

Magnetic interaction between antiferromagnetic and ferromagnetic films: $Co/Fe_{50}Mn_{50}$ bilayers on Cu(001)

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Introduction

The manipulation of systems involving a ferromagnetic (FM) material has often led to the discovery of unexpected magnetic properties, offering the possibility of new and interesting applications. More than forty years ago, a way to modify the magnetic behaviour of a ferromagnet has been found, by having it in direct contact with an antiferromagnetic (AFM) material. While the hysteresis loop of an FM material alone is centered at zero of the external field axis, an FM-AFM system exhibits a hysteresis loop displaced from zero by a certain amount. In other words, the magnetic interaction exerted by the antiferromagnet onto the ferromagnet induces in the latter just *one* stable configuration for the direction of the magnetization. When trying to switch the magnetization by 180° from this direction, a higher energy is needed than to switch it back. This directional anisotropy in the magnetization of the FM layer is induced by exchange interaction of the AFM layer, and it has therefore been dubbed *exchange anisotropy*. The shift in the hysteresis loop, being just one of the manifestations of the FM-AFM interaction, has been used in recent years in a new class of devices, promising candidates for applications in the field of magnetic recording media. Indeed this technological interest induced a flourishing of theoretical and experimental works, aiming on the one hand side to a fundamental understanding