taken to be \(10^9\) Hz, (Kneller et al, 1966). For a relaxation time of 100s, the superparamagnetic criterion is given by

\[ \Delta E = KV(1 - H/H_K)^2 = 25k_BT \]  

(2.18)

Hence, a distribution of particle sizes, anisotropy orientation, or coupling orientation give rise to a distribution of energy barriers. Accordingly, a distribution of blocking temperatures would appear given by,

\[ f(T_B) = k_B^{-1}f(\Delta E/25) \]  

(2.19)

For the case of sputtered, polycrystalline exchange biased systems such as the ones studied in this work, magnetisation switching similar to that described by equation 2.19 would be expected. The origins of the distribution of energy barriers to reversal will be discussed in section 6.2 and 6.3 in which the possible origins of the training effect will be investigated. To give an idea of the complexity of this task, it should be acknowledged the number of different models proposed for exchange bias, some of which are briefly described in chapter 4.
A typical hysteresis loop for an exchange-biased system has been shown in figure 17. Different stages on the hysteresis loop are marked with numbers according to the 7-point description (Goodman et al., 2001), which is described in section 2.11.

![Hysteresis Loop](image)

Figure 25 Decay of the initial magnetisation.

According to equation 2.7, after 100s at a temperature $T$, the initial magnetisation will decay to 37% of its initial value, after $10^3$s the rate of reversal will decrease rapidly with increasing time.

![Distribution of Energy Barriers](image)

Figure 26. Schematic of a distribution of energy barriers to reversal. showing an energy barrier to reversal $25k_B T$. Grains with $\Delta E_C = 25k_B T$ will undergo time dependent reversal. Those with lower $\Delta E_C$ will reverse, and those with higher $\Delta E_C$ will remain “blocked”.

Chapter 2: Magnetism of Thin Films

In the samples studied the granular AF layer, fcc IrMn \((a = 3.76\text{Å})\), is grown epitaxially on the F, fcc CoFe \((a = 3.6 \text{ Å})\), with a magnetic structure as shown in figure 23. Hence there are two easy axes available for the magnetisation of the AF spins. If they were not grown epitaxial there would be three easy axes. When the field applied to the exchange biased system reverses the direction of the F layer (points 2-4 in figure 17), the effect of the reversal of the exchange field on the AF grains and thermal activation promote the reversal of the AF ordered state.

As time progresses, the AF spins will become more decoupled from the anisotropy axes. When the thermal energy equals \(\Delta E = KV\), the spins decouple from their easy axes although they are still antiferromagnetically coupled to each other. This occurs when the temperature of the system reaches \(T_B\) for the grain, given by equation 2.6. If the temperature is increased to \(T_N\), the spins become decoupled from each other and lose their antiferromagnetic order.

The fact that the AF grains become "unblocked" and have superparamagnetic behaviour over \(T_B\), is used in the work developed in this thesis to modify the amount of AF material that remains in its original state and to "reset" the state of the AF after measurement. See chapter 6, section 2.

2.10 Domains in AF thin films

Systems exhibit superparamagnetism when the thermal energy \(25k_BT\) overcomes the energy barrier \(KV\). Néel derived his equation for superparamagnetism independently of the nature of the energy barrier involved. For example, in the case of systems subject to domain wall pinning, given that the energy barrier, \(\Delta E\), to unpin the domain walls is equal or less than \(25k_BT\) a similar relaxation would take place in 100s. Hence, superparamagnetism and time dependence would occur in CoFe or in IrMn if there are pinned domain walls.

Several authors have observed AF domains in thin films (see Stöhr et al., 1999, Scholl et al., 2000, and Hillebrecht et al., 2001). Observation of domains in exchange biased systems in both the F and AF layers has been claimed by Nolting et al., (2000) on
LaFeO$_3$/Co grown epitaxially by molecular beam epitaxy (MBE). X-ray magnetic dichroism spectro-microscopy measurements showed that the alignment of the $F$ spins is determined, domain by domain, by the spin directions in the $AF$ as shown in figure 28.

![Figure 28](image.png)

**Figure 28** Images of the domain structure in the $AF$ layer (a) and in the 12Å thick $F$ layer (b) grown on top of the same substrate region. Spectra shown recorded by PEEM. (Nolting et al., (2000))

Sputtered exchange coupled bilayers are granular. The grains that form each layer can be in a single domain state or contain domain walls. Domains can be also be formed by exchange coupled grains, i.e. magnetic domains can traverse the grain boundary apparently undisturbed as observed by Hubert and Schäfer (1998) on Fe polycrystalline thin films. As far as the author is aware, there has not been observed magnetic domains in polycrystalline $AF$ thin films.

Shull et al., (2003), using a magneto-optic indicator film technique (MOIF), observed that the domains in the $AF$ are stationary during magnetisation reversal of the $F$. In FeMnC/FeMn exchange coupled systems that showed zero exchange bias as deposited, they concluded that domain wall movement in the $F$ does not imply domain wall movement in the $AF$. The resulting domain structure is an exchange spring parallel to the $AF/F$ interface created during the reversal of the $F$ magnetisation.
The stabilisation of the AF domains has been claimed to be due to interface roughness (Malozemoff, 1987, 1988a, 1988b), or due to volume defects (Keller et al., 2002) such as grains, twin boundaries, or crystal defects. All of these are sources for domain wall pinning, which give rise to energy barriers. Changes in the magnetic state in the AF can arise within 100s by processes such as domain nucleation or wall motion when the energy barrier to unpin a domain wall is $\leq 25k_B T$, or by a change of the magnetisation state within a grain when the energy barrier to reverse its state is $\leq 25k_B T$, assuming that all spins within a grain behave coherently [Fulcomer and Charap (1972a, b), Nishioka et al. (1996)]. Any of these processes result in the change of the original state related to a local energy minimum, to another energy magnetisation state in another local minimum, which are separated by an energy barrier, $\Delta E$. 

Figure 29. Magneto-optic indicator film technique (MOIF) images of FeMn/Fe$_{76}$Mn$_{6}$C$_{18}$ bilayer during application and reduction of a field $\approx 12^\circ$ counter clockwise from the easy magnetisation direction in both directions. Black arrows indicate magnetisation direction in the domains (Shull et al., 2003).
2.11 The Seven-Point Model

This so-called model is a description of the distinct features of a hysteresis loop of an exchange biased system. The model, developed by Goodman et al. (2001), describes the different stages of the magnetisation processes that occur while the $F$ magnetisation is cycled in seven points depicted in figure 30.

The features described are as follows:

1. At point 1 in the hysteresis loop the $F$ is saturated in the positive field direction.
2. At point 2 the reversal of the $F$ is initiated. Nucleation of reverse domains in the $F$ appears due to the degree of saturation at point 1.
3. At point 3 the domains nucleated at point 2 grow. Their growth is promoted by the applied field, the exchange coupling inside the layer and the thermal activation. At this point the coercivity exhibits normal magnetic viscosity. A fast sweep rate from point 1 to point 3 leads to an increase in the value of the coercive point $H_{cl}$.
4. At point 4 the $F$ is saturated in the negative field direction. However, thermal activation of $AF$ reversal continues. Goodman et al., (2001) proposed that at this stage there is growth of domains within the $AF$ dominated by the exchange field exerted by the $F$ on the $AF$.

Figure 30 Schematic diagram of the magnetisation reversal of an exchange biased system.
5. At point 5 it would be expected that the $F$ would reverse back to positive magnetisation, as would be the case of a single layer $F$ material. This does not occur due to additional energy barriers arising from the pinning of domain walls by the $AF$. Hence an additional anisotropy appears in the $F$ via the exchange interaction between the $F$ and $AF$.

6. At point 6 the nature of reversal is different to the nucleation of domains that occurs in point 2. Domains develop via domain wall movement over pinning sites or via domain rotation. At this point, pinning must be significant to account for the viscosity effects at point 4. The $AF$ domain processes can only be observed via their effect on the $F$.

7. At point 7, Goodman et al. (2001) observed what they called anomalous behaviour of the second coercivity, $H_{c2}$. They believed that $H_{c2}$ is indeterminate and depends critically on the amount of time spent with the $F$ layer saturated, and therefore on the exchange field between $F$ and $AF$ layer on the growth of domains in the $AF$ layer.

Although the $AF$ layer is not affected by the applied field, the exchange field from the $F$ acting on the $AF$ influences the $AF$ domains (Goodman et al., 2001), which added to thermal activation processes gives rise to time dependence of the values of $H_{c2}$ and hence $H_{ex}$. This model provides a point description of the hysteresis loop that will be used along this thesis to refer at the different stages during the hysteresis loop.
Chapter 3

Exchange Biased Systems

3.1 The Nature of Exchange bias

An exchange bias system consists of an $AF$ material, in intimate contact with a $F$ material. These systems, when cooled down from $T_N < T < T_C$ to a temperature less than the $T_N$ of the $AF$ in the presence of a strong magnetic field, show some characteristic features.

In such structures, the ferromagnet acquires uni-directional anisotropy. This phenomenon is called exchange bias, because the hysteresis loop associated with the $F$-$AF$ structure can be centred about a non-zero magnetic field. It was discovered by Meiklejohn and Bean (1956), while they were studying small Co particles, which are ferromagnetic. These particles were covered with an outer layer of CoO, which is $AF$. The hysteresis loop of this system was not symmetrical about the origin but shifted to negative field when cooled in a positive applied field. The phenomenon of exchange bias has been studied for the last forty years due to its complexity, which arises from the competing interactions for magnetic order between the ferromagnetic and $AF$ layers.

The mechanism of exchange bias can be explained as follows:

1. At temperatures above $T_N$ and below $T_C$, the Curie temperature of the $F$ layer has all its spins magnetically ordered, meanwhile, the $AF$ is in a paramagnetic state. When a magnetic field is applied, all the $F$ spins are aligned parallel to the field (Fig. 31).

![Figure 31 Schematic of the spin structure of an exchange coupled bilayer. At zero applied field, and above the Néel temperature, the ferromagnetic spins stay ordered, while the $AF$ stays in its paramagnetic state.](image-url)
2. When the sample is cooled to below \( T_N \), in a field, the spins of the \( AF \) order. The spins in the closer layers to the interface align parallel to the ferromagnet's magnetisation, \( M_F \), due to the exchange interaction. This corresponds to point 1 in the seven-point hysteresis loop. See figures 32 and 33(a).

3. When the field applied begins to reverse, \( M_F \) tends to reverse. The spins of the \( AF \) layer do not change their orientation, as they are insensitive to the applied field. The \( AF \) spins exert an exchange field, on the \( F \) spins, which is in opposition to their reversal. This implies an increase in the field necessary to reverse the \( F \) spins. At this stage, the system is at point 2 as shown in figure 32 and 33(b). Eventually reversal is initiated by the nucleation of reversed domains.

4. Point 4 of the hysteresis loop in figure 32: At this stage the ferromagnetic layer is saturated in the reverse direction. The \( F \) spins and the \( F-AF \) exchange field are now antiparallel to the interfacial \( AF \) spins. See figure 33(c).

5. Point 5 of the hysteresis loop in figure 32: At this point the field applied starts to reverse towards positive values.

6. Point 6 of the hysteresis loop in figure 32: The exchange field exerted by the \( AF \) layer on the ferromagnet promotes the rotation on the ferromagnetic spins; hence, a weaker magnetic field is required to reverse their orientation. See figure 33(d).

Figure 32 Seven-point hysteresis loop model. The exchange field is given by the shift of the loop. The values \( H_{c1} \) and \( H_{c2} \) are used to obtain the coercivity, which is defined as the half width of the loop.
The unidirectional anisotropy acquired by the ferromagnet results in a hysteresis loop shifted by an amount $H_x$. This is known as the exchange field, which is applied by the $AF$ layer on the ferromagnet. It also exhibits a coercivity enhancement. This means that the values of the coercive fields change, resulting in a wider hysteresis loop as a consequence of the exchange anisotropy. Other effects associated with exchange bias is the uncharacteristic form of the $F$ magnetisation dependence on time, known as magnetic viscosity (Fulcomer and Charap, 1972a, 1972b), (see section 2.9), and the memory effect, which is related to the fact that the exchange biased system keeps a memory of the undergone field cooling process (Ambrose et al., 1997).

![Mechanism of a shifted loop](image)

**Figure 33** Mechanism of a shifted loop. Schemes of the spin structure in an exchange coupled bilayer at different stages of the hysteresis loop. (a) Point 1 in the seven-point model, (b), (c) and (d) correspond, respectively, to points 2, 3, and 6 in the seven-point model.

### 3.2 Historical Perspective

In an exchange biased system the competing interactions at the interface between the $F$ and the $AF$ spins lead to complex phenomena, which are the reason several models are proposed. Interest in the exchange bias phenomenon was merely academic until the late-eighties, when the Giant Magneto-Resistive effect (GMR) was discovered (Baibich, et al., 1988). The technological potentiality of this effect renewed the interests in the
exchange bias effect and new models appeared. The development of the techniques available to study the magnetic structure, magnetic properties, and the improvement of computing capabilities have lead to the development of the theories of exchange bias.

Meiklejohn & Bean (1957) and Jacobs & Bean (1963) showed a simple description of the mechanisms that would produce a shifted hysteresis loop. Néel’s (1967) studies highlighted the different processes that are involved in exchange bias. The models can be grouped into three categories. The first is that the reorientation of the ferromagnet through an applied magnetic field can involve the formation of magnetic domain wall structures in the AF. See Mauri et al. (1987), Koon (1997), Schulthess and Butler (1998), Stiles and McMichael (1999a, 1999b, 2001).

The second category was defined with considerations of domain formation and domain wall motion, pinning and depinning during reversal of the ferromagnet (Meiklejohn (1962), Schlenker and Paccard (1967), Schlenker (1968) and Néel (1967). Since this time, theories have been put forward to describe domain wall dynamics and wall dynamics in the ferromagnet (Malozemoff (1987), Nemoto et al., (1999), Kiwi et al., (1999a, 1999b), Nikitenko et al., (2000)), and also domain dynamics in the F (Li et al., (2000), Leighton et al., (2000)).

Considerations of time dependence and irreversible processes are also relevant, since exchange bias is intimately connected to the magnetisation process during reversal, (Schlenker (1968), Fulcomer and Charap (1972a,b), Schlenker et al., (1986), Nishioka et al.,(1996)). This has lead to more recent examinations of thermal effects (van der Heijden et al., (1998a, 1998b), Gökemeijer et al., (1999a, 1999b). Magnetic viscosity experiments and field rate studies have also been reported (Goodman et al., 1999, 2000). The importance of thermal processes on exchange bias and coercivity show that the magnetisation history of the sample is important (Miltényi et al., (1999), Stiles and McMichael (1999a, 1999b), Nogués et al.,(2000a)), and can result in a variety of behaviours including positive exchange bias (Nogués et al., 1996, 2000b) and domain wall processes in the AF (Nowak and Usadel (2002), Kim and Stamps (2005)).

The third category comprises the granular models. These models treat both F and AF as an assembly of grains, each of them is considered to be in a single domain state. The
first of them was published by Fulcomer and Charap (1972) who considered the effect of temperature fluctuation on non-interacting single domain AF grains exchange coupled to the ferromagnetic moment of the underlying film. They proposed an explanation of the temperature dependence of the coupling between F and AF given by the thermal instability of the grains. Nishioka et al. (1996) extended Fulcomer and Charap's model to explain the coupling dependence on the AF grain size. Stiles and McMichael (1999a, 1999b), developed a grain model that differs from Fulcomer and Charap in the fact that the AF grains have random orientations and contain domain walls. The AF grains are also independent, however they are coupled to the F not only by direct coupling but also by spin-flop coupling.

Even though there are several models proposed. None of them have managed to give a complete explanation for exchange bias. In fact some of the models predict correct values for certain materials, but not all, or do not contain temperature dependence. All models proposed until now are designed for uniaxial materials and do not contain considerations for biaxial materials such as IrMn studied in this work.

The work done in this thesis is focused on measurement reproducibility. Hence, control of the sample history is taken into consideration. The protocols developed are based on this idea and the interpretation of exchange bias broadly follows Fulcomer and Charap's (1972b) work. Temperature and time dependencies are studied under a grain model description. The AF magnetic behaviour is considered in terms of energy barrier distributions to reversal as discussed in section 2.9.

3.3 Exchange Biased Systems

The first systems where exchange bias was found were systems of fine particles. The materials studied were mainly ferromagnetic particles covered with their native AF oxide. Other materials have been used, such as nitrides or sulphides (Berkowitz and Takano, 1999). The size of these particles is normally in the nanometre range. They are produced by different methods, such as vapour deposition, electrodeposition, and reduction of the oxalate or mechanical alloying. These systems are non-ideal for studies of fundamental aspects of exchange bias, since there is always a distribution of particle...
sizes and shapes, which makes it difficult to identify the nature of the interface, stoichiometry, crystallinity, etc. of the AF layers.

There are some systems that don't have a clearly defined AF-F interface or that have multiple AF-F interfaces, where due to the arrangements of the magnetic ions, different areas or domains with AF or F interactions are created. In this category are spin glasses and some ferrimagnets. Other systems studied are coated AF single crystals (AF single crystal polished along a specific crystallographic direction, coated with a F material) (Nogués and Schuller, 1999).

The most widely studied materials have been in the form of thin films. In these systems the interface can be effectively controlled and characterised. These types of materials have been used to study different phenomena related to exchange bias, such as AF thickness, interface disorder, or orientation dependence of $H_{ex}$ (Nogués and Schuller, 1999).

The ferro-AF group can be divided in three main categories depending on the AF used: oxide, metallic, others.

- **Oxide antiferromagnets**

After works on oxidized F particles, most of the early work on exchange bias was on oxidized transition metal films, Co-CoO, Ni-NiO, Fe-FeO, or oxidized Fe$_{20}$Ni$_{80}$. To avoid some of the problems that these systems may present, antiferromagnet oxides have been sputtered directly from oxides from the metal or in a reactive oxygen atmosphere.

- **Metallic antiferromagnets**

The first fully metallic thin film was reported in 1964. Fe$_{20}$Ni$_{80}$/Mn bilayers were annealed to enhance diffusion, creating $AF$ Fe$_x$Ni$_y$Mn$_{1-x-y}$ compounds at the interface. One of the most studied AF systems was Fe$_{90}$Mn$_{10}$, which was the basis for most exchanged biased GMR spin valves (Baibich et al., (1988), Dieny et al., (1991)) until recently. Materials currently being used are PtMn and IrMn due to their higher thermal stability and higher anisotropy. The development of spin valves lead to research for new $AF$, with high $T_N$ and resistance to corrosion.
• **Other antiferromagnets**

Non-metallic or non-oxide systems exhibiting exchange bias include sulphides, fluorides and nitrides. The details of these structures are not very clear. Recently, FeF$_2$ has been extensively studied (Nogués et al., 1996). Since its structure is simple, it is suitable for the study of basic properties of exchange bias.

• **Ferrimagnets**

These systems are similar to AFs. They also have two sublattices with opposite magnetisation, but ferrimagnets have the peculiarity that the net magnetic moment is non-zero, as one of the sublattice magnetisations is larger than the other. Their magnetic structures can play the role of the AF or the ferromagnet in bilayer systems. These systems are difficult to analyse theoretically, due to the complexity of the two layers in the ferrimagnet (Nogués and Schuller, 1999).

### 3.4 Technological Importance of Exchange Bias

The application of exchange bias is generally related to the magnetic recording industry (Berkowitz and Kodama, 2005). Since the discovery of Giant Magnetoresistance in 1988 (Baibich et al., 1988) and its early implementation in spin-valves (Dieny et al., 1991), exchange bias is being used in all modern computers. The read-write head uses the exchange bias in the magnetic thin film sensors that read the bits of information from the hard drive. Figure 3.4.1 shows a schematic of a typical read-write head used in a computer disk drive. The recording medium is a magnetic thin film typically about 10nm thick, with high coercivity and remanence, which is on a flat disk that rotates at about 7200 to 15000 rpm. On top of the magnetic layer there is a 5 to 10nm thick carbon coating and a 1nm thick lubricant layer. The read-write head is suspended on a lever arm 10nm above the lubricant layer. The lever arm controls its radial position over the disk. The write element uses pulsed currents to write the bits by magnetising small regions in the recording layer. There are stray fields arising from consecutive bits magnetised in opposite regions, which added to the random grain size that form the magnetic layer create a noise region between bits.
As seen in figure 34 the read sensor is shielded from exposure to surrounding bits. The arrow depicted in the read sensor indicates the magnetisation direction of the sensing film. Its direction rotates depending on the magnitude and direction of the stray flux from the read bit. The thin film sensor consists of a multilayer structure called a spin valve. A schematic of the cross section of spin valve is shown in figure 36. The spin valve uses the GMR phenomenon, i.e., the change in the magnetisation direction of the sensor layer in the spin valve due to the stray field from the read bit produces a change in the resistance of the spin valve, which is monitored by a bias current that passes through it. The output signal is this change in the resistance. The GMR phenomenon was discovered in thin film multilayers of alternating ferromagnetic layers separated by non-magnetic spacers. The $F$ layers are exchanged coupled through the non-magnetic layer. The sign of the exchange coupling oscillates with increasing thickness of the non-magnetic layer (Parkin, et al., 1990). Figure 35 shows the case of a multilayer with antiferromagnetic coupling between the $F$ layers, given the right thickness for the non-magnetic layers. The resistance for this structure is a maximum. If a saturating field is applied, the ferromagnetic alignment of the $F$ layers leads to a minimum in the resistance across the multilayer.
The spin-valves used in the read sensor are formed by an $F$-$AF$ exchange coupled bilayer, a non-magnetic conducting spacer and a $F$ layer as shown in figure 36. The non-magnetic layer thickness is chosen to minimise the exchange coupling between the free and the pinned $F$ layers. The easy axis in the free $F$ layer is determined by its intrinsic anisotropy field direction and the residual exchange coupling to the pinned $F$ layer. The magnetisation of the free layer rotates under the influence of the stray field from the bits in the recording film. This produces a change in the resistance in the multilayer, sensed by a bias current in the plane of the field. The pinned $F$ layer and the easy axis direction of the $F$ layer are crossed to produce a linear response in the resistance output.
It is of technological importance to make the sensor as thin as possible. Typical thicknesses are <8nm for the AF and the F layers and 2nm for the spacer, which is normally Cu. The exchange field must be larger than the stray flux from the bits, which is normally about 100 Oe. The bias current is minimised to reduce the possibility of heating the sensor to temperatures close to the blocking temperature of the pinned AF. Materials frequently used were NiO, which has been substituted for Mn-based alloys such as IrMn and PtMn due to their higher $T_B$.

Structures like the one shown in figure 36 are improved by replacing the free F single layer for a trilayer of two antiferromagnetically exchange coupled Fs with a Ru or Cu interlayer. The purpose of this is to eliminate the curling of magnetisation on the edges of the free F layer close to the recording medium.

Spin polarised magnetic tunnel junctions have a basic structure similar to figure 35. The simplest structure is formed by two F layers separated by an insulator of thickness in the order of 1nm. The principle of operation is based on the production of electron transport across the trilayer by the application of a bias voltage to the F layers. The conductance between the F layers depends on the relative orientation of the magnetisation of both layers. The development of magnetic-random-access-memory (MRAM) based in magnetic-tunnel-junction structures has been of industrial interest since high tunnel magnetoresistance can be achieved with these structures, e.g. Moodera et al., (1995 and 1999). Figure 37 shows a schematic of a magnetic tunnel junction cell used for MRAM. It contains a F layer exchange biased to AF layer and separated by an insulating interlayer from the free F layer. A high resistance is achieved when the free F layer magnetisation is antiparallel to that of the pinned F layer. Resistance ratios larger than 100% have been reported by Parkin et al., (2004) and Yuasa et al., (2004) and recently 350% TMR by Parkin, 2005, as reported by S. Mao (York, 2005).
Figure 37 Schematic of an spin tunnel junction.

Figure 38 Schematic of cross-point architecture matrix of magnetic tunnel for MRAM junctions. Courtesy of Paulo Freitas

Other applications of the exchange-bias phenomenon are based as well in the use of AFs and antiparallel-pinned Fs. The use of a magnetic tunnel junction transistor using IrMn to bias the emitter has been reported by Parkin et al. (2003). Field sensors sensitive to picotesla fields are based on magnetic tunnel junctions (Tondra et al., 1998).
Chapter 4

Theoretical Models of Exchange Bias

4.1 Introduction

After fifty years of study there is no universal theoretical basis that explains the exchange bias phenomenon because the effects of exchange coupling are of short range. Hence a detailed model of the interface structure is needed to explain the behaviour of each exchange coupled system. In the majority of the cases this information is not available. Furthermore, differences in samples of the same materials produced in different growth systems lead to properties that have different values due to different growth conditions, such as different grain size distributions, crystallinity, interface quality and other defects such as impurities or oxygen inclusions. The spin structure of the $AF$, which governs the anisotropy of the system, is generally not known because the easy axis direction of the $AF$ grains is also unknown, as well as the spin structure at the interface, and if it is compensated, i.e., the same number of spins of both sublattices are present, or whether it is uncompensated or partially uncompensated.

This thesis relates to the effects of thermal activation on exchange biased systems. Whether the thermal activation effects on the reversal of the $AF$ material is due to the reversal of the magnetic state of the $AF$ grains or due to domain processes is not known. Due to all these matters, there is no unified theory that is able to explain the exchange bias phenomena for all different materials.

This chapter contains a selection of theoretical models which have made a significant contribution to the understanding of exchange bias phenomenon and which at the same time are of relevance to the work done in this thesis.
4.2 Early models

4.2.1 Meiklejohn and Bean's model

The model of exchange bias proposed by Meiklejohn and Bean suggests that the shift in the hysteresis loop appears due to a large anisotropy in the AF and a weaker exchange energy coupling the AF to the F. A shifted loop for an exchange couple is shown in figure 39.

![Figure 39 Schematic hysteresis loop for an exchange biased system.](image)

The mechanisms of exchange bias suggested by Meiklejohn and Bean (1956) were described in chapter 3.1 where a perfectly uncompensated AF interface spin structure is assumed. A saturating magnetic field is applied at a temperature above $T_N$, figure 40(a). After cooling the system in a field, the magnetisation of the ferromagnet remains pinned along the original direction for small negative fields. See figure 40(b). The magnetisation of the F is reversed when the field applied is large enough to overcome the interlayer exchange, as shown in figure 40(c). The effective field associated with the interlayer exchange gives the magnitude of the shift.

![Figure 40 Spin structures at different stages of the reversal of the ferromagnet, (a) before field-cooling, (b) and (c) after field-cooling. The F spins reverse when the applied field is large enough (c).](image)
Since they were working on oxidised cobalt nanoparticles, Meiklejohn and Bean considered single domain spherical particles with uniaxial anisotropy with their easy axis aligned parallel to the field, $H$, applied in the opposite direction to their saturation magnetisation, $M_S$. Examining the stability of the energy per unit area for this model (Stamps, 2000), we can find the values of the coercive fields, $H_{c1}$ and $H_{c2}$ (see fig. 39).

$$E = -HM_S t_F \cos \theta - J \cos \theta + K_F t_F \sin^2 \theta$$  \hspace{1cm} (4.1)

In this model, $t_F$ is the thickness of the ferromagnet, $J$ is the interlayer exchange between the ferromagnet and the AF, $K_F$ is the uniaxial anisotropy constant of the ferromagnet, and $\theta$ is the angle between $M_S$ and the uniaxial anisotropy easy axis. The field is aligned along the easy axis, and the magnetization is assumed uniform. Another important assumption is that the AF remains rigidly aligned along the direction of its easy axis, which is assumed to lie parallel to the ferromagnet easy axis. This restriction is the reason why this model is known as the rigid antiferromagnet model. The energy has extrema corresponding to saturation for values $\theta = 0$ and $\theta = \pi$. Stability arguments for these configurations give the values of the coercive fields, $H_{c1}$, $H_{c2}$, and the coercivity $H_C$,

$$H_{c1} = -\frac{2K_F t_F + J}{M_S t_F}$$  \hspace{1cm} (4.2)

$$H_{c2} = \frac{2K_F t_F - J}{M_S t_F}$$  \hspace{1cm} (4.3)

$$H_C = \frac{2K_F}{M_S}$$  \hspace{1cm} (4.4)

Since the coercive fields are different in magnitude, the hysteresis loop is biased. The bias field in this model is defined as the midpoint of the hysteresis loop, and is directly proportional to the exchange coupling:

$$H_{ex} = \frac{J}{M t_F}$$  \hspace{1cm} (4.5)

The bias can also be interpreted as an exchange coupled anisotropy field acting on the ferromagnet. The magnitude of the exchange field depends directly in the value of the exchange constant between the $F$ and the $AF$. This result leads to values of $H_{ex}$ which...
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are two orders of magnitude larger than the experimental values observed (Meiklejohn, 1962) and predicts a thickness dependence of \( H_{ex} \) proportional to \( 1/t_F \).

4.2.2 Néel's model. The continuum approximation.

Néel's exchange bias model (Néel, 1967) assumed an uncompensated \( AF \) spin interface coupled to the \( F \) layer, uniform magnetisation of each sublayer, being parallel to the interface and uniaxial anisotropy. Néel wrote a discrete hamiltonian for the magnetisation state of each of the sublattices. The main result of this continuum approximation is the formation of domains both in the \( F \) and the \( AF \) although a minimum thickness of both layers is required to allow for domain formation.

This model again failed to predict reasonable values for \( H_{ex} \), which were several orders of magnitude larger than those observed experimentally (Meiklejohn, 1962). It was also expected that compensated interfaces would not give rise to \( H_{ex} \) and that the increased roughness at the interface would increase \( H_{ex} \).

4.3 Domain walls in the \( AF \)

Domains in exchange biased bilayers has been observed by several authors. The study of domains on wedged-NiFe and uniform FeMn samples by Nikitenko et al. (2000), using a magneto-optical technique, showed domain wall motion that was asymmetric in the forward and reverse branch of the hysteresis loop. This asymmetry, incompatible with a static \( AF \) spin structure, probed the existence of a moving domain wall within the \( AF \), forming an exchange spring in the \( F \).

4.3.1 Domain walls parallel to the interface

Mauri et al. (1987) proposed a model which resulted in a lower interfacial energy than that of Meiklejohn and Bean, and gave more reasonable values for \( H_{ex} \) compared to the experimental results. They assumed that the magnetisation of the \( F \) and the order in the
AF sublattices are parallel to the interface and coupled to each other across a perfectly plane interface. They also assumed a F thickness smaller than the domain wall width and the formation of a domain wall inside the AF parallel to the interface. The energy required to form such a (90°) domain wall is $2\sqrt{A_{AF}K_{AF}}$, where $A_{AF}$ is the exchange stiffness and $K_{AF}$ the crystalline anisotropy in the AF. The magnetic energy per unit area of an interface like this is given by

$$E = 2\sqrt{A_{AF}K_{AF}} (1 - \cos \alpha) + A_{F-AF} / \xi [1 - \cos(\alpha - \beta)] + K_{F}t_{F} \cos^{2} \beta + HM_{S}t_{F} \left(1 - \cos \beta\right)$$

(4.6)

where $\alpha$ is the polar angle formed by the spins in the closest AF sublattice layer to the F with the AF uniaxial anisotropy axis (z axis), and $\beta$ is the angle formed by the F spins with the z axis. $A_{F-AF}$ is the exchange stiffness at the interface, $K_{F}$ is the ferromagnet anisotropy constant, $M_{S}$ is the saturation magnetisation of the F, $t_{F}$ is the thickness of the F, and $\xi$ the interface thickness. Minimising eq. 4.6 with respect to $\alpha$ and $\beta$ for a given value of the applied field $H$ gives the value of the $H_{ex}$. In the case of the interface energy being much less than the domain wall energy ($\alpha$ small)

$$H_{ex} = -\frac{A_{F-AF}}{\xi M_{S}t_{F}}$$

(4.7)

The substitution of $J = A_{F-AF} / \xi$, gives the same expression as eq. 4.5 obtained by Meiklejohn and Bean. In the case of the interface energy being much larger than the domain wall energy, the exchange field is

$$H_{ex} = -2\sqrt{A_{AF}K_{AF}} / M_{S}t_{F}$$

(4.8)

Magnetisation curves are constructed assuming coherent rotation showing loop asymmetry for the case of the interface energy being of the order of the domain wall energy. In the case of the interface energy being larger, 180° domain walls appear in the AF. This model does not explain the enhanced coercivity of the F when it is exchange
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coupled to the AF, or the decrease of $H_{ex}$ and $H_C$ when the field is cycled repeatedly. The pinning of the domain walls due to imperfections appears as an explanation of the effects of the AF on the coercivity in the same way as in the Néel (1967) model.

4.3.2 Random field model. Domain walls perpendicular to the interface.

Malozemoff (1987) introduced a random interface roughness between the AF and the F. This gives rise to compensated and uncompensated areas at the interface. In consequence, there is a random field that acts on the interfacial spins. Due to anisotropy energy minimisation the AF breaks up into domains, with domain walls perpendicular to the interface. Assuming that the F is in a single domain state, uniaxial anisotropy in the AF, and a fixed size of the AF domains given by

$$L \approx \pi \sqrt{A_{AF} / K_{AF}}$$  (4.9)

the exchange field predicted is

$$H_{ex} = \frac{2z \sqrt{A_{AF} K_{AF}}}{\pi^2 M_S t_F}$$  (4.10)

The number $z$ is of the order of unity (Imry and Ma, 1975). This parameter arises from interface irregularities, whose local energy magnitude is taken to be on average $2zJ$, where $J$ is the exchange constant, assumed to be constant on both sides of the interface. This model assumes that $A_{AF}$ and $K_{AF}$ are temperature independent to a first approximation. Equation 4.10 is similar to eq. 4.8 resulting in values of $H_{ex}$ of the same order of magnitude. Malozemoff suggests an explanation for the reduction in $H_C$ when the F magnetisation is repeatedly cycled. Movement of the AF walls would generate the reduction of $H_C$. The results of this model depend crucially on the concentration of defects.

Malozemoff (1988a, 1988b) also considered the case of AF materials with cubic anisotropy, such as FeMn, and IrMn. The latter is studied in this thesis. The result of
random interfacial fields in these materials would be to stabilise the domains which would have a different topology to those formed in uniaxial materials. In the case of the uniaxial materials an AF domain width independent of temperature would be expected for uniaxial materials while in the case of cubic materials it would be expected to vary as \((1 - T / T_N)^{1/2}\). Hence, domains formed at high temperatures would be larger than those formed at low temperatures.

The predicted variation of \(H_{ex}\) with temperature is \((1 - T / T_N)^{1/2}\) for uniaxial systems and \((1 - T / T_N)\) for cubic systems. This is discussed in chapter 6.3, where measurements on IrMn/CoFe bilayers show that it is only true for certain temperature regions. Furthermore, the model of Malozemoff (1987,1988) is proposed for a single crystal AF, whereas in this work polycrystalline AF/F bilayers have been studied. Malozemoff (1988b) also predicted a critical thickness for the AF \(t_{AF}\) below which there is no \(H_{ex}\). Above this critical thickness \(H_{ex}\) decreases with increasing \(t_{AF}\) up to a second critical thickness at which \(H_{ex}\) achieves a minimum value.

All the models described above and many others consider the exchange stiffness, \(A_{AF}\), \(A_{F-AF}\), and the anisotropies, \(K_F\), \(K_{AF}\) to be first approximations to the models. In fact, these parameters are considered to be uniform in the \(F\) and \(AF\) layers, and temperature independent. Ultimately they are used as fitting parameters to predict the values of \(H_{ex}\). In section 6.2, it is shown that the degree of order in the \(AF\) is temperature dependent and controls the \(H_{ex}\).

4.3.3 Random interface and orthogonal magnetic arrangements.

The experimental observation of perpendicular spin arrangement at the \(AF-F\) interface was made by Jungblut et al.(1994, 1995) and Nogués et al.(1996a) showing that compensated and uncompensated interfaces lead to similar results for \(H_{ex}\). Koon (1997) gave a microscopic explanation for exchange bias in which the spins in the \(AF\) close to the interface align perpendicular to the direction of the \(F\) spins close to the interface due to magnetic frustration of those spins when the interface is compensated. This type of configuration is known as spin-flop coupling. When the \(F\) is reversed exchange bias would appear as result of the formation of a domain wall parallel to the interface.
Schulthess and Butler (1998) showed that spin-flop coupling does not lead to the formation of a parallel domain wall in the AF and therefore would not lead to exchange bias, although it does lead to an increase in the value of $H_C$ by two possible mechanisms (Schulthess and Butler, 1999). One is the reversal of some small AF domains during the reversal of the F magnetisation, leading to irreversible effects in the AF. This mechanism leads to a coercivity that is dependent on the magnetic history of the sample. The other mechanism is the reduction of domain wall widths when the F is coupled to the AF increasing $H_C$ due to pinning of domain walls. In this case $H_C$ would depend on the morphology and the spin-flop coupling rather than the magnetic history.

The mechanisms proposed for the loop shift are random fields at the interface originating at defects and interface roughness on the same basis as the Malozemoff theory. These would give rise to the formation of domains in the AF with walls perpendicular to the interface. The increase of $H_{ex}$ with increasing interface roughness (Morán et al., 1995) is explained when the random field is due solely to interface roughness. Interface roughness can also decrease $H_{ex}$ (Nogués et al., 1996b). This would happen when the random field is induced by uncompensated regions through lattice strain. These results are also explained in terms of the domain state model, which is described in section 4.5. Assuming that the interface is of a fractal nature and that the density of defects that can pin a domain wall increases with $d_w^{-1}$, where $d_w$ is the domain wall width, the coercivity is then expected to vary as (Schulthess and Butler, 1999)

$$H_C \propto 1/t_F^{3/2}$$

(4.11)

where the parameter $t_F$ is the thickness of the F layer, in contrast with the $H_{ex}$ variation with $1/t_F$, as generally observed. A similar expression was derived by Zhang et al., (1999) who assumed random interactions at the interface and the formation of a domain wall in the F. Keller et al., (2002) based on the domain state model (Nowak et al., 2002), briefly described in section 4.5, performed measurements that showed how roughness is not the main parameter that influences $H_{ex}$. Contrary, measurements on Co/CoO showed that $H_{ex}$ was controlled by the concentration of defects within the AF layer by keeping the interfacial roughness constant and varying the defect concentration.
4.3.4 Domain wall processes in the antiferromagnet

Several authors showed that a perfectly rigid AF can not predict the observed values of exchange bias. The existence of a twisted magnetic structure within the AF by distributing the twist across a number of spins leads to more reasonable values for $H_{ex}$ (Néel, 1967, Mauri et al., 1987, Koon 1997, Kiwi et al., 1999b).

Stamps (2000) addressed two different types of exchange bias depending on whether the processes involved are reversible formation of partial domain walls or whether irreversible processes give rise to asymmetric hysteresis loops. However, asymmetric hysteresis can be caused without complete loss of partial wall bias when mixed interfaces are present resulting in shifted asymmetric loops. The energy barriers to partial domain wall formation are considered to be of two types, those involved with in-plane rotation of the magnetisation of the F and those related to out-of-plane rotation. Consideration of these energy barriers showed that $H_C$ can appear in exchange biased systems without the need of anisotropy in the F (Stamps, 2000) in contrast with the rigid AF models.

Thermally averaged behaviour of exchange bias was studied in terms of mean field theory (Kim et al., 1999, McGrath and Camley, 2000, and Stamps and Wee, 2000). This was applied to both compensated and uncompensated interfaces. As a result a decrease of the $H_C$ with increasing temperature is predicted. This result is at odds with data presented in this thesis of thermal activation in IrMn/CoFe systems (See chapter 6.2). Thermal behaviour studies (Stamps, 2000) described by a fluctuation mechanism (Fulcomer and Charap, 1972b, van der Heijden et al., 1998a) showed a initial $H_C$ increase with increasing temperature, which is more in accordance with the results presented in chapter 6.2. Also, $H_C$ increased with decreasing sweep-rate as result of the increase of the time for thermal activation effects in the AF, leading to irreversible changes in the AF state (Goodman et al., 2000). Stamps and Wee (2000) considered the temperature dependence of $H_{ex}$ and $H_C$ to have similar origins, namely wall pinning and de-pinning mechanisms, although the resulting temperature dependencies were different.
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The theoretical study of the effects of inclusion of magnetic impurities within the AF can account for some of the features observed in exchange biased systems (Kim and Stamps, 2005a). Such impurities modify the domain walls in the AF giving rise under certain conditions to $H_C$ enhancement and asymmetric hysteresis loops. These are a reduction of the AF wall energy due to the presence of defects which induces no irreversible behaviour and which is dominant at low defect densities. The other is related to the pinning of the AF wall formed during reversal in a metastable state. This effect is more important at moderate and high defect densities and results in irreversible behaviour, leading to $H_C$ enhancement. At finite temperatures similar domain wall processes are predicted using a local mean-field theory (Kim and Stamps, 2005b). In this case, pinning is due to displacement of the domain wall from the interface due to thermal fluctuations and similarly depinning of metastable states is predicted to occur from thermal effects at temperatures close to the ordering temperature of the AF giving rise to a reduction of the blocking temperature.

4.4 Granular Models

Fulcomer and Charap (1972a) studied the exchange bias phenomenon on NiFe films. They partially oxidised the NiFe samples ($F$), getting part of the surface covered with NiFe oxide (AF). They investigated the $H_C$ and $H_{ex}$ of these oxidised NiFe layers over a range of sweep-rates, varying the frequency at which they measured the hysteresis loops. Films that were free of any surface oxide did not show a displacement of the easy-axis loop along the field axis. $H_C$ and $H_{ex}$ were found to be very weak functions of temperature. Oxidised films showed striking exchange anisotropy effects. Displaced loops appeared below a transition temperature ranging from 30 to 90K as the film was progressively oxidised.

They observed that $H_{ex}$ decreases with increasing temperature and goes to zero at a temperature that is dependent upon the amount of oxidation. $H_C$ was observed to increase at higher frequencies. $H_C$ shows a peak around the temperature at which the $H_{ex}$ goes to zero. This result is of great controversy nowadays. Some authors state that this peak occurs in the proximity of this temperature, called the blocking temperature ($T_B$). Another remarkable result was that while the loop shift increases with increasing
frequency, the coercivity decreases with increasing frequency (Fulcomer and Charap, 1972, a) in contrast to normal $F$, whose coercivity increases with increasing frequency (de Witte et al., 1993).

These phenomena were attributed to exchange anisotropy arising from small nickel oxide particles forming on the surface that increase in quantity and size as oxidation proceeds. They suggested that there is only one possible mechanism responsible for the temperature and frequency dependence of coercivity and the anisotropy field, as well as for the appearance of a displaced loop well below the Néel point of the oxides. Fulcomer and Charap (1972b) proposed a thermal fluctuation after effect model that was based in the theory of magnetic aftereffect for very small particles (Neel, 1949) to explain the features described previously. The $AF$ layer is regarded as an assembly of uniaxial particles with a distribution of particle sizes with no net moment. These $AF$ particles or grains remain fixed during the reversal of the $F$ layer. They considered the effect of thermal activation of those particles on the energy of such system (Fulcomer and Charap, 1972b).

According to their theoretical model, the magnetisation of the film biases, and is biased by the particles through an interfacial exchange coupling. Above a blocking temperature ($T_B$), dependent on the size of the oxide particles, these are able to reverse rapidly due to thermal fluctuations, exhibiting as a consequence of this, quasi-paramagnetic behaviour. As described in §2, the exchange field vanishes at the blocking temperature $T_B$. This temperature is the blocking temperature for the largest of the $AF$ particles. Studies carried out in this work following a careful and methodologically detailed procedure revealed that this peak does not correlate with the median $T_B$. This is described in chapter 6.2.

Assuming a physically reasonable distribution of particle sizes, the model by Fulcomer and Charap was in good agreement with their results. Even though it is probably incomplete in certain aspects. For example, they suggested a non-interacting coherent rotation mechanism. However, it is very likely that the $AF$ grains interact and it is possible that the reversal involves the formation of domain walls (Mauri, 1987, Malozemoff, 1988, van der Heijden, 1998a, 1998b). Stiles and McMichael (1999a,
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1999b, 2001) proposed a similar and more general granular model which contains fewer restrictions in their assumptions.

Stiles and McMichael (1999a) considered polycrystalline \( F-AF \) bilayers where the \( F \) is supposed to be in a uniform saturated magnetic state with no or very few domain walls. The \( AF \) is assumed to have uniaxial anisotropy, and to be formed by independent \( AF \) grains; each with randomly distributed easy axes. The coupling between \( F \) and \( AF \) is via direct and spin-flop coupling. The interface is considered to be rough. Hence, the coupling is frustrated to a large extent due to compensation of the \( AF \) sublattices present at the interface. Therefore, each \( AF \) grain will have a small net moment. The formation of domain walls parallel to the interface in the \( AF \) is allowed. Stiles and McMichael introduced the concept of a critical angle, \( \alpha_{\text{crit}} \), to account for irreversible transitions in the \( AF \). Partial domain walls which lie at an angle larger than \( \alpha_{\text{crit}} \) would switch to another state with reverse \( AF \) order to that at the interface. Defects in the \( AF \), which provide stabilisation and nucleation sites for reversal at small \( \alpha_{\text{crit}} \) and grain boundaries, which provide nucleation sites are considered in the model by allowing larger values than this critical angle. For \( AF \) thicknesses less than a partial domain wall width, domain walls would unwind.

Schlenker et al. (1986) showed that "distributions of coercivities in the \( AF \) grains" can give both reversible and irreversible effects. Stiles and McMichael (1999a) showed that distributions of other properties, such as crystallographic orientation in the \( AF \), of direct coupling strengths or distributions of critical angles, \( \alpha_{\text{crit}} \), can also produce this behaviour. At the interface of the \( F-AF \) system there is direct collinear coupling and indirect spin-flop coupling. The direct coupling will depend on the degree of compensation and the interfacial area of the spins. On average, the direct coupling per spin, \( J_{\text{int}} \), is shown to be \( J_{\text{int}} / \sqrt{N} \), where \( J_{\text{int}} \) is the interfacial exchange constant between the \( F \), which is supposed to have uniform magnetisation in the \( AF \) grain and \( N \) is the number of interfacial spins per \( AF \) grain. For spin-flop coupling, it is assumed that the \( F \) spins are orthogonal to the \( AF \) spins, the effective spin-flop exchange per spin, \( J_{\text{sf}} \), is given by \( J_{\text{int}}^2 / J_{\text{eff}} \), where \( J_{\text{eff}} \) is the effective exchange coupling between \( AF \) spins, which is proportional to \( J_{\text{AF}} \). Hence the interfacial exchange \( J_{\text{int}} \) is reduced by \( 1/\sqrt{N} \) for direct coupling, while for spin-flop coupling it is reduced by \( J_{\text{int}} / J_{\text{eff}} \).
Therefore, exchange biased systems where $J_{\text{int}} \equiv J_{\text{AF}}$, spin-flop coupling will be stronger than direct coupling, making the AF interfacial spins coupled orthogonally to the $F$ spins. Hence the coupling at the interface will be dominated by the strongest of these interactions. However, it has been shown that spin-flop coupling does not contribute to the unidirectional anisotropy (Schulthess and Butler, 1999). This would imply that the loop shift is caused by direct coupling at the interface between the $F$ and the predominant $AF$ sublattice in each grain. According to Stiles and McMichael, exchange bias exists because the $AF$ order is set in the presence of the $F$ magnetisation. The strength of the bias would be set by the lesser of the strength of the average direct coupling and domain wall energy in the $AF$. Irreversible processes are produced in the $AF$, as domain walls are wound past $\alpha_{\text{crit}}$, leading to irreversible transitions to another state with reverse $AF$ order to that in the interface. For this to occur the coupling needs to be strong enough to reverse $AF$ spins far from the interface. These irreversible transitions put the system in a lower energy state. This implies that on average, the easy direction of the $AF$ points towards the magnetisation of the ferromagnet.

Stiles and McMichael (2001) proposed a model for the temperature dependence of exchange bias based on the same concepts as those described above. Temperature dependence arises from thermal instabilities in the state of the $AF$ grains, as described for superparamagnetic grains in section 2.9, and from the domain wall energy in the $AF$ grains. They assumed that the domain wall energy varies as

$$\sigma = \sigma_0 (1-T/T_N)^{5/6}$$

(4.12)

where $T_N$ is the Néel temperature and $\sigma_0$ is the domain wall energy at 0 K. The $5/6$ factor results from the assumption of $\sigma \propto (A_{AF}K_{AF})^{1/2}$ and the approximations of $K_{AF} \propto m_{AF}^3$ for the uniaxial anisotropy and $A_{AF} \propto m_{AF}^2$ for the exchange stiffness.

Thermal instabilities in the $AF$ arise from thermal fluctuations, which cause small $AF$ particles to switch their magnetic state. These $AF$ grains are considered not to interact with external applied fields, are assumed to be of a columnar nature and longer than an $AF$ domain wall. Grain size determines the type of switching. Small grains will switch by coherent rotation while big grains would switch by domain wall nucleation and
motion. The switching rate is determined in a similar way as for superparamagnetic grains. An energy barrier, $\Delta E$, separates the set $AF$ state and the reversed $AF$ state. Hence the energy barrier, according to Stiles and McMichael (1999, b) is given by the final reversed energy state plus a domain wall far from the interface. These energy barriers will be higher for those grains which are more strongly coupled to the $F$. At a temperature $T$ the probability of remaining in an initial state for a period $t$ is given by

$$P(t) = \exp(f_0 t) \cdot \exp(-\Delta E/k_B T)$$

(4.13)

Where, as described in section 2.9, $f_0$ equals $10^9$ Hz, $k_B$ is Boltzmann's constant. The value of $\Delta E$ is dependent on the coupling strength distribution, the distribution of the axes of the $AF$ grain orientations and the domain wall energy at that temperature. The fast switching transition between states is approximated by a probability chosen to be either zero or one with the border between both states given by

$$\frac{\Delta E}{k_B T} = \ln(f_0 \tau) = 20$$

(4.14)

where $\tau = 1$s. In chapter 2.9, it has been shown that when $\tau = 100$s, then $\Delta E/k_B T = 25$. The chosen value of $\tau$ depends on the measurement time. Hence, considering a measurement time of the order of one second, a particular grain will be stable if $\Delta E < 20k_B T$ and unstable if $\Delta E > 20k_B T$. The model allows instabilities in both states. Thus, after a time $\tau$, these grains can occupy any of the states with equal probability. The critical angle description given before is closely related to the approximation of the state of each $AF$ grain, as stable, partially stable, i.e. remains in one of the states as the energy barrier for that state is high, but the other has a low energy barrier, or unstable where both states have low energy barriers, so the grain will flip from one state to the other on a fast time scale compared to the measurement time. A particular grain will behave according to these processes depending on the temperature of the system. High temperatures make the grain more unstable in the given measurement time, while low temperatures increase the relaxation time, which makes the grains more stable as the relaxation time approaches the measurement time. The manner in which each grain behaves determines its contribution to the unidirectional anisotropy, and therefore to the shift of the loop. Consequently, the thermal activation of the switching causes a
blocking temperature, $T_B$. Above $T_B$, the AF order in the grains is not stable and therefore those grains do not contribute to the unidirectional anisotropy.

The unidirectional anisotropy saturates at low temperatures depending on ratio between the average interfacial coupling energy, $J_{\text{net}}$, to the zero temperature domain wall energy, $\sigma_0 a^2$, where $a$ is the area of the interface per spin. The smallest of both determines the size of the unidirectional anisotropy. If $J_{\text{net}}$ is the smallest, the unidirectional anisotropy saturates at low temperature due to the assumed temperature independence of the interfacial coupling at low temperatures.

A distribution of grain sizes and hence of blocking temperatures would explain the history dependence found in some systems. If there are such distributions, each grain would start to contribute to the unidirectional anisotropy at a different temperature. If the magnetisation direction in the F is changed as temperature is reduced, different grains will contribute to the unidirectional anisotropies in different directions.

Regarding the increase in the values of the coercivity in relation to those observed for a single F layer, the model of Stiles and McMichael (2001) exhibited two contributions to the enhancement of $H_C$. The first is due to inhomogeneous reversal in the F, which arises from the inhomogeneous environment to which the F is coupled in the exchange biased system, taking into account the random distributions of easy axes in the AF grains. The second is due to irreversible transitions occurring in the AF grains. The temperature dependence of $H_C$ is then governed by the behaviour of these factors. At low temperatures the contribution of inhomogeneities is higher. This contribution depends on the ratio of the exchange coupling of the F to the AF grains to the intergranular exchange coupling in the F, given by the exchange stiffness. As a result $H_C$ is predicted to decrease as $1/t_F^2$. At higher temperatures, irreversible transitions in the AF grain are predominant. This effect is independent of the ratio of the interlayer coupling to F exchange stiffness. In this range of temperatures $H_C$ is estimated to decrease as $1/t_F$. This model also accounts for the observed peak in the value of $H_C$ with temperature close to the blocking temperature. This is associated with instabilities in the AF grains. These simulations have been carried out with a finite sweep-rate. For
large values of $t_F$, the behaviour of both $H_C$ and $H_{ex}$ are obscure due to the dependence of both on the sweep-rate and the time-scale of the measurements.

### 4.5 Domain Models

Nowak *et al.*, (2002) proposed a different approach to explain the exchange bias phenomenon. Based on the fact that the models of Stiles and McMichael (1999a, 1999b, 2001) does not explain the occurrence of instabilities in the uncompensated interface during a hysteresis loop, Nowak *et al.*, (2002) reported on a model of exchange bias in which the $F$ is exchange coupled to a diluted $AF$. The $AF$ is modelled as an Ising system, and Monte Carlo simulations used to study the behaviour of the $F$-$AF$ bilayer. They include the effect of the whole $AF$ layer on the basis of its influence on the formation of domains close to the interface. The basic concept of this model is that on field cooling, in which the $AF$ spins are in contact with the $F$ spins, it is also exposed to the applied field, there is a development of a domain state in the $AF$ which carries magnetisation. The magnetisation of the domain is a metastable state which is formed and becomes frozen during the field cooling process. The model assumes that the domain wall widths are close to zero, and the domains have small size, so that it would be possible to fit a large number of domains in the system. The dilution of nonmagnetic defects in the $AF$ promotes the formation of domains in the $AF$, increasing its magnetisation ($M_{AF}$) and in consequence, $H_{ex}$ would increase. The existence of $M_{AF}$ is shown to give rise to a vertical shift of the resulting hysteresis loops from the simulation. The whole $AF$ would contribute to this $M_{AF}$.

The temperature dependence of $H_{ex}$ obtained is a linear decrease with increasing temperature. The exchange field goes to zero at a temperature $T_B$, smaller than $T_N$. The reduction of $H_{ex}$ with repeated field cycling is explained as being due to a loss of magnetisation in the $AF$ during the hysteresis loop due to a rearrangement of the $AF$ domain spins. In their simulations, this loss is estimated to be less than 10% of the $M_{AF}$. These two predictions of exchange bias are discussed in chapter 6.

Keller *et al.*, (2002) studied the exchange bias dependence on dilution on Co/CoO with nonmagnetic inclusions of CoMgO. They studied various exchange bias phenomena
such as the vertical shift in the magnetisation, the dependence of $H_{ex}$ on temperature the cooling field dependence, training effects, and the variation of $H_{ex}$ with the thickness of the AF. Their results are consistently described by the domain state model (Nowak et al., 2002).

The appearance of positive exchange bias due to a high applied field during field cooling (Nogués et al., 2000) is explained as being due to the sign of the exchange coupling between the F and the AF. Negative F-AF coupling overcome by a strong field applied during field cooling would bias the $M_{AF}$ resulting in positive $H_{ex}$.

The dependence of $H_{ex}$ on the AF thickness, $t_{AF}$, has also been studied. For a low dilution, increasing $t_{AF}$ increases the energy of the domain walls. The energy required to form domain walls can be reduced by increasing the domain sizes and reducing the number of domain walls. Hence a decrease of $H_{ex}$ is expected. For higher dilutions an increase in $t_{AF}$ would have a smaller effect on the domain wall energies, leading to a constant $H_{ex}$. For very small $t_{AF}$, $H_{ex}$ is almost zero up to a critical thickness where $H_{ex}$ is set, as explained by Stiles and McMichael (1999a). The main achievement of this model is that it explains features of exchange bias without the need to assume compensated or uncompensated interfaces. Without domain formation, it predicts that there is no $H_{ex}$ for compensated interfaces, and a very high $H_{ex}$ for uncompensated interfaces. The inclusion of defects favours the formation of domains, independently of the nature of the interface.

Using a different approach in the numerical treatment, Scholten et al., (2005) performed mean-field calculations in a diluted system similar to the one described by Nowak et al., 2002, with the difference that this time the interface is assumed to be compensated. The results obtained are similar to those of Nowak et al. According to this model the $H_C$ enhancement shows a maximum around the Néel temperature, independent of the dilution of the AF. The enhancement of $H_C$ persists at temperatures higher than $T_N$, although it decreases slowly for thick AFs. Nowak et al. investigated the origins of this coercivity enhancement as well as the origin of $H_{ex}$. They concluded that both $H_C$ and $H_{ex}$ have different origins. They observed larger coercivity enhancement for antiferromagnetic coupling at the interface between F-AF than for the case of ferromagnetic coupling. The enhancement of $H_C$ is due to the coupling to the AF layer as well as the appearance of a shift in the hysteresis loop. They considered the...
interfacial magnetisation of the AF as the sum of two components, an irreversible metastable AF magnetisation, $M_{AF}^{irr}$, which does not change when going through the hysteresis loop at low temperatures, and a reversible AF magnetisation, $M_{AF}^{rev}$, which follows the field. They concluded that $H_{ex}$ exists only if the interfacial magnetisation contains a component that is frozen during field reversal. Hence, $M_{AF}^{irr}$, would contribute to $H_{ex}$. Regarding the enhancement of $H_C$, they predict the peak with temperature. The peak shifts to lower temperatures for increasing $t_{AF}$, while $H_{ex}$ decreases with increasing temperature. Depending on the degree of dilution, the peak in $H_C$ is found to appear at the same temperature at which $H_{ex}$ vanishes, $T_B$. For higher dilutions the peak is found to shift to lower temperatures. Hence, an assignment of the origin of the coercivity enhancement to $M_{AF}^{rev}$, and the origin of $H_{ex}$ to $M_{AF}^{irr}$ is predicted by these simulations and observed experimentally (Keller et al., 2002). In fact the percentage of the AF spins that contribute to $M_{AF}^{irr}$, i.e. that part of the AF spins that are dragged with the F, increases with temperature (Nogues et al., 1999). Therefore, a direct proportionality between each pair of parameters would be expected, leading to the simultaneous occurrence of a minimum in $M_{AF}^{irr}$ and a maximum of $M_{AF}^{rev}$ at the temperature at which $H_{ex}$ vanishes, i.e. $T_B$. In chapter 6 it will be shown that $H_{ex}$ vanishes at different temperatures depending on the measurement procedure, and that the peak in $H_C$ does not correlate with the median blocking temperature. Thus, different locations for such particular state that maximises $M_{AF}^{rev}$ can be found. Scholten et al. (2005) suggest that both the enhancement of $H_C$ and $H_{ex}$ are not directly related as one of them can be changed without changing the other.

### 4.6 Experimental Studies

Experimental studies by van der Heijden et al., (1998a, 1998b) showed that when the $M_F$ is antiparallel to the unidirectional anisotropy in the AF the decrease of $H_{ex}$ increases with temperature and is independent of the field applied to the F. This dependence is interpreted as a thermally assisted relaxation process of the magnetic order in the AF grains in the same manner as explained by Fulcomer and Charap (1972b). This can lead not only to a decrease of $H_{ex}$ but also to a reversal of $H_{ex}$. The
results obtained by van der Heijden (1998a) also showed that the time dependence of $H_{ex}$ cannot be described as a single exponential function as proposed by Fulcomer and Charap (1972b), but needs to be described by a distribution of relaxation times, $\tau$. Due to the fact that the $AF$ grains are non identical, neither are the domains that form in the $AF$. The relaxation of the magnetic order of the $AF$ grains, is not only dependent on the magnetisation direction of the $F$, but also on the temperature and the cooling rate of the field cooling procedure in which the $AF$ order is set (van der Heijden et al., 1998b, Gökemeijer et al., 1999a, 1999b). Similar results obtained for different thicknesses in the $AF$, showed the limitations of the assumption made by Fulcomer and Champ (1972b) that the $AF$ grains were monodomain. The study of magnetisation reversal in FeMn spin valves (van der Heijden et al. 1998a) showed that reversal over a distribution of energy barriers is similar to the thermal activation of reversal in a ferromagnet. van der Heijden et al. (1998b) observed the decrease of $H_{ex}$ with increasing temperature while the field applied is kept in reverse bias. This was interpreted as a thermally assisted reversal of the grains in the $AF$.

Goodman et al., (1999) studied the effect of the sweep-rate and time at negative saturation on exchange biased systems. They found that each of these two effects is dominant over the other depending on the time spent at negative saturation. When the time spent in negative saturation is less than a certain time the sweep-rate is the dominant and due to viscosity effects a decay of the values of $H_{ex}$ was observed. Otherwise, the effect of time spent in negative saturation dominates. This effect was observed to be manifest as a decay of the $H_{ex}$ due to thermal reversal of the $AF$ grains over a distribution of energy barriers to reversal $f(\Delta E)$, which occurred while the $F$ was kept in negative saturation (Goodman et al., 2001). This study did not address the origin of $f(\Delta E)$, although they suggested interfacial effects as a possible mechanism in the same context as Stiles and McMichael (1999a).

The study of the temperature dependence of thermal activation at negative saturation on the hysteresis loop leads to a progressive shift of $H_{ex}$ towards zero. As the amount of $AF$ grains that reverse increases $H_{ex}$ achieves positive values. Differentiation of the variation of $H_{ex}$ with respect to the temperature of thermal activation gives a measure of the distribution of the energy barriers to reversal $f(\Delta E)$, (O'Grady et al., 2002).
The model of Stiles and McMichael (1999b) describes exchange bias in a similar way to the interpretation that it is given in this thesis. This will be described in chapter 6 where results of the experiments are shown and interpreted. The main difference between the granular models described and the interpretation of exchange bias is the fact the systems studied in this thesis are cubic systems, hence, the uniaxial anisotropy used in the model is an approximation for biaxial systems. Furthermore, the fact that most of the parameters related to exchange bias are distributed or unknown, it is possible to find experiments that would fit any model, or vice versa. This is why, measurement reproducibility is the basic pillar of the experiments performed in this work.

It seems unlikely that there is a single explanation for all exchange bias effects. In this thesis it is shown that the effects of interface and the effects of volume of the $AF$ appear to be independent. From the data obtained, it is very clear that any model that ignores thermal effects cannot predict exchange bias. This explains the use of fitting parameters in the models. Also, the majority of experimental measurements reported in the literature are not made on samples for which the magnetic and thermal history is known. It is the objective of this work to develop methods of measurement where this issue is rectified.
Chapter 5

Measurement Techniques and Protocols

5.1 Introduction

As explained in chapter 4, the critical aspect of this work is the development of measurement protocols. The objective is not to do a survey of samples. The samples selected for use were IrMn/CoFe in a range of thickness of IrMn. They were selected because IrMn is the favourite material for application in recording heads (Carey, 2003). The development and use of the measurement protocol is a lengthy process. It can take up to two weeks to fully characterise one sample. Therefore, only two samples were selected for measurement. As the focus of the work is on thermal activation, samples with the thinnest AF layers, which are most thermally active, were selected. This is important because for the need of CPP read heads and ultimately perpendicular recording, the overall thickness of the head stuck is a critical parameter and thinner AF layers must be used. It should be noted that in a head stuck the thickest layer is the one formed by the AF.

The samples used in this work are described in the next section. The equipment used to develop the measurement protocols subject of this thesis is described in sections 5.3 and 5.4. After development of the protocols measurement and thermal characterisation of an exchange bias sample should be straight forward. Each of the protocols developed is a further step in the characterisation of the subject exchange biased system. Each protocol is formed by different stages, some of them common to all the protocols. These stages are such as saddle pointing, AF reset, and cool to the free thermal activation temperature. The measurement protocols are described in section 5.5. Each of the experiments is described briefly and the measurement protocol followed is given in section 5.6.

The physical reasons to develop the protocols like they are is given in chapter 6 where the experiments are described. In chapter 6, it is presented the experimental procedure
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with its motivation, as well as the results of the experiment and the corresponding discussion.

5.2 Sample Preparation

Exchange biased thin films are produced mainly by dc, or rf sputtering (see section 2.2). Sputtered magnetic thin films are polycrystalline, while single-crystal samples are grown by Molecular Beam Epitaxy (MBE).

The samples used in this work were grown by magnetron sputtering on silicon wafers with (111) texture. The multilayer structure is the following: //Seed(5nm)/Cu(1nm)/Ir$_{30}$Mn$_{70}$(t$_{AF}$)/Co$_{90}$Fe$_{10}$(10nm)/Ta(5nm), where t$_{AF}$ is the AF thickness, equal to 3 and 5 nm. Both samples were deposited in a field and post annealed at 250°C for two hours. Samples were provided by M. J. Carey, from Hitachi Global Storage Technologies, San Jose, CA.

5.2.1 Polycrystalline Samples

Sputtered samples are polycrystalline and formed of grains each of which is single crystal. Magnetic properties of the polycrystalline materials depend crucially on the distribution of grain sizes (Vopsaroiu et al., 2005). The grain size distribution has been probed by use of the cumulative percentage method, and is best fitted by a lognormal distribution (Park et al., 2000). The lognormal distribution of the grain size, by which the logarithm of the particle diameter has a Gaussian distribution, also applies to other types of films such as ultrafine metal particles (Granqvist and Buhrman, 1976).

Magnetic properties of exchange coupled systems are very sensitive to the grain size distribution. It has been ten years of work to reduce thermal activation due to small grains. In $F$ material it has been shown that thermal activation arises from crystallinity. This arises from the dependence of the anisotropy on the crystallinity. Recent work by Tsunoda et al. (2005) showed that the increase in the degree of order in the IrMn layer, measured as the increase in $L_{12}$ phase (figure 41) that could be formed in the IrMn,
resulted in giant exchange anisotropy (1erg/cm²). The increase in the degree of order was obtained by substrate heating and thermal annealing (Tsunoda et al., 2004). This indicates that the crystallinity of the AF grain in polycrystalline grains may be critical. Hence, the use of seed layers and thermal annealing to improve crystallinity and reduce thermal effects. These are discussed in section 6.2. Depending on the degree of crystallinity, the interface quality and how susceptible to thermal activation, only a certain portion of the AF grains will contribute to exchange bias. The percentage of grains in the AF needed to produce exchange bias was probed to be much less than the expected in measurements of thermal activation shown in section 6.2. At 300 K the percentage of active AF grains for IrMn(5nm)CoFe(10nm) resulted to be 11%, while for IrMn(3nm)CoFe(10nm) is 80%.

Manzoor et al., (2005), have shown that FeMn/NiFe exchange biased bilayers are more thermally active for smaller grain size in the AF layer. Their results showed that $H_{ex}$ increased with increasing AF grain size, which was measured as the median of the distribution. This effect shows that for FeMn the energy barriers to reversal are grain size dependent although it is not clear if this is a volume or area dependence. The influence of the AF grain size on the $H_C$ was small, suggesting different origins for $H_{ex}$ and $H_C$. All these results were obtained for samples with the same parameters in the multilayer structures (composition thicknesses, and the F grain size) while keeping the AF grain size as the only variable and measured at room temperature.

Exchange bias features have been observed in both single crystal and polycrystalline samples and spin glasses (Kiwi, 2001). This shows that exchange bias is an intrinsic property of the materials. Most of the studies of exchange bias to investigate the magnetic structure of the F-AF interface have focused in single crystal AF films due to the fact that single crystals are easier to simulate in theoretical models (Matsuyama et al., 2000). Nogués et al., (1996b), found that $H_{ex}$ increases with decreasing interface roughness FeF2/F single crystal samples grown on MgO seed layers, Notling et al., 2000, observed the alignment of spins of polycrystalline Co grown over single crystal LaFeO₃.
5.2.2 Single Crystal Samples

Single crystal samples are mostly grown by MBE. This deposition technique is based in the evaporation of the materials to be deposited by heating them to temperature close to their melting point. Other techniques used, such as chemical vapour deposition (Yang et al., 2000), have in common with MBE that the deposition rates are very slow to achieve epitaxial single crystal growth, even when single crystals are used as substrate (Riedling et al., 1999, Fitzsimmons et al., 2000). The miniaturisation of GMR spin-valves for read heads used in the magnetic recording industry will reach a stage where the dimensions of the spin valves will be similar to the thin film grain diameter. This implies that spin valves could be formed by single crystal elements whose magnetic and transport properties might differ significantly from polycrystalline spin-valves (Sato et al., 2005). It is known that the change from single crystal to polycrystalline structure modifies the formation of domain walls in the AF (Gökemeijer et al., 1999a). There are two main differences between single crystal and polycrystalline thin films. Polycrystalline films...
have random easy axes in both the F and AF grains and the interaction between grains varies and in some cases is very weak.

In the F, despite of its granular nature there is strong RKKY coupling, as commonly found in soft films. The nature of exchange in granular alloyed AF is not known. However, due to the requirement for atomic ordering on a crystallographic lattice it seems unlikely that strong intergranular exchange would occur.

Single crystal films have defined anisotropy directions along the film and the interactions between spins are stronger (Li et al., 2001). The use of single crystals to study exchange bias systems has been based mainly in AF single crystals coated with a F (Nogues et al., 1999). The growth direction of the AF determines the anisotropy values. Epitaxial IrMn(110) grown on single crystal MgO(110), which has a very well defined crystal structure and very smooth interface, showed higher anisotropy than in the other (001), (111) orientations (Sato et al., 2005). Single crystals have the advantage that they can exhibit a more perfectly compensated or uncompensated spin structure, depending on the growth direction, leading to different properties such as interface roughness, F/AF relative spin orientations, different magnitude of Hex (Berkowitz and Takano, 1999, Nogués and Schuller, 1999).

5.2.3 Effects of Deposition in a Field

In the deposition of a F/AF bilayer, the order in which the F and the AF are deposited plays a significant role. If the AF is grown first, it will grow with a certain texture depending on the seed layer that it is used (Tsang et al., 1981, Kerr et al., 2005). After this the F layer is deposited and to induce uniaxial anisotropy in the F and orient its magnetisation, a dc field is applied in the plane of the film during deposition (Ambrose et al., 1998). The magnitude of the field that is normally used for deposition of CoFe varies in the range of 50-300Oe (Jung et al., 2003). The application of a field during deposition, reduces the anisotropy dispersion in the F as an effect of the applied field on the magnetocrystalline anisotropy of the F grains during growth.
The effect of the deposition of the $F$ prior to the deposition of the $AF$ in a dc field provides uniaxial magnetic anisotropy in the $AF$ due to the exchange interaction of the $F$ spins acting on the $AF$ spins. If the $F$ is deposited in zero field, the $AF$ will have no unidirectional anisotropy as the $F$ spins will be deposited with no preferential direction. To obtain higher unidirectional anisotropy it will be necessary to field anneal the bilayer. These effects have been observed on CoFe/IrMn spin valves (Anderson et al., 2000). When the $AF$ is grown first the exchange bias obtained is less than for the opposite case. When these samples are field annealed $H_{ex}$ increases for the samples with the $AF$ layer deposited first while for the ones with the $AF$ grown on top both magnitudes decrease. These effects are suggested to be due to the $H_{ex}$ dependence on the crystalline texture and the impact of thermal annealing on texture and interdiffusion (Anderson et al., 2000). In comparison, field annealed samples with the AF on top show larger $H_{ex}$ than those with the $AF$ layer at the bottom (Kerr et al., 2005). Growth order also influences the temperature dependence of $H_{ex}$, which resulted to be quasi-linear for samples with the $F$ grown first, while $H_{ex}$ showed a plateau and a sudden drop for temperature close to $T_B$ when the $F$ was grown on top (Ambrose et al., 1994). No explanation has been suggested for this by the authors. A possible explanation for this could be based on different anisotropy temperature dependence due to different degrees of crystallographic ordering (Tsunoda et al., 2005), or due to different grain growth results as a consequence of different growth order.

5.2.4 Effects of Annealing

The effects of annealing have been subject of many studies of exchange biased systems as a means to improve the performance of GMR and MR heads (Gregg et al., 2002). They were mainly aimed at improving the thermal stability of the exchange bias field. The effects of annealing on an exchange bias system depends on the temperature of the anneal, the time of the anneal, the stage of sample growth at which it has been applied and whether a field is applied during the anneal. The system most studied in the literature has been FeMn/NiFe, although PtMn/CoFe and IrMn/CoFe are the most promising for industrial applications.
Typical temperatures of thermal annealing are 250-350°C. Studies by Imakita et al., (2005) on an Ir27Mn73/Co70Fe30 exchange coupled bilayer containing CrNiFe/Cu as a under layer showed the increase of the lateral size of the Cu grains before growth of the AF/F bilayer with increasing temperature of annealing. The increase of the grain size has a narrowing effect on the distribution of blocking temperatures of the AF grains through the change in grain size distribution of the IrMn layer. Hence thermal stability of $H_{ex}$ is achieved when the sample is annealed under a field of 1 kOe for 1 hour. Figure 42 shows a TEM image of the Cu underlayer.

![TEM image of Cu underlayer](image)

**Figure 42** TEM plane view of a Cu 50nm underlayer, deposited on Cr-Ni-Fe 5nm buffer layer at 250°C in ultra-high-vacuum, annealed at 250°C, 30Oe for 10min (Imakita et al., 2005).

An increase in the exchange field and coercivity through annealing has been reported by several authors. Studies on NiFe/CrMn by Xi et al.,(2000), reported an increase of $H_{ex}$ and $H_C$ after annealing at 310°C in a 600Oe applied field as consequence of the diffusion of Mn into the NiFe layer. This produces an AF NiMnFe interlayer increasing the exchange field. As a matter of fact, the $T_N$ of alloyed NiMnFe exceeds the $T_C$ of NiFe. Figure 43 shows VSM hysteresis loops for this system after annealing. It shows the results of field anneal on coercivity, exchange bias and saturation magnetisation.

The diffusion of Mn in the F layer reduces the magnetic moment of the F as observed by Rickart et al., (2005), on PtMn/CoFe exchange biased bilayers after rapid thermal
anneal (5.5°C s\(^{-1}\)) up to 320°C in a field of 3kOe. Another feature of annealing is the promotion of structural changes in the sample. A one minute anneal induces a structural phase transition in the PtMn from fcc to AF fct without cooling in a magnetic field. Toney et al., (1991), observed two kinds of change on the exchange field depending on the time of annealing on NiFe/FeMn. Moderate annealing produced a change in the dependence of \(H_{ex}\) with temperature from linear to Brillouin shape dependence, reflecting the magnetisation of the AF with temperature. Auger spectroscopy showed significant interdiffusion. At extended annealing times; interdiffusion of Mn on NiFe produced a NiFeMn alloy which increased the \(H_{ex}\).

![Figure 43 VSM hysteresis loops of annealed Ni\(_{41}\)Fe\(_{19}\)/Cr\(_{50}\)Mn\(_{30}\) samples (Xi et al., 2000).](image)

The effects of annealing described up to this point can be summarised as a narrowing of the local blocking temperatures, increase of lateral grain size in the AF, phase transitions, diffusion, interface degradation through interdiffusion, alloying, and a decrease of the saturation magnetisation.

Kerr et al., (2005), studied the effects of field annealing on top and bottom spin valves with IrMn/CoFe as the exchange biased layer. It was observed that for bottom spin valves the \(H_{ex}\) increased drastically with the magnitude of the field applied during annealing, showing that the interfacial spin structure of the IrMn improves with the anneal field strength. The explanation provided for this was based on the uncompensated interfacial spin structure. The annealing temperature promotes diffusion of Mn increasing the number of defects and therefore the number of pinning sites,
increasing the number of uncompensated interfacial spins. The effect of the annealing field is to improve the alignment of the uncompensated interfacial spins in the direction of the applied field. For the sake of clarity, table 3 quotes some of the results obtained in this study.

<table>
<thead>
<tr>
<th>Anneal Field (x10^4 Oe)</th>
<th>$H_{ex}$ (Oe)</th>
<th>AR/R</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>230</td>
<td>7.8%</td>
</tr>
<tr>
<td>0.5</td>
<td>360</td>
<td>8.2%</td>
</tr>
<tr>
<td>5.5</td>
<td>390</td>
<td>8.2%</td>
</tr>
</tbody>
</table>

Table 3 Exchange field dependence on the anneal field strength (Kerr et al., 2005).

It was also observed that in the case of bottom spin valves although the $H_{ex}$ dependence on temperature of anneal has a peak around 180°C the magnetoresistance stayed approximately independent of temperature up to 300°C. For top spin valves $H_{ex}$ as well as the magnetoresistance decreased with increasing annealing temperature. It was observed that interdiffusion of Mn from IrMn to the CoFe/Cu interfaces was more pronounced than in the case of bottom spin valves. Therefore, although there is interdiffusion in both top and bottom spin valves, it is activated by different thermal processes in each case.

5.3 The Vibrating Sample Magnetometer

All the measurements contained in this thesis were made using a Vibrating Sample Magnetometer, VSM. The first VSM was developed by Foner in 1956 (Foner, 1956, 1959, 1996). This instrument has been shown to be very versatile and have high sensitivity, being simple and inexpensive and offering advantages over force methods for magnetic measurements (Wolf, 1957). These advantages include temperature variation, sample rotation and sample changing and reproducible sample positioning.

Since 1956 this instrument has been improved and modified for specific measurement needs (Mallison, 1966; Flanders et al. 1993), being used for most accurate measurements of the properties of recording media and magnetic materials. Commercial
VSMs are equipped with features such as computer control and automatic data acquisition, temperature control and sample positioning (Mee and Daniel, 1996). The VSM used to perform the experiments described in this thesis is a modified *EG&G Princeton Applied Research Model 4500 VSM*.

### 5.3.1 Principle of Operation

The principle of operation of the *PAR 4500 VSM* is the induction of a magnetic moment on the sample studied by means of an applied DC magnetic field to the sample. This field is generated by a water-cooled electromagnet that can apply fields up to 1 Tesla. The sample is placed in the centre of the uniform magnetic field and vibrated at small amplitude with known frequency and phase. The vibration of the magnetic sample is perpendicular to the magnetic field lines. This produces a field distortion around the sample position and therefore a change in the magnetic flux of a set of 4 pick-up coils placed near the sample. The voltage output from the pick-up coils is processed to give a measurement of the magnetic moment induced as emu/volt. These pick-up coils are clamped to the poles of the electromagnet to avoid vibration of the coils with respect to the applied field. A schematic of this VSM is shown in figure 44.

The *PAR 4500 VSM* used is controlled with LabView software designed in the magnetism group. The original analogue lock-in amplifier and synchronous detector have been replaced with an *EG&G 7265 DSP lock-in amplifier*. This resulted in an increase of the sensitivity to less than $5 \times 10^{-6}$ emu for a time constant of 100ms. The field resolution depends on the field range selected for the type of measurement, being less than 1% in the range of applied fields less than 1kOe, and better than 0.1% in the range of applied fields below 10 kOe. Set fields are stable to better than 10e/hour.

The vibration mechanism is a head transducer assembly with a power amplifier. It produces a sinusoidal oscillation of the sample perpendicular (z-direction) to the DC applied magnetic (x-direction) field via a carbon fibre rod. The frequency of oscillation is constant, 81.366Hz and its amplitude is 2mm. The 4 pick-up coils are arranged according to Mallison’s design (Mallison, 1966) as shown in figure 45. Each pair of coils is located in each pole along the z-direction, with their centre of symmetry in the...
middle of the homogeneous field space. Each pair of coils is wound in series opposition. The purposes of this are to add the induced voltages coming from the north and south poles of the sample and cancel any induced changes in the field. The ac voltage induced in the pick-up coils, \( V \), is proportional to the sample moment and to the amplitude and frequency of the oscillation. The voltage \( V \) is given by Faraday's law,

\[
V = -N \left( \frac{d\Phi_f}{dt} \right) = NA \left( \frac{dm}{dt} \right)
\]  

(5.3)

\( N \) is the number of turns in the coil, \( \Phi_f \), is the change in flux after a common field has been cancelled, \( t \) is the time, \( A \) is the cross sectional area, and \( m \) is the magnetic moment. The induced signal in the coils is compared to a reference signal produced by a three plate capacitor placed below the transducer that produces the oscillation. This reference signal is also proportional to the amplitude and frequency of the oscillation.

To obtain reproducible data it is necessary to relocate the sample exactly in the same position relative to the pick-up coils. To obtain good repositioning the sample has to be "saddle pointed" in the centre of symmetry of the pick-up coils. This is done using micrometer positioners, which change the position of the sample along \( x, y \) and \( z \) axes. At the centre of symmetry it is found that the output from the pick-up coils is a maximum in the \( z \) and \( y \) directions and a minimum in the \( x \) direction. This point is easier to find if the order \( z, x, \) and \( y \) positioning is followed.

To obtain absolute measurements of the magnetic properties of a material the VSM needs to be calibrated against a reference signal from a material with known properties. For accurate calibration the sample used for calibration must be of similar size and geometry as the sample of interest to obtain a similar geometric response from the coils. In the case of thin films, a Palladium or Nickel foil of known mass, and therefore magnetic moment is used. These are chosen depending on the value of the moment of samples to be measured. As Palladium is a Pauli paramagnet, it has a linear response to the applied field in a wide range of temperatures. Hence, a range of calibration moments can be obtained by applying different fields. In practice the process is aimed at obtaining the emu/volt constant of calibration. The field is calibrated with a hall probe.
This hall probe is calibrated using a NMR proton magnetometer with a frequency tuned according to the broadcast frequency standard.

Figure 44 Schematic of a VSM.

Figure 45 Schematic of a set of pick-up coils.
5.4 Temperature Control

The most of the work involved in this thesis is related to the temperature dependence of the magnetic state of exchange biased systems. The cryostat used to achieve the desired measurement environments is an **Oxford Instruments Ltd CF1200** continuous flow cryostat. This cryostat has two vacuum jackets. An outer jacket isolates the cryostat from temperature interactions with the exterior environment. The inner jacket is the sample space. A vacuum insulated transfer tube delivers the coolant to the cryostat through a capillary. Along the feed capillary the coolant runs down the outer jacket to the bottom of the sample space. There, the coolant is fed to a copper heat exchanger, which is fitted with a temperature sensor and a heater. The heater is a 70Ω resistor, located inside the heat exchanger as well as the temperature sensor.

A roughing pump and a diffusion pump connected were used to vent the cryostat. This vacuum system provided pressures below $10^{-5}$ mbar in the outer jacket. When using liquid nitrogen as coolant, the pressure in the inner jacket was as low as $10^{-3}$ mbar. In order to achieve low temperatures quickly, the vacuum in the outer jacket would be removed whilst quenching the temperature by dipping the bottom of the cryostat in liquid nitrogen. This method provided temperature variations of more than 40K/min.

When the coolant was liquid helium this procedure is not possible, the sample space needs to be filled with an exchange gas such as He gas. Thus, a quenching process to reduce the temperature would be redundant.

The temperature is controlled with an **Oxford Instruments Ltd ITC4 Intelligent Temperature Controller**. The principle of operation of the temperature controller is governed by a proportional, integer, and derivative control. The values of these parameters must be selected correctly to obtain optimum control over temperature. The approach to the set temperature with a same set of parameters may vary with the range of temperatures involved. This means that the values chosen for a certain system had to be varied depending on the measurement temperature.

The cryostat produces a slight shielding of the signal produced by the sample. Also, careful control of the sample position relative to the cryostat walls must be taken. It is required that the sample, sample holder and the carbon-fibre rod do not touch the walls.
of the cryostat at any point. The effect of this would be the addition of noise to the measurement. The cryostat must not touch the pick-up coils of the VSM. This fact would add noise, which would be proportional to the applied field. Another source of noise is the formation of eddy currents around the jacket of the sample space, which would have a stronger effect at lower temperatures. This manifests as a reduction in the signal that is picked-up by the search coils. To prevent the formation of eddy currents the inner jacket is fitted with a cooper belt with vertical slots.

Recently some changes have been made in the Oxford Instruments Ltd CF1200 to increase the temperature range of utilisation of the cryostat. This implied the substitution of the platinum resistor Pt-100 sensor, which was originally fitted in the heat exchanger, for a 27Ω Rhodium-Iron resistance thermometer. Following this process, three point calibration was required. The cryostat could also be used as an oven for temperatures up to 400K. The materials used in the construction of the cryostat limited the highest temperature of measurement. In consequence, these changes allow temperature measurements in the range of 4.2K to 400K. The temperature resolution obtained is better than 0.5 K over experimental time scales. New sample holders had to be made to work in this range of temperatures.

5.5 Measurement Protocols

The protocols developed in this thesis are aimed at obtaining measurement reproducibility. The structure is the same for all measurements and is self contained, and complete. The format is similar to those used in industry according to ISO 9002 standards. A measurement protocol contains all the necessary steps to perform the experiment. Hence, the measurement process is simplified, clearer, and more efficient. The history of the sample for the experiment is registered, allowing simple and fast revision. In the case of any special occurrence during the experiment, there is a section for comments. The following sections describe these protocols for each experiment in a short paragraph followed by a copy of the measurement protocol itself.
5.5.1 Resetting of the AF state

The sample is aligned with the unidirectional anisotropy axis parallel to the applied field and saddle pointed in the VSM. The process of saddle pointing has been described previously in section 5.3.1. Following this step a positive field is applied which is strong enough to saturate the ferromagnet. The temperature is increased while the sample is kept under the influence of the applied field. This situation is maintained for a certain amount of time. The temperature is then lowered to the temperature of measurement. At this temperature a hysteresis loop is measured cycling the field from positive to negative values while the temperature is kept constant.

For IrMn(3nm)/CoFe(10nm) a temperature of 373K and 90min under a saturating field resulted to be good conditions to reset the AF. For IrMn(5nm)/CoFe(10nm) the use of the same conditions were sufficient to delete previous thermal activation and obtain reproducible data. See section 6.2.2. The procedure to determine the AF state resetting conditions is described in figure 46.

AF Reset
1. Saddle point
2. Align sample
3. Temperature of measurement
4. $T_m =$
5. $(P, I, D) (T_{reset}) =$
6. Positive Saturating Field
7. $H_{sat} =$
8. \Rightarrow Go to point 1 $t_1 =$
9. Heat to $T_{reset} =$
10. Time needed to reach $T_{reset}$
11. $t_1 =$
12. $t_e =$
13. $t (T_{reset}) =$
14. $(P, I, D) (T = T_m) =$
15. Cool to $T = T_m$
16. Time to reach $T_m$
17. $t =$
18. Time spent in point 1:
19. $t =$
20. Measure the loop \Rightarrow

Figure 46(a) AF reset measurement protocol.
5.5.2 Thermal Activation Free Temperature

The alignment stage is the same as the previous experiment. Field and unidirectional anisotropy axis are set parallel. This is followed by the resetting of the magnetic state in the antiferromagnet. The conditions for this are characteristic of the sample under study. The outline of the process is the following: apply a positive saturating field to the ferromagnet, increase the temperature, and maintain these conditions for a fixed time. Then, the temperature is lowered to the temperature of measurement. This measurement is similar to the one described previously. In this case the variable is the temperature of measurement while previously the variables were the $AF$ resetting conditions. The criteria that show when it is achieved the thermal activation free temperature, $T_{NA}$, is given in section 6.2.2. A copy of the measurement protocol for this experiment is shown in figure 47.

<table>
<thead>
<tr>
<th>Thermal Activation Free Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Saddle point and alignment section</td>
</tr>
<tr>
<td>2. Temperature of No thermal Activation?</td>
</tr>
<tr>
<td>3. $T_{NA} =$ ____________________</td>
</tr>
<tr>
<td>4. $(P, I, D)$ (100°C) = ____________</td>
</tr>
<tr>
<td>5. Positive Saturating Field</td>
</tr>
<tr>
<td>6. $AF$ Resetting Section ____________</td>
</tr>
<tr>
<td>7. Cool to $T = T_{NA}$ _______________</td>
</tr>
<tr>
<td>8. Time to reach $T_{MA}$ t = ___________</td>
</tr>
<tr>
<td>9. Time needed to stabilise $T_{MA}$</td>
</tr>
<tr>
<td>10. $t_9 (T_{MA}) =$ ________________</td>
</tr>
<tr>
<td>11. Time spent in point 1:</td>
</tr>
<tr>
<td>12. t = ___________________</td>
</tr>
<tr>
<td>13. Negative Saturating Field</td>
</tr>
<tr>
<td>14. $H_{sa} =$ ________________</td>
</tr>
<tr>
<td>15. Go to point 4 $t_i =$ ________</td>
</tr>
<tr>
<td>16. Time at Point 4 = _______ min</td>
</tr>
<tr>
<td>17. Total t at Point 4 = __________</td>
</tr>
<tr>
<td>18. Measure the loop ⇒ ________</td>
</tr>
</tbody>
</table>

Figure 47(a) Measurement protocol for determination of $T_{NA}$.

5.5.3 Reversal of the Magnetic State in the $AF$

This procedure allows reordering the magnetic state of the $AF$ and reversing its initial magnetic state. The first steps are to align and saddle point the sample. To obtain reproducible data the state in the $AF$ is reset by sample dependent conditions found in
previous experiments. The temperature is then lowered to $T_{NA}$. At this point the field is reversed to saturate the ferromagnet in the opposite direction. The next step is to increase the temperature to $T_{Act}$, the temperature of thermal activation. Negative saturation at $T_{Act}$ is maintained for a fixed amount of time. Then the temperature is reduced to $T_{NA}$. Immediately after this, the field is cycled from negative saturation, $-H_{sat}$ to positive saturation, measuring the hysteresis loop. The procedure can be repeated for differing values of $T_{Act}$ but measurements are always made at $T_{NA}$ to avoid changes in the $AF$ state during the field sweep. A copy of the measurement protocol for this experiment is shown in figure 48.

Reversal $AF$ Magnetic State

1. Saddle point and alignment section
2. $T_{NA} =$
3. $(P, l, D)$ (100°C) =
4. Positive Saturating Field
5. $AF$ Resetting Section
6. Cool to $T_{NA}$
7. Negative Saturating Field
8. $H_{sat} =$
9. ⇒ Go to point 4 $t_l =$
10. $(P, l, D)$ $(T_{Act}) =$
11. Time $Act$ at Point 4 = min
12. Heat to $T = T_{Act} =$
13. $t_l =$
14. $t_f =$
15. Total time at $T_{Act}$ ⇒ $t =$
16. Cool to $T_{NA}$
17. Time to reach $T_{NA}$ ⇒ $t =$
18. Time needed to stabilise $T_{NA}$ $t_s$
19. $(T_{NA}) =$
20. Total $t$ at Point 4 =
21. Measure the loop ⇒

Figure 48(a) Measurement protocol for studying magnetic order reversal in the $AF$.

5.5.4 Angular Dependence of the Exchange Field and Coercivity

The first steps are alignment and saddle point the sample with the unidirectional anisotropy axis parallel to the direction of the applied field. After this, a positive field is applied and temperature is raised to a certain value for a fixed amount of time. The field was then set to zero after cooling and the angular position of the sample set to the angle at which $H_{ex}$ and $H_C$ are to be measured. A hysteresis loop is measured starting at positive fields. Comments about this process and how it could be improved are given in
Chapter 5: Measurement Techniques and Protocols

sections 6.4 and 7.2. The measurement protocol for this experiment is shown in figure 49.

Angular Dependence of $H_a$ and $H_C$

1. Saddle point and alignment section
2. Align sample $\Rightarrow \Theta_0$
3. Angle Reading in Vibrating Head for this alignment $\Theta_o = \square$
4. Temperature of measurement $T_m = \square$
5. Temperature of No thermal Activation $T_{NA} = \square$
6. Positive Saturating Field
7. AF Resetting Section
8. Measurement protocol for studying the angular dependence of $H_a$ and $H_C$.

5.5.5 Study of Training Effects

The procedure starts with the alignment and saddle-point of the sample with the unidirectional anisotropy axis parallel to the direction of the applied field. The AF is reset as before. Immediately after this the temperature is set to the value at which the measurement is going to be made. At this temperature, the field is cycled over a number of loops, subsequently, the measurements of the hysteresis loops are done. The measurement protocol for this experiment is shown in figure 50.

Training Effects

1. Saddle point and alignment section
2. $T_{NA} = \square$
3. Positive Saturating Field
4. AF Resetting Section
5. Select Field Values for Measurement
6. Cool to $T_m = \square$
7. Measure the loop $\Rightarrow$

Figure 49(a) Measurement protocol for studying the angular dependence of $H_a$ and $H_C$.

Figure 50(a) Measurement protocol for the study of training effects.
Chapter 5: Measurement Techniques and Protocols

5.5.6 Measurement Protocol Files

Experiment /File: AF Reset /

Name: Luis E Fernández Outón

Date: __________________________

Sample: __________________________

Saddle point □
Align sample □

Temperature of measurement

\( T_m = \) __________________________

\((P, I, D) (T_{reset}) = \) __________________________

Positive Saturating Field

\( H_{sat} = \) __________________________

⇒ Go to point 1 \( t_1 = \) __________________________

Heat to \( T_{reset} = \) __________________________

Time needed to reach \( T_{reset} \)

\( t_1 = \) __________________________

\( t_f = \) __________________________

\( t(T_{reset}) = \) __________________________

\((P, I, D) (T = T_m) = \) __________________________

Cool to \( T = T_m \)

Time to reach \( T_m \)

\( t = \) __________________________

Time spent in point 1:

\( t = \) __________________________

Measure the loop ⇒

\( H_{sat} = \) __________________________ ⇒ \( H_{sat} = \) __________________________

Step = - __________________________

\( H_{sat} = \) __________________________ ⇒ \( H_{sat} = \) __________________________

Step = + __________________________

SET SAMPLE DRIVE ON
Start Measurement
Go to Zero Field
CHECK RESULTS

Figure 46 AF reset measurement protocol.
Figure 47 Measurement protocol for determination of $T_{NA}$. 

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Chapter 5: Measurement Techniques and Protocols

Experiment / File: Reversal AF magnetic state

Name: Luis E Fernández Outón

Date: __________________________

Sample: __________________________

Saddle point __________________________ □
Align sample __________________________ □

Temperature of No thermal Activation

\( T_{NA} = \) __________________________ □
\( (P, I, D)(100^\circ C) = \) __________________________ □

Positive Saturating Field

\( H_{s} = \) __________________________ □

⇒ Go to point 1 \( t_S = \) __________________________ □

Heat to 100°C \( t_S = \) __________________________ □
Time stabilise 100°C \( t_S(100^\circ C) = \) __________________________ □

Resetting Time at 100°C \( t_S = \) min □

Select Field Values for Measurement □

Total Time at (T=100°C)

\( t_S + \) Reset time = __________________________ □

\( (P, I, D)(T_{NA}) = \) __________________________ □
Cool to \( T_{NA} \) __________________________ □
Time to reach \( T_{NA} \Rightarrow t = \) __________________________ □

Time needed to stabilise \( T_{NA} \)

\( t_S(T_{NA}) = \) __________________________ □

Time spent in point 1:

\( t = \) __________________________ □

Negative Saturating Field

\( H_{-s} = \) __________________________ □

⇒ Go to point 4 \( t_F = \) __________________________ □

\( (P, I, D)(T_{NA}) = \) __________________________ □

Time \( T_{Adv} \) at Point 4 = min □

Heat to \( T = T_{Adv} = \) __________________________ □

\[ H_{+, w} = \]

\[ \rightarrow \text{ Go to point } 1 \Rightarrow t_S = \]

\[ \text{Heat to } 100^\circ C \Rightarrow t_S = \]

\[ \text{Time to stabilise } 100^\circ C \]

\[ t_S(100^\circ C) = \]

\[ \text{Resetting Time at } 100^\circ C = \text{min} \]

\[ \text{Select Field Values for Measurement} \]

\[ \text{Total Time at (T=100°C)} \]

\[ t_S + \text{Reset time} = \]

\[ (P, I, D)(T_{NA}) = \]

\[ \text{Cool to } T_{NA} \]

\[ \text{Time to reach } T_{NA} \Rightarrow t = \]

\[ \text{Time needed to stabilise } T_{NA} \]

\[ t_S(T_{NA}) = \]

\[ \text{Time spent in point 1:} \]

\[ t = \]

\[ \text{Negative Saturating Field} \]

\[ H_{-s} = \]

\[ \rightarrow \text{Go to point } 4 \]

\[ (P, I, D)(T_{NA}) = \]

\[ \text{Time } T_{Adv} \text{ at Point } 4 = \text{min} \]

\[ \text{Heat to } T = T_{Adv} = \]

\[ \text{MEMO} \]

\[ \text{Figure 48 Measurement protocol for studying magnetic order reversal in the AF.} \]
Experiment / File: Angular dependence of $H_a$ and $H_c$

Name: Luis E Fernández Outón
Date:
Sample:

Saddle point
Align sample $\Rightarrow \Theta_0$
Angle Reading in Vibrating Head for this alignment $\Theta_0 =$

Temperature of measurement $T_m =$

Temperature of No thermal Activation $T_{N\!A} =$

$(P, I, D) (T_{read}) =$

Positive Saturating Field
$H_{+s\!a\!l} =$
$\Rightarrow$ Go to point 1 $\Rightarrow t_1 =$

Heat to $T_{read} \Rightarrow t_1 =$
Time stabilise $T_{read}$
$t_s(T_{read}) =$
Resetting Time at $T_{read}$ min

Select Field Values for Measurement

Total Time at $(T_{read})$
$t_s +$ Reset time =

$(P, I, D) (T_{NA}) =$

Cool to $T_{NA}$
Time to reach $T_{NA} \Rightarrow t =$

Time needed to stabilise $T_{NA}$
$t_s (T_{NA}) =$

$Go to Zero Field \Rightarrow t =$
Time spent in point 1 $\Rightarrow t =$

Align Angle of Measurement:
Angle Increment from $\Theta_s \Rightarrow \Delta \Theta =$
Angle of Measurement $\Theta =$

Saddle point

Measure the loop $\Rightarrow$

$H_{-s\!a\!l} =$ $\Rightarrow H_{+s\!a\!l} =$
Step $=$ $-$

$H_{-s\!a\!l} =$ $\Rightarrow H_{+s\!a\!l} =$
Step $=$ $+$

SET SAMPLE DRIVE ON
Start Measurement
Go to Zero Field
CHECK RESULTS

MEMO

Figure 49 Measurement protocol for studying the angular dependence of $H_a$ and $H_c$. 
Chapter 5: Measurement Techniques and Protocols

Experiment /File: Training Effects /

Name: Luis E Fernandez Outón

Date:
Sample:

Saddle point
Align sample

Temperature of No thermal Activation

\[ T_{NA} = \]  
\[ (P, I, D) (100°C) = \]

Positive Saturating Field

\[ H_{sat} = \]  
\[ \Rightarrow \text{Go to point 1} \Rightarrow t_1 = \]

Heat to 100°C\[ t_1 = \]
Time stabilise 100°C
\[ t_s(100°C) = \]

Resetting Time at 100°C \[ \text{min} \]

Select Field Values for Measurement

Total Time at (T=100°C)
\[ t_5 + \text{Reset time} = \]

\[ (P, I, D) (T_m) = \]

Cool to \[ T_m \]
Time to reach \[ T_m \Rightarrow t = \]

Time needed to stabilise \[ T_m \]
\[ t_s (T_m) = \]

Time spent in point 1:
\[ t = \]

Measure the loop \[ \Rightarrow \]

\[ H_{sat} = \]  
\[ \Rightarrow \, H_{sat} = \]

Step =  

\[ H_{sat} = \]  
\[ \Rightarrow \, H_{sat} = \]

Step =  

Number of Cycles \[ n = \]

Figure 50 Measurement protocol for the study of training effects.
Chapter 6

Thermal effects on exchange biased bilayers

6.1 Introduction

Much of the published literature related to the phenomenon of exchange bias is not specific in terms of the exact experimental conditions used to obtain the measurements. In most of the cases the magnetic history prior to measurement of the exchange bias system is not described. Furthermore, it is often not appreciated that the state of the AF material can change either by maintaining the material with the F layer saturated in the opposite direction to that in which it was originally set, or due to unseen thermal processes occurring in the AF. Changes in the AF may occur during what appears to be a normal measurement of the properties of the F. These facts are the main motivation to develop the measurement protocols described in section 5.5.

Following these protocols measurements have been made on IrMn/CoFe exchange biased systems where we have ensured that the measurements are reproducible. This means that the history of the sample under study must be known prior to any measurement. Also any change in its magnetic state that might have occurred during a previous measurement needs to be erased in order to obtain a reproducible initial state.

6.2 Thermal activation in IrMn exchange biased systems

It has been shown previously by experiment that $H_{ex}$ derives from the amount of AF material that exists in a state such that its orientation remains unaltered when the ferromagnet is reversed (O'Grady et al., 2002). It has been shown experimentally that the bulk magnetisation of the ferromagnetic layer drives these changes in $H_{ex}$ (Zhou et al., 2001). Gökemeijer et al., (1999a) studied $H_{ex}$ in CoO/NiFe and FeMn/NiFe thin films by field cooling. By lowering the temperature from above $T_N$ (292K for CoO, 690K for bulk IrMn) to a lower temperature so that order in the AF is established, it was found that the ordered state in the AF was dependent of the state in which the system
was cooled. In a similar way, Miltényi et al. (1999) showed that it is possible to set $H_{ext}$
to different values depending on the remanent state of the $F$ during field cooling.

Due to the lack of magnetic moment in the $AF$ material, the observation of thermal
activation effects in the $AF$ layer is impossible due to the difficulty of detecting
variations in the order of the $AF$ material. The variation in the order in the $AF$ has been
studied through the effect its order has on the $F$.

Both $AF$ and $F$ layers in the exchange biased system are composed of grains, which are
distributed in size according to a lognormal distribution as stated in section 5.2.1. Due
to their polycrystalline nature there will be a distribution of anisotropy easy axes in the
whole ensemble of grains that form each layer. The dispersion in the anisotropy easy
axis of the $AF$ is dependent of the deposition process used. See sections 5.2.3 and 5.2.4.
Also, effects such as domain wall pinning, which may arise from crystalline
imperfections or grain boundaries, may give rise to distributed energy barriers due to
reversal of the magnetic state of each $AF$ grain. Therefore, the character of the
distribution of energy barriers to reversal in the $AF$ will be a crucial influence on the
behaviour of the state of the $F$.

![Figure 51 Effect of thermal activation on IrMn(5nm)/CoFe(10nm) measured at 100 K.](image-url)
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Figure 51 shows the effects of thermal activation on the AF grains under the influence of a reversed applied field for 30 minutes at two different temperatures. Increased thermal activation results in a shift of the hysteresis loop towards positive field values. The measurements shown were taken at the same temperature, 100K. At this temperature it has been determined that no further thermal activation occurred during measurement or at least if there is any, it is not significant, even if a reversed field is applied for longer times. (see section 6.2.2).

The reversal of F particles and grains by thermal activation has been widely studied as described in section 2.9. When a negative field is applied to an exchange biased system, the reversal of the F will be determined by the exchange interaction with the underlying AF, which is exerted through the interface of the materials. In a microscopic picture, the reversal of a F grain will depend on the strength of the exchange coupling to the neighbouring AF grains as much as on the properties of the F itself. The AF grains exert an exchange field on the neighbouring F grains that prevents them from reversing their state of magnetisation when a negative field is applied. Some of the F grains will change their spin orientation and reverse, therefore, the exchange interaction between F and AF will be modified. The F grains that are reversed exert an exchange field on the AF grains that promotes the reversal in the AF orientation of axis along which order is established. It should be noted that this effect occurs below $T_N$ for the AF, and the AF order is maintained with spins paired in opposite directions. However the spins at the interface reverse orientation to maintain a “near” parallel alignment with a resulting reversal of the direction of all the spins in the AF grain to maintain the antiparallel state.

As described in section 2.9, the energy barriers to reversal, $\Delta E$, are distributed within the AF. When an energy barrier, $\Delta E$, is overcome the state of the AF grain will flip to a reversed state. Figure 52(a) shows a schematic of the energy barriers to reversal for the original AF state after field cooling. Figure 52(b) shows the distribution of energy barriers to reversal after thermal activation of AF grains with low energy barriers to reversal. Those AF grains with higher energy barriers remain in the original state. The amount of AF grains that remain in their original spin state determines $H_{ex}$. See figure 51. In section 6.2.3 it is shown how $H_{ex}$ varies as the amount of AF material that remains unaltered is tailored by thermal activation.
6.2.1 Reset of the order in the antiferromagnetic layer

In order to obtain reproducible measurements, it is necessary to reset the magnetic state of the antiferromagnet. When the reset of the AF is achieved, hysteresis loops measured under the same circumstances would result in their superposition if the magnetic state of the sample measured were the same in both cases prior to measurement. O’Grady et al. (2002) developed a procedure to reset the AF by which it is possible to reproducibly and reliably obtain the same magnetic state by taking advantage of thermal activation to “delete” any change which occurred in the AF from the original state due to thermal activation over the energy barriers to reversal. They investigated the reversal of the AF in two spin-valves formed by FeMn/NiFeCo/Cu/NiFeCo. To reset any previous thermal activation of the AF grains or AF domains, the sample was held at 373 K in a field of +300 Oe for 45 min. These conditions derive from the experimental fact that it is possible to completely reverse the state of the AF within a 15 minute period. Hence, maintaining the sample at positive saturating field for three times that period ensures that the state of the AF is fully reordered.
Chapter 6: Thermal Effects on Exchange Biased Systems

Figure 53 Schematic of distribution of energy barriers to reversal showing two energy barriers. Grains with $\Delta E < \Delta E_{c1}$ reversed their state due to thermal activation at point 4 of the hysteresis loop. Application of resetting conditions, at point 1, activates those energy barriers and reorders the state of those grains.

For the materials involved in our studies, i.e. IrMn(3nm)/CoFe(10nm) and IrMn(5nm)/CoFe(10nm), the influence of a positive applied field which is aligned parallel to the unidirectional anisotropy and heating to a temperature of 373K for a period of 90 min was established by experiment to result in maximum shift of the hysteresis loop once the sample was cooled to a thermal-activation-free temperature, $T_{NA}$. At this temperature thermal activation is minimised, if not "completely" eliminated. The experimental method by which this temperature is obtained is described in section 6.2.2.

Figure 54 shows the reproducibility of order for IrMn(3nm)/CoFe(10nm) for different conditions. The hysteresis loops shown were measured at 77K. Heating at 373K for 90min at point 1, typically 2000Oe, (figure 53) ensures that the state of the AF is reproducible. The values of the coercive points $H_{c1}$ and $H_{c2}$ obtained for each set of resetting conditions are shown in table 4. The AF is fully reordered when heated at 383K for 22.5min when previously measured at 77K. For measurement times of 30 minutes at temperatures up to 373K, the resetting conditions obtained for both IrMn(3nm)/CoFe(10nm) and IrMn(5nm)/CoFe(10nm) are 373K for 90 min at point 1. Measurements shown in this figure were obtained by following the measurement protocol.
Figure 54 Reproducibility of order in the $AF$ for IrMn(3nm)CoFe(10nm)

Table 4 Values obtained after heating at 373K and 383K for different resetting times at point 1.

These same conditions allow erasure of any thermal activation in the $AF$ during measurement. This is justified by the fact that the time spent to reset the $AF$ at point 1 is more than 3 times larger than the typical measurement time, which can range from 10 to 30 minutes (See section 2.9).
6.2.2 Determination of thermal-free-activation temperature, $T_{NA}$.

In order to obtain the intrinsic behaviour of the exchange biased material it was necessary to establish a temperature at which the hysteresis loop could be measured without thermal activation taking place during the time of measurement. This was achieved by resetting the AF following the criteria indicated before, i.e. heating at 373K for 90 minutes, and subsequently cooling the sample to progressively lower temperatures, where holding the sample at reverse bias, i.e., at point 4 of the hysteresis loop in figure 53 for a fixed time did not result in any change in the subsequently measured hysteresis loop. The measurement procedure followed was described in section 5.5.2, figure 47. Figure 47(b) shows an schematic diagram of this measurement protocol. From these experiments we determined that a reasonable value for $T_{NA}$ was 100 K for IrMn(5nm)/CoFe(10nm) and 77 K for IrMn(3nm)/CoFe(10nm).

Above $T_{NA}$, thermal activation produces changes in the AF that increase with time according to a series expansion of $ln(t)$ of an order that depends on the characteristics of $f(\Delta E)$ as described in section 2.9. Hence, the effect of thermal activation after 1 minute at point 4 and after 15 minutes at that point is a difference in the position of $H_{c1}$ and $H_{c2}$ and therefore $H_{ex}$. It is important to note that these loops are measured after 1 and 15 minutes spent at negative saturation (point 4), and measured starting at that point. The magnitude of the shift will be larger for those samples which are more thermally active, i.e. those that suffer more changes in the AF order. At $T_{NA}$ and below, the superposition

![Figure 47(b) Schematic of describing the measurement protocol for the determination of $T_{NA}$ shown in fig. 47, p.105.](image)
of the hysteresis loops obtained after 1 and 15 minutes is expected with no shift in the position of $H_{c1}$ and $H_{c2}$ and therefore the same $H_{ex}$.

Figure 55 shows the effect of thermal activation at point 4 (see figure 53) for 15 minutes at different temperatures of activation for the sample with IrMn layer thickness of 5nm. Note that in the figure only the demagnetising branch of each loop corresponding to $H_{c2}$ is shown. Note that due to thermal activation of the AF grains the shift is higher for higher temperatures.

![Figure 55](image)

Figure 55 Recoil loops after exposure to reverse bias fields for various times at different temperatures for IrMn(5nm)/CoFe(10nm).

Figure 56 shows the shift that $H_{c2}$ experiences when the IrMn(5nm)/CoFe(10nm) sample is held at reverse bias for 15 and 30 minutes at 100K after correction of the paramagnetic components.

The results obtained for IrMn(3nm)/CoFe(10nm) are shown in figure 57. Two measurements of hysteresis loops are shown. One hysteresis loop corresponds to the case of 1 minute at point 4, the other to 15 minutes. Previous to either measurement the state of the AF was reset, and then quenched to the temperature of measurement 77K.
Figure 56 Expanded view of hysteresis loops from fig.55 after thermal activation in reverse bias for 15 and 30 minutes for IrMn(5nm)/CoFe(10nm).

The value of $H_{c1}$ is the same in both cases as a consequence of the fact that at these temperatures the thermal activation of the AF grains is a minimum. There is a small difference in the value of $H_{c2}$, which is less than 5 Oe. It is not within error although this difference is small enough to be insignificant in terms of thermal activation of the AF grains. This shift in the value of $H_{c2}$ for this particular measurement will be discussed in section 6.3.

Figure 57 Hysteresis loops measured at the $T_{M} = 77$ K for IrMn(3nm)/CoFe(10nm) after 1 and 15 minutes in reverse bias (point 4).
It is very important to stress that these hysteresis loops, related to the determination of $T_{NA}$, are measured starting from negative saturation, after resetting the $AF$ in positive saturation, i.e. forward bias, as indicated in the measurement protocol of figure 47. Therefore, the first reversal of the $F$ magnetisation is not included in the measurement of the hysteresis loops shown, e.g. measurements shown in figure 57. This allows interpretation of the cause of the shift of the value of $H_{c1}$ as a result of thermal activation in the $AF$ and removes the shift in $H_{c1}$ due to spin reorientation. The origins of the shift of the value of $H_{c1}$ and $H_{c2}$ related to training effects and its dependence on temperature will be discussed in section 6.3.

### 6.2.3 Dependence on thermal activation of $H_{ex}$ and $H_{C}$

Once that the thermal-activation-free temperatures have been determined for each of the samples the maximum shift in the loop, $H_{ex}$ and $H_{C}$ can be obtained with certainty that the results obtained are reproducible. Following the measurement protocol described in figure 48 (section 5.5.3) and sketched in figure 48(b) the dependence of both $H_{ex}$ and $H_{C}$ on the degree of order of the $AF$ can be studied. The results obtained are shown in figures 58 and 59.

![Figure 48(b) Schematic describing the measurement protocol for the determination of $T_{NA}$ shown in fig. 48, p.106.](image)

All measurements shown are made at the thermal activation free temperature $T_{NA}$, therefore no thermal activation occurs during the measurement. Hence, it is plausible to say that no change in the state of the $AF$ occurs due to thermal activation during the time of measurement.
The measurement of each hysteresis loop starts at point 4 (see hysteresis loop in figure 53). This way the variation of $H_{ex}$ and $H_C$ with the temperature of thermal activation,
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\( T_{\text{Act}} \) can be interpreted exclusively in terms of thermal activation during the time spent at point 4.

The heating stage in reverse bias is used to progressively change the order in the AF from the original state to the reverse orientation. The data displayed in figures 58 and 59 show the variation of \( H_C \) and \( H_{\text{ex}} \), with the temperature of thermal activation, \( T_{\text{Act}} \), at which the AF was heated at point 4 for IrMn(5nm)/CoFe(10nm) and IrMn(3nm)/CoFe(10nm). As \( T_{\text{Act}} \) is increased more AF material becomes thermally active. AF grains with higher energy barrier to reversal, \( \Delta E \), reverse their order under the influence of the exchange interaction that the negative saturated F exert on them. In consequence, the amount of AF that biases the F is reduced and the hysteresis loop shifts towards zero.

The exchange field, \( H_{\text{ex}} \), equals zero when the amount of AF that remains with the original state is equivalent to the amount of AF material whose state was reversed by thermal activation. This would be the case of having reversed half the area under the curve of \( f(\Delta E) \) due to thermal activation. The temperature of activation, \( T_{\text{Act}} \), at which this situation is achieved is the median of the blocking temperatures, \( T_B \), of the distribution of blocking temperatures \( f(T_B) \) within the AF.

![Hysteresis Loops](image)

Figure 60 Part of the sample set of hysteresis loops obtained after thermal activation at different temperatures for IrMn(5nm)/CoFe(10nm) and IrMn(3nm)/CoFe(10nm).

In the case of a symmetrical distribution of energy barriers such as the one shown in figure 53, the position of \( T_B \) would be that at which \( f(T_B) \) is maximum. This would not
be the case for asymmetrical distributions of energy barriers. The values obtained for the mean blocking temperature are the following:

<table>
<thead>
<tr>
<th>IrMn($t_{AF}$)/CoFe(10nm), $t_{AF}$ (nm)</th>
<th>Mean blocking temperature, $\overline{T_b}$ (K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>310</td>
</tr>
<tr>
<td>3</td>
<td>200</td>
</tr>
</tbody>
</table>

Table 5 Mean blocking temperatures obtained for the samples measured.

From the shift of the hysteresis loop of the $F$, information about the state of order of the $AF$ is obtained. Conversely, the position of the hysteresis loop of the $F$, i.e., $H_{ex}$ can be considered as a measurement of degree of order of the $AF$ layer, and ultimately of the magnetisation within the $AF$.

### 6.2.4 Measurement of the distribution of energy barriers to reversal in IrMn based exchange bias systems

For an experiment such as the one described in previous section, in which the $AF$ is exposed to a reversed exchange field for a fixed and constant amount of time, the variation of $H_{ex}$ with the temperature of activation, $T_{Acn}$ allows the mapping of the energy barrier distribution to reversal, $f(\Delta E)$. This procedure ensures as well that the measurement is not affected by thermal activation during measurement. The resetting conditions of the $AF$ previously established ensure that the $AF$ state is that for which the $H_{ex}$ is maximum. The variation of $H_{ex}$ with $T_{Acn}$ from the original state to that in which the magnetisation state of the $AF$ is completely reversed is a sigmoidal-type curve.

Inspection of figure 58 shows that it was not possible to completely reverse the loop shift in the sample with a 5nm thick IrMn due to experimental temperature restrictions. The highest temperature that could be achieved in the equipment used was 400K. Furthermore, higher temperature would jeopardise the sample's condition, giving rise to possible damage of the interface between the $F$ and $AF$. Hence a complete distribution of the energy barriers within the material could not be obtained. Figure 59 shows that
the order in the \( AF \) was completely reversed, as denoted by the saturation of \( H_{ex} \) at 400K. Temperatures lower than 100K would be required to show the saturation of \( H_{ex} \). Equipment restrictions did not allow temperatures below 100K by the time these measurements were made. In the case of achieving saturation at low temperatures, the absolute value of the exchange field would be the same as that obtained at high temperatures. Both situations would correspond to the cases of having the \( AF \) state 100% with the original forward orientation or 100% with reversed orientation, respectively. These results demonstrate that the shift in the hysteresis loop derives exclusively from the degree of order in the \( AF \), as it has been previously reported (O’Grady et al. 2002). As the temperature of thermal activation approaches to \( T_N \) a decay of \( H_{ex} \) to zero would be expected.

Differentiation of the shift in the hysteresis loop, \( H_{ex} \), with respect to the temperature of thermal activation, \( T_{act} \), gives a measurement of the energy barrier distribution to reversal in the \( AF \), \( f(\Delta E) \). The exchange field measured in the experiment described in section 6.2.3 would be related to \( f(\Delta E) \) by an expression of the type of eq.6.1, assuming that all the \( AF \) in the distribution contribute equally to the exchange bias and that this contribution can be represented by an average field \( H_0 \).

\[
H_{ex} = H_0 \left[ \int_0^{\Delta E_c} f(\Delta E)d(\Delta E) - \int_{\Delta E_c}^{\infty} f(\Delta E)d(\Delta E) \right]
\]

Figures 61 and 62 show, respectively, the results obtained from these measurements for the distribution of energy barriers to reversal within the \( AF \) layer of the IrMn(5nm)/CoFe(10nm) and IrMn(3nm)/CoFe(10nm) samples studied. As it was not possible to reverse completely the order in the IrMn(5nm) sample, it was not possible to map completely \( f(\Delta E) \) for this sample. The distribution of energy barriers for IrMn(5nm)/CoFe(10nm) was differentiated assuming the distribution to be symmetrical. Both distributions in figures 61 and 62 are normalised to unity.

From figures 61 and 62 it is clear that a distribution of energy barriers exists within the IrMn with significant barriers extending to low values. This is similar to the results obtained for FeMn based samples studied by O’Grady et al. (2002).
For applications the amount of AF that remains thermally stable at room temperature and at those temperatures at which devices based on the exchange bias phenomenon work, which can range from 70°C to 100°C is critical. From figures 61 and 62, it is possible to estimate that percentage. For IrMn(5nm)/CoFe(10nm) at 300K the amount of AF that is pinning the F is 53% of the total whilst for IrMn(3nm)/CoFe(10nm) it is only 16%. These percentage values are estimated as the percentage of the area under the curve of the distribution of energy barriers, $f(\Delta E)$, between the temperature of interest, i.e., 300K, and infinity. The area under the curve between $T_{A1}$ and the temperature of interest correspond to superparamagnetic spins with lower blocking temperature than those of interest. In other words, the percentage of the AF that remains stable at a given temperature $T$ is represented by the second integral in eq.6.1, where the lower limit corresponds to the highest value of the energy barriers that can be activated at that temperature. The first integral in eq.6.1 corresponds to the amount of AF that is unstable at $T$. An ideal AF material would be that which is 100% thermally stable at the working temperature.
6.2.5 Coercivity and temperature of thermal activation.

The behaviour of $H_C$ with the temperature of activation, $T_{Ael}$, is also shown in figures 58 and 59. A peak in $H_C$ is observed in both graphs even though the temperature at which $H_C$ was measured was the same for all measurements. Hence, it is expected that some relation would exist between $H_C$ and the degree of order in the AF. The peak does not occur at the mean of the distribution of blocking temperatures in the AF, for any of the samples. For the sample with IrMn 5nm thick, the peak occurs about 60K below $T_B$, whereas for the IrMn(3nm) it is 50K above. Consequently, there does not appear to be any correlation whatsoever between the mean of the distribution and the peak in the coercivity.

The enhancement of $H_C$ for the exchange biased system has been qualitatively claimed to appear due to an increase in the energy required to reverse the $M_F$ due to the exchange interaction between the $F$ and the $AF$ (e.g. Nogues and Schuller, 1999). The coercivity has been found to peak at temperatures close to the highest value of the blocking temperatures in the $AF$ when the system is measured in a similar manner than that used for the measurement of training effects (see figure 50), (Nogues and Schuller,
Pan et al., (2004), showed that the position of the peak with the temperature of activation relative to $T_B$ varied depending on the ratio of Fe to Mn contained in their specimen Fe/Fe$_{85}$Mn$_{15}$/Cu(001). This was explained in the same terms as the enhancement of $H_C$, due to an increasing amount of AF material that is being dragged by the F as the temperature of measurement increases towards the maximum blocking temperature within the $f(T_B)$. However, the protocol followed in the measurements described in section 6.2.3, ensures that the amount of AF that the F drags during measurement of a hysteresis loop is kept fixed to a minimum amount for all measurements. The amount of AF material, expected to be formed by superparamagnetic grains, can be considered negligible compared to the AF material that remains unaltered during measurement. Hence, the origin of the peak in $H_C$ remains somewhat obscure and it is hard to imagine a mechanism whereby the amount AF material ordered in two different senses could lead to such a peak or to the position of such a peak relative to the mean of the blocking temperature distribution.

### 6.2.6 Grain size and AF thickness dependence of exchange bias features

Two samples that differ in the AF thickness have been measured, IrMn(3nm)/CoFe(10nm) and IrMn(5nm)/(10nm). Although it could not be measured, the grain size distribution within the IrMn layer is expected to increase with increasing thickness of IrMn (Fuke et al., 1999). Therefore, the sample with IrMn(5nm) is expected to have larger grain size than the sample containing IrMn(3nm). This applies to DC magnetron sputtering; however, there are other sputtering techniques that allow control of the grain size independently of the layer thickness, such as controlled plasma sputtering (Vopsaroiu et al., 2004, 2005).

Inspection of figures 58 and 59 shows that the maximum value of the exchange field is larger for the sample with thinner IrMn, as it predicted from the theoretical models described in chapter 4. All models agreed that $H_{ex}$ varies inversely with the AF thickness [e.g. Meiklejohn and Bean (1956), Mauri et al. (1987), Malozemoff (1987), Stamps (2000)]. The variation of the exchange field with the temperature of thermal activation is more pronounced for the sample with smaller crystal grain size, i.e.
IrMn(3nm). Smaller grains are easier to activate as their volume contributes to smaller anisotropy energy. Hence, the effect of temperature on thermal activation is larger for IrMn(3nm) than for IrMn(5nm). As a result of this, figures 58 and 59 show that it was possible to reverse the order in the AF “almost completely” in the sample with thinner IrMn, therefore, smaller grain size. Also, the value of the mean blocking temperature is lower for IrMn(3nm) due to smaller grain size. The values of $H_C$ are expected to vary $1/t_{AF}$ where $\lambda$ is a number related to the spin structure and the material and is generally close to 1. Typical values are 0.3, 1 (Sang et al., 1999). The results obtained show that the larger values of $H_C$ correspond to the sample with lower AF thickness. Figures 61 and 62 show that the distribution of energy barriers to reversal is considerably wider for IrMn(5nm) than for IrMn(3nm) which might be due to a wider distribution of the grain size or the anisotropy.

The results obtained support a grain size dependent model of energy barriers. The exchange field originated from domain wall formation at the interface would be independent of the AF thickness as long as the AF is thick enough to support a domain wall. The way that $H_{ex}$ is reduced with thermal activation is consistent with a grain size model as shown in figures 61 and 62. Also results by Sadia Manzoor et al., (2005), show that there is a direct correlation between the grain size or potentially the grain volume and exchange bias. Moreover, it is hard to imagine any mechanism leading to formation of domains in polycrystalline AFs, as the RKKY interaction is quite improbable due to the very small magnetic moment in the grains.

6.2.7 On the controversy of measurements of blocking temperatures.

If the blocking temperature, $T_b$, is defined as the temperature at which the exchange field, $H_{ex}$ equals zero, the meaning of $T_b$ intrinsically depends on the description of the process by which the exchange field is made zero. The standard measurement method to determine the blocking temperature of an exchange biased system involves the determination of the hysteresis loop of the material as the temperature is raised until a point is reached at which the shift of the hysteresis loop becomes zero, i.e., the hysteresis becomes symmetric about the magnetisation axis.
Theoretical studies of blocking temperature, $T_B$, and the dependence of $H_{ex}$ on temperature are described in chapter 4 for the standard measurement method, similar to the measurement of training effects shown in figure 50 (pag. 109) for the measurement of one hysteresis loop. In this case the interpretation of $T_B$ is generally conceived as the temperature beyond which the thermal energy is such that it is able to detach the $AF$ spins from the anisotropy axes of the $AF$ grains by overcoming the magnetic anisotropy energy. At such temperature $H_{ex}$ is zero, as the spins of the $AF$ grains are all dragged by the $F$ when this is cycled. According to Fulcomer and Charap's interpretation (1972), $T_B$ will correspond to the blocking temperature of the $AF$ grain with the largest anisotropy energy. Above the blocking temperature the magnetisation of the uncompensated $AF$ grain will follow the magnetisation of the $F$, i.e. will become superparamagnetic. Below $T_B$ that grain will be "blocked", i.e. will not undergo magnetisation reversal with the reversal of the $F$ magnetisation.

Figures 58 and 59 show that heating to temperatures higher than the mean blocking temperature leads to the hysteresis loop shifting in the opposite direction to that in which it was originally set. Hence, the standard measurement of the blocking temperature distribution is not a measurement of the energy barriers to reversal within the $AF$. Hysteresis loops measured at increasing temperatures, even after resetting of the $AF$, lead to a progressive increase of the amount of $AF$ that is being dragged by the $F$. Such spins do not contribute to $H_{ex}$, whereas those that are not dragged are subject to thermal activation during measurement. Hence, $H_{ex}$ shifts towards zero and does not become positive. This procedure is therefore similar to an AC field demagnetising process.

As explained (section 6.2.3 and 6.2.4), the exchange field is zero when half of the distribution of energy barriers is in its original state, and the other half is in the reversed state. This is achieved by thermal activation of the $AF$ grains while kept in reverse bias (fig. 48, pag.107). This was shown to be exclusively dependent on the temperature of activation, $T_{act}$, at which the thermal activation took place. Hence, the blocking temperature value obtained is the mean blocking temperature of the distribution of energy barriers in the $AF$. In the case of IrMn(5nm)/CoFe(10nm), $T_B$ is 310K and for IrMn(3nm)/CoFe(10nm) it is 200K.
In the experiment described in section 6.2.3, and figure 48, the time spent in the thermal activation stage at reverse bias is 30 minutes. The thermal activation of the AF will be ongoing at a logarithmic rate during this stage. The time at negative saturation selected is much larger than the relaxation time of the AF grains in these samples. After this time, the number of reversals of the AF grains will be a small fraction of the number of reversals that have occurred in the first minutes. When the time of thermal activation in reverse bias or the time of measurement are of the order of the relaxation time of the AF grains, the shift of the hysteresis loop and therefore $T_B$, will depend on these temporal parameters.

Figure 63 shows the variation of $H_{ex}$ obtained from measurements following the protocols in figures 48 and 50. Progressive reversal of the AF order following the protocol in figure 48 leads to $T_B$, whereas a measurement procedure following the protocol in figure 50 leads presumably to the highest $T_B$ within the blocking temperature distribution.

Figure 63 Variation of $H_{ex}$ from application of protocol of figure 48 and 50 for two different materials, (a) IrMn(3nm)/CoFe(10nm) (b) FeMn(10nm)/NiFe(10nm). Figure 62 (b) is courtesy of Sadia Manzoor (to be published).
6.3 Contributions to exchange bias on IrMn/CoFe bilayers. Training effects.

It is known that training effects cause the reduction in the exchange field and coercivity with the number of the hysteresis loops measured repeatedly on an exchange biased system (Nogués et al., 1999). The effects of training on IrMn/CoFe samples have been studied before by other authors (e.g. Wang et al., 2002). In the scope of getting to a deeper knowledge of training this section describes the study of the effect of training on IrMn(3nm)/CoFe(10nm) and IrMn(5nm)/CoFe(10nm) and the dependence of this effects on temperature. Therefore, training effects were measured in a range of temperatures from 4.2K up to 400K.

The measurement protocol followed was described in section 5.5.5. Figure 50 shows the measurement procedure followed which is sketched below in figure 50(b). In order to obtain measurement reproducibility and to get the same state in the spin configuration of both AF and F, all measurements were done after resetting of the AF. Also the F was kept in saturation while the temperature was changed to the value of measurement. When the temperature was stabilised, hysteresis loops were measured consecutively. The number, n, of hysteresis loops measured was set to a maximum of 3. After this, changes in the values of \( H_{ex} \) and \( H_C \) due to repeated measurements were all within measurement error. Thus, no further information would be obtained from a larger number of measurements. The value of \( n \) at which both \( H_{ex} \) and \( H_C \) stabilise depends on the materials used and their susceptibility to suffer changes due to thermal activation.

In order to obtain a better description of the changes observed in the hysteresis loops due to field cycling and variation of the temperature of measurement, the behaviour of the coercive points \( H_{cl} \) and \( H_{c2} \) have been studied. The following figures show a sample set of the hysteresis loops obtained.
Figure 50(b) Description of the protocol for the measurement of training effects in fig. 50, p.109.

Figure 64 Sample set of hysteresis loops obtained at different temperatures of measurement for IrMn(3nm)/CoFe(10nm).
6.3.1 Measurements

It can be seen in figures 66 (a) and 67(b) that the hysteresis loops shift towards zero with cycling. Part of this shift must derive from thermal activation processes as described in section 6.2.3. However, inspection of figures 64 and 65 show high asymmetry in the shape of the loops for the first cycle of measurements, i.e., the hysteresis loops corresponding to \( n = 1 \). This asymmetry disappears with field cycling and increasing temperature. The asymmetry is more pronounced for the branch of the loop corresponding to point 3 in the hysteresis loop shown in figure 58 and consequently has a larger effect on \( H_{c1} \) than on \( H_{c2} \). This asymmetry is discussed in section 6.3.3 where the loop shape is studied in depth. Also there is a reduction in the values of \( H_{ex} \) and \( H_C \) with field cycling and increased temperature of measurement (see figures 66 and 67). Given the fact that the coercive points \( H_{c1} \) and \( H_{c2} \) are affected...
differently by training, figures 68 and 69 show the temperature dependence of $H_{cl}$ and $H_{c2}$ with field cycling at a range of temperatures.

![Figure 66](image)

**Figure 66** Variation of $H_{m}$ (a) and $H_{c}$ (b) with the temperature of measurement and field cycling for IrMn(3nm)/CoFe(10nm).

![Figure 67](image)

**Figure 67** Variation of $H_{m}$ (a) and $H_{c}$ (b) with the temperature of measurement and field cycling for IrMn(5nm)/CoFe(10nm).

The evolution of $H_{cl}$ with field cycling shown in figures 66(a) and 66(b) can be more easily interpreted by inspection of figures 68 and 69. The different effect of field cycling on $H_{cl}$ and $H_{c2}$ is clear in these figures. While the value of $H_{c2}$ is almost independent of field cycling, $H_{cl}$ exhibits a pronounced shift between the first and second loops, and its value evolves with increasing number of cycles, $n$, towards a constant value. The number of loops required to saturate $H_{cl}$ depends on the degree of activation of the AF to spin reorientation (see section 6.3.3).
Figures 66(b) and 67(b) show the behaviour of $H_C$ with increasing temperature of measurement. Three different stages can be distinguished in $H_C(T)$ for IrMn(3nm) in figure 66(b). At temperatures below 25K $H_C$ increases abruptly due to a steeper variation on $H_{c1}$ at that temperatures whilst $H_{c2}$ increases monotonically. However, for IrMn(5nm) $H_{c2}$ has a constant value in that range of temperatures. Similar effects were observed by Takano et al. (1997) at temperatures below 75K for CoO on MgO substrates, who argued to be due to spin freezing effects. Such effects, discussed in section 6.3.4, disappear above 25K in the samples measured. Above this temperature $H_C$ increases up to a certain temperature. This is clearly observed in fig. 66(b). However in figure 67(b) there is a minimum around 100K which might be due to a sample repositioning that took place at that temperature. Any small angular error in the process of realignment of the sample may lead to significant variations in $H_C$ as described in section 6.4. Higher temperature measurements would be required to observe the peak in $H_C$ for the IrMn(5nm) sample.

![Figure 68 Variation of $H_{c1}$ and $H_{c2}$ with the temperature of measurement and field cycling for IrMn(3nm)/CoFe(10nm).](image)

The maximum in $H_C$ is not well understood, and its origin remains unclear (Nogués et al. (1999), Schulthess and Butler (1999), Leighton et al. (2000)). A possible explanation for the reduction of $H_C$ at high temperatures could be that thermal activation
at high temperatures of the AF spins reduces the stiffness in the AF grains and therefore the energy required to reverse the F would decrease due to reduction of the energy required by the AF spins to be dragged with the F. In the intermediate range of temperatures spin frustration due to spin reorientation processes could be a competing interaction that would give rise to the peak in $H_C$, see figure 66(b), giving rise to a constant difference between $H_C(n=1)$ and $H_C(n=2)$.

![Graph](image)

Figure 69 Variation of $H_{c1}$ and $H_{c2}$ with the temperature of measurement and field cycling for IrMn(5nm)/CoFe(10nm).

### 6.3.2 Thermal activation

In section 2.9 and 6.2 it has been shown that thermal activation processes depend on time according to a series expansion of $ln(t)$. In order to compare measurements done at different temperatures careful control has been taken to ensure, that equal periods of time are spent at positive and negative bias for all measurements. Therefore, the time spent with the F at positive and negative saturation is constant for all measurements within an error of 1 min. This was achieved by selecting correctly the maximum values of the applied field before a measurement was done. This means that in some cases the measurement had to be repeated to balance the times spent at saturation. This is in
contrast with most measurements where fixed values of the saturating field are applied. Given that $M_F$ generates the $H_{ex}$ seen by the AF, this measurement protocol is more appropriate when thermal activation of the AF is known to be occurring during measurement. From the data shown in section 6.2.2 it is clear that there is a thermal activation regime responsible for part of the training effect above $T_{NA}$, i.e., 77K for IrMn(3nm) and 100K for IrMn(5nm).

The effect of thermal activation on training is shown in figures 70 and 71 where the variation of the exchange field with the number of cycles is shown for different temperatures for both samples. The data presented in these figures has been normalised to the $H_{ex}(n=1)$. The degree of thermal activation increases with increasing temperature, which is manifest in the variation of $H_{ex}$ as a function of $n$. Both figures show that this variation increases with temperature and that it is significant at temperatures above $T_{NA}$.

Figure 70 Variation of $H_{ex}$ with the number of field cycles and the temperature of measurement for IrMn(3nm)/CoFe(10nm).
Figure 71 Variation of $H_{ex}$ with the number of field cycles and the temperature of measurement for IrMn(5nm)/CoFe(10nm).

However, inspection of figures 64 and 65 show that the shift occurred due to field cycling for $H_{c1}$ and $H_{c2}$ are not the same, being more pronounced for $H_{c1}$. Figures 68 and 69 show how $H_{c1}$ shifts with field cycling. The shift experienced by $H_{c1}$ from the first cycle ($n = 1$) to the second loop ($n = 2$) is much larger than the shift experienced between cycles of higher order. The shift experienced by $H_{c2}$ is very small, varying from 1 to 4 Oe, and does not change with cycling. The fact that the shift in $H_{c2}$ with cycling for a given temperature of measurement is almost negligible shows that spin reorientation is the major contribution to the variation of $H_{c1}$ and $H_{c2}$ during field cycling. The measurement procedure used ensured that equal time was spent at positive (point 1 in figure 53) and negative saturation (point 4 figure 53). Therefore thermal activation occurred while the $F$ is kept in negative bias cancels that which occurred while the $F$ is at positive bias. Hence the evolution of $H_{c1}$ and $H_{c2}$ with field cycling, at a given temperature is due to spin reorientation effects.
6.3.3 Spin reorientation

Recently, it has been suggested (Hoffmann, 2004) that the shift in $H_{c1}$ and the change in the shape of the hysteresis loop from $n=1$ to $n=2$ derives from a spin reorientation effect that occurs within the $AF$ due to the cubic anisotropy of metallic $AF$ alloys and the uniaxial anisotropy of the $F$ layer, which is generally a soft material grown in a magnetic field. This is the case of the samples studied in this thesis, whose structure has been described in sections 2.8 and 5.2.1. Figure 72 shows a schematic of the spin reorientation that would occur in such exchange biased systems when a saturating applied field is reversed after field cooling.

The initial biaxial spin configuration in the $AF$ changes to a uniaxial spin configuration which would correspond to a lower energy state (Hoffmann, 2004). This new state would set and remain in such a configuration for successive field cycles. This would explain the significant shift in $H_{c1}$ from $n=1$ to $n=2$, whereas $H_{c1}$ would not shift significantly for continuing cycling. According to Hoffmann’s model of spin reorientation, this would be the only origin of the training effects. This model was proposed for systems in their ground state, thus, it does not explain how temperature would affect the spin reorientation within the $AF$ due to the reversal of the $F$.

![Figure 72 Schematic of spin reorientation in the AF spins during the first reversal of the F (loop 1) and during second reversal of the F, showing the two AF spin sublattices as suggested by Hoffmann (2004).](image)

Following the protocol designed for these measurements (see figure 50) the temperature dependence of both $H_{c1}$ and $H_{c2}$ has been measured. The measurements show that $H_{ex}$ is determined by a combination of thermal activation and spin reorientation in the $AF$, which would affect $H_{c1}$, whereas $H_{c2}$ would depend on thermal activation within the $AF$.
alone and spin reorientation would be practically negligible. The maximum shift observed in $H_{c2}$ due to spin reorientation is less than 5 Oe (see figure 57).

Figures 68 and 69 show that spin reorientation is almost constant, from the shift in $H_{c1}$ and $H_{c2}$, with field cycling for a wide temperature range. However, for high temperatures, the shift in $H_{c1}$ is reduced until it disappears. At the same range of temperatures $H_{c2}$ ceases to be linear with the temperature of measurement. This is indicative of a temperature dependence of the spin reorientation which seems to occur over a distribution of energy barriers for that range of temperatures. This is clearly seen in figure 68 for IrMn(3nm)/CoFe(10nm). For IrMn(5nm)/CoFe(10nm), figure 69, the range of temperatures over which spin reorientation occurs over a distribution of energy barriers at higher temperatures. This implies a grain volume dependence of the effect.

Although Hoffmann (2004) proposed a mechanism for spin reorientation, his model is a ground state model and does not include a temperature dependence. At the time of writing a mechanism by which spin reorientation is temperature independent at certain temperatures and dependent at higher temperatures over a distribution of energy barriers is unknown. However, a possible mechanism that can be suggested is similar to Hoffmann’s, due to a single valued energy barrier for each AF grain that varies in temperature in the same manner as the one shown in figure 68 for $H_{c1}$ for the first $F$ reversal in the hysteresis loop and as $H_{c2}$ for the second $F$ reversal. Such an energy barrier would represent the stiffness of the coupling between the AF sublattices and would lead to a grain size dependent spin reorientation. At temperatures above $T_B$ and close to the maximum of the blocking temperature distribution the reduction of this stiffness with the temperature of measurement would lead to a reduction in the shift of $H_{c1}$ from $n=1$ (first loop) to $n=2$, leading to a metastable state of the spin configuration whose sublattice stiffness would evolve with temperature as shown for $H_{c1}(n>1)$. In fact the curve corresponding to $H_{c2}(n)(T)$ resembles $H_{c1}(n>1)(T)$, where $T$ is the temperature of measurement. The shape of the curve corresponding to $H_{ex}(n)(T)$ is a reflection of these two effects (see figures 66(a) and 67(a)).
The asymmetry of the hysteresis loop and its dependence on temperature has been studied to discern whether spin reorientation is temperature independent. Figure 73 and 74 shows the evolution of the shape of the hysteresis loop with the temperature of measurement for both samples studied. Dark coloured loops are the measured hysteresis loops. The light coloured loops correspond are the same loops inverted. This way it is possible to show graphically the differences in the symmetry of both branches for each hysteresis loop \((n = 1, 2, 3)\) for a range of temperatures. The graphs shown in figures 73 and 74 show how the hysteresis loops become more symmetric with increasing \(n\) and increasing temperature of measurement. For IrMn(3nm)/CoFe(10nm) the loop becomes symmetric at 275 K for \(n = 2\). For IrMn(5nm)/CoFe(10nm) the loop becomes symmetric at 40K for \(n = 2\). Therefore, if spin reorientation is assumed to be the origin of the shape asymmetry of the hysteresis loops, these results would imply that spin reorientation is not temperature independent. Hence, to explain \(H_{c2}\) it must be taken into account the temperature dependence of spin reorientation and thermal activation effects on the AF state must be taken into account.

It is noticeable that added to an improvement in the symmetry of the hysteresis loop around point 2 (see figure 53) there is still a significant asymmetry between points 3 and 4 (see figure 53) in figures 73 and 74. Hence it could be that there is a small training effect on \(H_{c2}\) due to spin reorientation. It seems that the shift observed in \(H_{c2}\) when the field is cycled might be due to a small element of spin reorientation that takes place when the \(F\) is saturated in a negative direction. The magnitude of the observed shift in \(H_{c2}\) from \(n=1\) to \(n=2\) and from \(n=2\) to \(n=3\) is not big enough to determine a temperature dependence of spin reorientation at \(H_{c2}\) (see tables 6 and 7). However, the temperature dependence of the training in of \(H_{c2}\) is dominated by thermal activation of the AF (see figures 68 and 69). These results show that to measure the distribution of energy barriers to reversal it is necessary to measure the hysteresis loop that corresponds to \(n = 2\). Otherwise, the values obtained for \(H_{ex}\) as function of the temperature of activation would contain the dependence on spin reorientation. Moreover, the study of training effects and thermal activation to obtain the temperature dependence of \(H_{ex}(n)\) cannot be normalised to \(H_{ex}(n=1)\), but to \(H_{ex}(n=2)\) as this term would be almost free of spin reorientation effects (see table 4 and 5). Note that figures 69 and 70 have been normalised to \(n=1\) as it is generally done in the literature. However these figures were used to show just the qualitative behaviour of \(H_{ex}(n)\) as a function of temperature.
Figure 73 Sample set of hysteresis loops and their inverse loop to show asymmetry at points 2 and 6 for IrMn(3nm)/CoFe(10nm).
Figure 74 Sample set of hysteresis loops and their inverse loop to show asymmetry at points 2 and 6 for IrMn(5nm)/CoFe(10nm).
### Chapter 6: Thermal Effects on Exchange Biased Systems

#### Table 6

<table>
<thead>
<tr>
<th>$T(K)$</th>
<th>$H_{cl}(n)$ (Oe)</th>
<th>$H_{c2}(n)$ (Oe)</th>
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<th>$\Delta H_{c2}$</th>
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$H_{cl}$ and $H_{c2}$ as a function of the cycle number, $n$, and the temperature of measurement for IrMn(3nm)/CoFe(10nm) and shift in $H_{cl}$ from $n=1$ to $n=2$ and the shift in $H_{c2}$ from $n=1$ to $n=3$.

#### Table 7

<table>
<thead>
<tr>
<th>$T(K)$</th>
<th>$H_{cl}(n)$ (Oe)</th>
<th>$H_{c2}(n)$ (Oe)</th>
<th>$\Delta H_{cl}$</th>
<th>$\Delta H_{c2}$</th>
<th>$H_{ex}(n)$ (Oe)</th>
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<td>-56  48  -47</td>
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$H_{cl}$ and $H_{c2}$ as a function of the cycle number, $n$, and the temperature of measurement for IrMn(5nm)/CoFe(10nm) and shift in $H_{cl}$ from $n=1$ to $n=2$ and the shift in $H_{c2}$ from $n=1$ to $n=3$. 
6.3.4 Spin freezing

From figures 68 and 69 it appears there is different low temperature behaviour for $H_{c1}$ and $H_{c2}$. Both figures show a dramatic increase in $H_{c1}$ whereas $H_{c2}$ plateaus at a temperature substantially below $T_{NA}$ close to 25K. This phenomenon results in a sudden increase of $H_C$ at such temperatures, as shown in figures 66(b) and 67(b). Given our detailed analysis of the thermal activation and the spin reorientation this effect clearly arises due to a third mechanism. It is known that due to frustration there are disoriented spins at the interface between $F$ and $AF$ which are generally represented in theoretical models as an interfacial exchange stiffness constant [e.g. Malozemoff (1987), Stiles and McMichael (2001)]. It seems that the observation of this dramatic increase in $H_{c1}$ and the constant behaviour of $H_{c2}$ below 25K are due to a spin freezing type of behaviour similar to that of a spin glass structure. This was suggested by Hillebrands (2005). In this effect it is believed that there are interfacial spins in either the $F$ or $AF$ at the interface that are disordered. Hence they do not transmit the exchange interaction effectively. However, if these spins order over a given range of temperatures this will increase the interfacial exchange coupling constant. Similar results have been obtained by Takano et al., (1997), at temperatures below 50 K for CoO/MgO. Sadia Manzoor (to be published) obtained similar results on FeMn/NiFe showing spin freezing behaviour below 50K for FeMn grain sizes below 4nm, as shown in figure 75. In this instance this effect is far removed in temperature from the normal training and thermal activation effects that occur at much higher temperatures. However in other systems it is perfectly possible that the distribution of temperatures over which the spin freezing takes place can overlap with the spin reorientation effect and possibly the thermal activation of the $AF$.

From figure 63 results obtained from thermal activation within the $AF$ above $T_{NA}$ and results obtained from the study of training effects at lower temperatures can be compared. It has been shown in section 6.2 that following the protocol in figure 48 leads to a maximum absolute value of $H_{ex}$ for $T_{NA}$ and below. However for temperatures below $T_{NA}$, training measurements obtained by following protocol 50, show how spin freezing affects $H_{ex}$, leading to higher absolute values for that range of temperatures.
Thus in this instance there are three distinct and apparently independent contributions to both the exchange bias and the training in $F/AF$ bilayers. An example of this type of behaviour is shown in figures 66, 67, 68 and 69. Furthermore, as previously reported (Vopsaroiu et al. 2004), FeMn/NiFe samples grown with controlled grain size were produced with differing grain sizes. Figure 76 shows room temperature measurements of $H_{ex}$ and $H_C$ for the FeMn system different grain sizes. These data show a pronounced increase in thermally activated training effects as the grain size is reduced. This is indicative of a grain size and probably grain volume dependent energy barrier distribution in the $AF$. These data is in contrast to previous reports of an increased stability with reducing grain size in oxide systems (Takano et al., 1997). Also, a significant increased spin reorientation effect in this system is observed as the grain size is reduced (see figure 74). This might be due to an increase in the number of uncompensated spins at the interface arising due to an increased interface roughness as the grain size gets smaller.

![Image](https://example.com/image.png)

**Figure 75** Variation of $H_{ex}$ with the temperature of measurement for FeMn/NiFe with different grain sizes of the $AF$. Courtesy of Sadia Manzoor.

Hence, three different effects contribute to the origin of exchange bias and training effects; thermal activation, spin reorientation, and spin freezing. Determination of different dependences of thermal activation and spin reorientation processes might
account for the observed peak in $H_C$ with the temperature of thermal activation shown in figure 58 and 59.

![Figure 76 Exchange field, $H_{ex}$, and coercivity, $H_C$, as a function of the median grain size diameter $D_m$. (Sadia Manzoor et al., 2004)](image)

### 6.4 Angular dependence of $H_C$ and $H_{ex}$ in IrMn/CoFe bilayers

The symmetry of the anisotropy in IrMn($t_{AF}$)/CoFe(10nm) was also studied with 3nm and 5nm thick IrMn. Both $H_C$ and $H_{ex}$ were studied as function of the angle, $\theta$, at which the external field is applied relative to the easy anisotropy axis direction induced during sample growth. The measurement procedure described in figure 49 was followed with certain restrictions. Limitations in the capabilities to control the sample position and angle rotations within the cryostat made it impossible to control temperature variations. Hence measurements were made at a set temperature of 25°C. As described in the measurement protocol, figure 49, the order in the AF was set to the same state before each measurement by applying a saturating field to F in the direction of the exchange anisotropy axis ($\theta = 0$) for 15 minutes at the temperature of measurement 25°C. These conditions were sufficient to achieve reproducibility for this type of measurement. The time spent in reverse bias, i.e., with the F reverse saturated (point 4 in figure 53), was significantly shorter than the time spent with the F positively saturated during the resetting stage. The time spent with the F saturated during measurement was about 5
minutes for all measurements. Furthermore, as the sweep-rate was constant for all measurements, the maximum magnetic field applied selected for each measurement was chosen so that the time spent at point 4 and at point 1 (see figure 53) was the same and constant for all measurements. Having taken all these factors into consideration, measurements were taken after \( AF \) resetting at different values of \( \theta \).

The results obtained for \( H_{ex} \) measured for different angles of the \( AF \) anisotropy axis in relation to the applied field are shown in figures 77 and 78.

![Figure 77 and 78 Exchange field versus angle of measurement for IrMn(5nm)/CoFe(10nm) (77) and IrMn(3nm)/CoFe(10nm) (78). Solid lines are the best fit to the data.](image)

These data show the unidirectional characteristic of the \( H_{ex} \) described by Ambrose et al., (1997), showing the same symmetry as observed in their study, i.e.,

\[
H_{ex}(\theta) = H_{ex}(\theta + 2\pi) \quad (6.2)
\]

\[
H_{ex}(\theta) = H_{ex}(-\theta) \quad (6.3)
\]

Such symmetry conditions yield an expression of the form,

\[
H_{ex} = \sum_{n} a_n \cos(n\theta) \quad (6.4)
\]

which is valid irrespective of the model used to describe the exchange coupling. The coefficients that appear in the series expansion depend on the spin structure of the magnetic layers. These cosine terms are related to the microscopic interaction between the magnetic moments of the sublattices. Note that a first order expansion would lead to
the angular dependence predicted by Meiklejohn and Bean (1956) for $H_{ex}$ for the case of perfectly uncompensated interfaces, with $a_i = K_F$; where $K_F$ is the uniaxial anisotropy constant of the $F$. The data shown in figures 77 and 78 satisfy the conditions given by equations 6.2 and 6.3. Also, 

$$H_{ex}(\theta + \pi) = -H_{ex}(\theta)$$  

(6.5)

therefore, the series expansion (6.3) has to be an odd cosine series which reflects the unidirectional characteristic of $H_{ex}$.

Figures 77 and 78 exhibit a broad maximum ($\theta \pm 30^\circ$) displaced from $\theta = 180^\circ$, similar to the case of the minimum which is also broad. Figure 77 shows this effect more clearly. The positions of the maxima and minima are consistent with the single cosine function suggested by Meiklejohn and Bean (1956). However the form of the curve requires a higher order cosine expansion. From the data obtained no further evidence is found to support a four-fold anisotropy in $H_{ex}$ as proposed by Tang et al., (2000).

The solid lines shown in figures 77 and 78 are best fits to the data obtained from

$$H_{ex}(t_{AF} = 5nm) = -165.5(\cos(\theta) - 0.11\cos(3\theta) - 0.01\cos(5\theta) - 0.01\cos(7\theta))$$  

(6.6)

$$H_{ex}(t_{AF} = 3nm) = -20.04(\cos(\theta) - 0.12\cos(3\theta))$$  

(6.7)

Figures 79 and 80 show the angular dependence obtained for $H_C$ for IrMn(5nm)/CoFe(10nm) and IrMn(3nm)/CoFe(10nm) respectively. Here the symmetries observed are given by equations 6.8, 6.9, and 6.10, which lead to a series expansion of even cosine terms reflecting the uniaxial character of the anisotropy that gives rise to $H_C$. 

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Figure 79 and 80 Coercivity versus angle of measurement for IrMn(5nm)/CoFe(10nm) (79) and IrMn(3nm)/CoFe(10nm) (80). Solid lines are the best fit to the data.

\[
H_c(\theta) = H_c(\theta + 2\pi) \quad (6.8)
\]

\[
H_c(\theta) = H_c(-\theta) \quad (6.9)
\]

\[
H_c(\theta + \pi) = H_c(\theta) \quad (6.10)
\]

The data in figures 79 and 80 were fitted using

\[
H_c(t_{AF} = 5nm) = 19.16(1 + 0.46\cos(2\theta) - 0.15\cos(4\theta)) \quad (6.11)
\]

\[
H_c(t_{AF} = 3nm) = 38.56(1 + 0.25\cos(2\theta) + 0.06\cos(4\theta)) \quad (6.12)
\]

The sharpness in the maxima in $H_c$ also requires a higher order term to obtain a fit. These data also show the critical importance of the sample alignment for valid measurements of exchange biased materials. It is clear from these figures that whilst the fit to the cosine series expansion is good, it is not perfect. There are a number of reasons for this associated with the measurement procedure. Measurement of such systems require the sample to be in a uniform and reproducible state at the beginning of each measurement. Also the magnetic history in terms of field, time and temperature must be known and invariant for each measurement. Hence, a measurement procedure such as that described in figure 50 would be necessary to achieve such conditions. Also polycrystalline samples, such as the samples used in this experiment, have significant non-uniformities in terms of grain size and its distribution, and particularly interface
quality. Hence the overall quality of the fit to the data in spite of the difficulties and limitations is more than acceptable.

These measurements show that there are several anisotropies involved in the exchange bias effect, implied by the Fourier cosine expansions of $H_C$ and $H_{ex}$ (Ambrose et al., 1997). Whether there is any dependency on the direction of rotation of the sample has not been determined by this experiment. Also, the results obtained in section 6.2 also lead to believe that thermal activation within the AF might lead to $H_C$ and $H_{ex}$ variations during measurements.
Chapter 7

Conclusions and Further Work

7.1 Conclusions

The principle aim of this work was to develop simple measurement procedures which would lead to measurement reproducibility. However, this work lead to some other results that made all the effort worthwhile: the precise and detailed study of a set of samples and the confirmation that the protocols developed are effective in other materials and were successfully used by other researchers. The protocols allow for the understanding of the changes occurred in the exchange biased systems during measurements. With the measurement reproducibility it has been shown that the maximum coercivity of the hysteresis loop of an exchange biased system does not correlate to the median blocking temperature. All the results obtained are consistent with a grain size dependency of the exchange biased phenomenon.

The main achievement was the most detailed measurement study ever reported of a small set of samples. The materials used, IrMn/CoFe, were chosen because they are the most commonly used in the production of read heads. They are chosen because the large AF anisotropy exhibited even for very small IrMn thicknesses. Also, IrMn/CoFe bilayers are reasonably stable with the temperature. This system is more stable than FeMn/NiFe, and although PtMn/CoFe is yet more stable it is also much more expensive. Improvement of the IrMn/CoFe system to exhibit higher anisotropy and better stability than PtMn/CoFe is now under investigation. Tsunoda et al., (2005), have already shown that it is possible to obtain IrMn/CoFe with higher anisotropy than PtMn based systems by substrate heating during the deposition of the IrMn and post deposition thermal annealing of the system. Other disadvantage of PtMn-based samples is the high temperatures required in its annealing. Such high temperature can damage the adjacent layers that constitute the read-heads.

The thermal stability of the exchange biased bilayers also depends on the median grain size of the antiferromagnet layer. The grain size is also controlled by the thickness of
the layer in the case of magnetron sputtered systems (Carey et al., 2001). Therefore, the thicknesses chosen for the IrMn layer, were 3nm and 5nm. The 3nm IrMn sample has been shown to be very thermally unstable, revealed by a pronounced shift of the hysteresis loop with the temperature of thermal activation, the temperature of measurement and the number of repetitions of field cycling. The other thickness (5nm) was expected to be less thermally active but still to show some activation so that thermal stability could be studied and compared. A thicker (>5nm) IrMn layer would be too stable for the study of thermal activation. An estimation of the threshold thickness for thermal stability would be in the range of 7 to 10nm IrMn.

An interesting and challenging part of the development of the protocols was to understand that changes induced in the ferromagnet had implication on changes in the environment of the antiferromagnet. Such changes are only seen when the ferromagnet is measured for a second time. The reversal of the magnetisation of the ferromagnet, led to changes in the antiferromagnetic state caused by the exchange interaction through the interface between the materials. Time and temperature proved to be crucial parameters for the changes in the magnetic state of the antiferromagnet. Those changes can only be "seen" through the effect they have on the adjacent ferromagnet.

The measurement protocols have been successfully used with other materials (Sadia Manzoor, et al., 2005, Vallejo-Fernández et al., 2006) and it has been proven that these procedures work. The important aim of measurement reproducibility has been achieved. Induced changes in the exchange biased system due to the application of a magnetic field at a given temperature and during a certain time can be erased, and the reproducibility of the original state defined by $H_{ex}$ can be obtained to within a 1% error. This is achieved as long as a saturating field is applied at a higher temperature for a time as long as three times the time over which the changes took place.

Using the measurement protocols developed for the study of thermal activation it was proven that there is no correlation between the maximum coercivity and the median blocking temperature. This has been shown by published work described in section 6.2 and also observed by other researchers (Sadia Manzoor et al., 2005, and Vallejo-Fernandez et al., 2006).
The detailed and careful use of the procedures lead to the identification of three different contributions to exchange anisotropy, namely spin freezing, spin reorientation and thermal activation (section 6.3). Each of these contributions was shown to be temperature dependent and to be dominant at different temperature ranges. Spin-flop freezing occurs below 25K for IrMn/CoFe. Also the increase in the magnitude of the exchange field due to spin freezing at low temperatures has been shown to be dependent on the grain size distribution, being larger for smaller grains. Thermal activation is also grain size dependent (Sadia Manzoor et al., 2005). The shift produced in $H_{c1}$ with field cycling has been shown to be due exclusively to spin reorientation. $H_{c2}$ remains almost unaltered with field cycling so long as equal times are spent in positive and negative magnetisation. Measurements done following the protocol of figure 50 ensured that thermal activation that occurs in the AF during measurement was negligible. The thermal activation during reverse bias averages with that occurred at positive bias. Therefore no significant shift is observed in $H_{c2}$ due to field cycling at a fixed temperature. Any small changes in $H_{c2}$ may be due to a further spin reorientation.

The contribution of spin reorientation to the exchange field is also accompanied by hysteresis loop asymmetry. The shift of $H_{c1}$ and $H_{c2}$ with field cycling at different temperatures showed that spin reorientation is almost temperature independent from 25K to 225K for IrMn/CoFe. Above this temperature spin reorientation is temperature dependent and disappears at temperatures close to the maximum temperature in the blocking temperature distribution (~375K). Also, in this range of temperatures, steps in subsequent loops also decrease. From these results it is clear that spin reorientation occurs over a distribution of energy barriers. This appears to be in agreement with the symmetry improvement of the hysteresis loop shape with the temperature of measurement. The asymmetry of the loop shape is thought to be due to spin reorientation processes such as that described by Hoffmann (2004), although that mechanism of spin reorientation is only proposed for zero temperature measurements.

According to the observed temperature dependence of the symmetry of the loop shape and the observed variation of $H_{c1}$ and $H_{c2}$ with field cycling, there is a temperature dependence of the spin reorientation contribution to the exchange field. However, the overlapping of the contributions from spin reorientation and thermal activation in the range of temperatures measured makes it difficult to distinguish the weight of each contribution to the exchange field.
All results obtained are consistent with a grain size dependence interpretation of the exchange bias phenomenon and in accordance with the results obtained from grain size effects on the exchange biased FeMn/NiFe (Sadia Manzoor et al., 2006). All these results lead to an interpretation by which exchange bias is grain-size dependent and probably grain-volume dependent. Also, the identification of these above origins of exchange bias showed that each of their effects occurs over a distribution of energy barriers. This interpretation is in agreement with the grain models of exchange bias [e.g. Fulcomer and Charap, 1972(a,b), Nishioka et al., 1996, Takano et al. 1997, Tsunoda and Takahashi, 2002]. This is in contrast with the majority of models published in the literature, which are based on the formation of magnetic domains at the interface between the ferromagnet and antiferromagnetic layers.

7.2 Further Work

The measurement procedures developed have been applied to study the grain size dependence of exchange bias on FeMn/NiFe systems (Sadia Manzoor et al., 2005). The results obtained showed that thermal activation is grain size dependent. Also in section 6.3 it was shown that there are three different contributions to exchange bias that happen to occur over a distribution of energy barriers. The materials used for this study showed these three effects tend to overlap in certain ranges of temperatures. In fact spin freezing occurs at relatively low temperatures, less than 25K for IrMn/CoFe systems, and up to 50K for FeMn/NiFe, whilst spin reorientation and thermal activation occur in a wider range of temperatures. Spin reorientation occurs up to temperatures close to the maximum of the blocking temperature distribution and thermal activation occurs above the free-thermal-activation temperature, $T_{NA}$. At the time of writing the optimum material was not found in which these three effects do not overlap, so that the distributions of energy barriers could be identified separately. For both FeMn/NiFe and IrMn/CoFe both spin freezing and spin reorientation overlap at low temperatures, for higher temperatures there is also overlapping of spin reorientation and thermal activation.

The study of thermal activation shown in section 6.2 has only been applied to polycrystalline samples. The application of the protocol shown in figure 50 to single-
crystal-antiferromagnet exchange biased systems has not been studied yet although
there has been an attempt in collaboration with the Technische Universität
Kaiserslautern, a partner of the research training network with which this project was
linked. However, the production of single crystal FeMn/NiFe requires the growth of a
thin ferromagnetic layer to maximise the exchange bias, which in turn resulted too thin
to provide a reasonable magnetisation signal to be detected by the vibrating sample
magnetometer. The growth of single crystal epitaxial exchange biased systems would
shed light in the understanding of spin reorientation training effects and thermal
stability to a larger state.

The study of the symmetry properties of the exchange anisotropy has been restricted to
room temperature measurements due to the incapability to control simultaneously the
sample position relative to the pick-up coils and its angular position within the cryostat.
The measurement protocol shown in figure 49 is for temperature measurements of the
asymmetry of the exchange anisotropy. The acquisition of a new vector Vibrating
Sample Magnetometer with temperature capabilities will allow these measurements to
be made. Also the use of a vector VSM and the protocol shown in figure 50 would
extend the present knowledge of training effects and would provide information relative
to the effect of the spin reorientation and spin freezing on the magnetisation of the
ferromagnet measured along the hard axis of the exchange anisotropy.

Recently, it has been shown that for IrMn/CoFe exchange biased systems substrate
heating during deposition of the IrMn layer has an influence on its crystallographic
structure. Samples subject to substrate heating at this stage exhibit an increased
exchange anisotropy constant (Tsunoda and Takahashi, 2005). Also post deposition
field annealing increases the exchange anisotropy for a range of annealing temperatures
(Tsunoda et al. 2005, Imakita et al., 2005). Research is being undertaken on samples
grown in the conditions described with a plasma sputtering system that allows grain
size control (Vopsaroiu et al., 2004). By using the protocols shown in figures 46-48 and
50 and the study of the grain size distribution within the IrMn layer for each substrate
temperature, it is expected that a correlation between the enlargement of the median
grain size and the thermal stability will be found.
Further work needs to be carried out to obtain a detailed description of the complex contributions to exchange bias. The use of measurement protocols that provide detailed information about the measurement procedures proves to be of great help for the modelling of exchange biased systems. Due to the complexity of the interactions involved in the exchange bias phenomenon, large scale computational modelling is needed to describe such systems. Currently, the results obtained in the development of this thesis are being considered for the development of an exchange biased model for biaxial anisotropy systems. Also, the domain state model (Nowak et al., 2002) is currently being modified by the same author and collaborators by grain size dependence by removing the periodical boundary conditions and introducing finite size discretisation of the lateral size of the spin ensemble.
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Abbreviation</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$a$</td>
<td>Area</td>
<td>Area of the $AF$-grain-interface per spin</td>
</tr>
<tr>
<td>$A$</td>
<td>Cross sectional area</td>
<td></td>
</tr>
<tr>
<td>$a$</td>
<td>Lattice parameter</td>
<td></td>
</tr>
<tr>
<td>$A$</td>
<td>Exchange stiffness</td>
<td></td>
</tr>
<tr>
<td>$A_{AF}$</td>
<td>Exchange stiffness of the antiferromagnet</td>
<td></td>
</tr>
<tr>
<td>$AC$</td>
<td>Alternate Current</td>
<td></td>
</tr>
<tr>
<td>$AF$</td>
<td>Antiferromagnet</td>
<td></td>
</tr>
<tr>
<td>$A_{F,AF}$</td>
<td>Exchange stiffness at the interface between $F$ and $AF$ layers</td>
<td></td>
</tr>
<tr>
<td>$AGM$</td>
<td>Alternating Gradient Magnetometer</td>
<td></td>
</tr>
<tr>
<td>$a_n$</td>
<td>Coefficient in the cosine series expansion of $H_{ex}$</td>
<td></td>
</tr>
<tr>
<td>$CIP$</td>
<td>Current in plane</td>
<td></td>
</tr>
<tr>
<td>$CPP$</td>
<td>Current perpendicular to plane</td>
<td></td>
</tr>
<tr>
<td>$D$</td>
<td>Median grain diameter</td>
<td></td>
</tr>
<tr>
<td>$DC$</td>
<td>Direct current</td>
<td></td>
</tr>
<tr>
<td>$d_w$</td>
<td>Domain wall width</td>
<td></td>
</tr>
<tr>
<td>$EB$</td>
<td>Exchange Bias</td>
<td></td>
</tr>
<tr>
<td>$F$</td>
<td>Ferromagnet</td>
<td></td>
</tr>
<tr>
<td>$f(T_B)$</td>
<td>Distribution of blocking temperatures</td>
<td></td>
</tr>
<tr>
<td>$f(AE)$</td>
<td>Distribution of energy barriers</td>
<td></td>
</tr>
<tr>
<td>$f_0$</td>
<td>Attempt frequency</td>
<td></td>
</tr>
<tr>
<td>$FcC$</td>
<td>Face Centred Cubic</td>
<td></td>
</tr>
<tr>
<td>$GMR$</td>
<td>Giant Magneto Resistance</td>
<td></td>
</tr>
<tr>
<td>$H_C$</td>
<td>Coercivity</td>
<td></td>
</tr>
<tr>
<td>$H_{c1}$</td>
<td>First coercivity point. Value of the coercive point for the first branch of the hysteresis loop</td>
<td></td>
</tr>
<tr>
<td>$H_{c2}$</td>
<td>Second coercivity point. Value of the coercive point for the second branch of the hysteresis loop</td>
<td></td>
</tr>
<tr>
<td>$H_{ex}$</td>
<td>Exchange bias field or exchange field</td>
<td></td>
</tr>
<tr>
<td>$H_K$</td>
<td>Anisotropy field</td>
<td></td>
</tr>
<tr>
<td>$H_r$</td>
<td>Remanent coercivity</td>
<td></td>
</tr>
<tr>
<td>$H_D$</td>
<td>Demagnetising field</td>
<td></td>
</tr>
</tbody>
</table>
List of Symbols and Abbreviations

$I$ Electric current
$J$ Exchange integral
$J_{AF}$ Exchange coupling constant between $AF$ spins
$J_{eff}$ Effective exchange coupling between $AF$ spins
$J_i$ Exchange integral between the spins of $i$ and $j$ atoms
$J_{int}$ Interfacial exchange constant between the $F$ and a $AF$ grain
$J_{net}$ Direct coupling per spin at the $F$-$AF$ interface
$J_{sf}$ Effective spin-flop exchange per spin
$K$ Unidirectional anisotropy constant
$K$ Crystalline anisotropy
$K_{AF}$ Antiferromagnet anisotropy constant, uniaxial anisotropy in the antiferromagnet
$K_{eff}$ Effective anisotropy
$k_B$ Boltzman's constant
$K_F$ Ferromagnet anisotropy constant, uniaxial anisotropy in the ferromagnet
$L$ $AF$ domain size
$L(\alpha)$ Langevin function
$L_{ex}$ Exchange length
$L$ Orbital angular momentum
$m$ Magnetic moment
$M$ Scalar value of the magnetisation
$m_{AF}$ Magnetic moment of the antiferromagnet
$M_{AF}$ Magnetisation of the $AF$
MBE Molecular Beam Epitaxy
$M_F$ Vectorial magnetisation of the ferromagnet
$M_F$ Magnetisation of the ferromagnet
$M_{AF}^{rev}$ Reversible component of the magnetisation of the $AF$
$M_{AF}^{irr}$ Irreversible component of the magnetisation of the $AF$
MOIF Magneto-optic indicator film technique
MR Magnetoresistance, magnetoresistive
MRAM Magnetic Random Access Memory
$M_S$ Saturation magnetisation
List of Symbols and Abbreviations

\( N \)  
Number of spins at the interface

\( N \)  
Number of turns in the coil

\( n \)  
Cycle number. Index that indicates the number of hysteresis loops measured consecutively.

\( N_D \)  
Demagnetising factor

\( P(t) \)  
Probability of remaining in an initial state for a period \( t \)

\( R \)  
Resistance

\( r_{3d} \)  
3d shell radius

\( r_a \)  
Atomic radius

\( r_c \)  
Critical radius of a magnetic particle

\( RF \)  
Radio Frequency

\( r_{ij} \)  
Interatomic distance

\( \text{RKKY} \)  
Indirect exchange interaction

\( S \)  
Spin angular momentum

\( S(H) \)  
Magnetic viscosity coefficient

\( t \)  
Thickness

\( t \)  
Time

\( T \)  
Temperature

\( T_{\text{Act}} \)  
Temperature of thermal activation.

\( t_{\text{AF}} \)  
Antiferromagnet thickness

\( T_B \)  
Blocking temperature

\( T_B^{\text{\textbar}} \)  
Median blocking temperature

\( T_C \)  
Curie temperature

\( \text{TEM} \)  
Transmission electron microscopy

\( t_F \)  
Ferromagnet thickness

\( \text{TMR} \)  
Tunnelling Magneto Resistance

\( T_N \)  
Néel temperature

\( T_{\text{NA}} \)  
Thermal-activation-free temperature

\( V \)  
Voltage

\( V \)  
Particle volume

\( V_p \)  
Critical volume of a magnetic particle

\( z \)  
Parameter in the order of unity that arises from interface irregularities (Imry and Ma, 1975)
List of Symbols and Abbreviations

\( \alpha \)  
Angle formed between the \( AF \) interfacial spins and the \( AF \) uniaxial anisotropy axis

\( \alpha_{\text{crit}} \)  
Critical angle in the model of Stiles and McMichael (1999,a)

\( \beta \)  
Angle formed between the \( F \) spins and the \( AF \) uniaxial anisotropy axis

\( \Delta E \)  
Energy barrier

\( \Delta E_c \)  
Critical energy barrier

\( \Delta \sigma \)  
Interfacial coupling energy

\( \mu_r \)  
Magnetic relative permeability

\( \mu_s \)  
Magnetic moment due to spin angular momentum

\( \theta \)  
Angle formed by the applied magnetic field and the anisotropy easy axis of an exchange biased system.

\( \lambda \)  
Parameter related to the spin structure and the material

\( \mu \)  
Magnetic permeability

\( \mu_B \)  
Bohr's magneton

\( \mu_0 \)  
Magnetic relative permeability in vacuum

\( \nu \)  
Number of interfacial spins per \( AF \) grain

\( \xi \)  
Interface thickness (between \( AF \) and \( F \))

\( \phi_f \)  
Magnetic flux

\( \sigma \)  
Domain wall energy

\( \sigma_0 \)  
Domain wall energy at zero Kelvin

\( \tau \)  
Relaxation time
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Appendix: Publications


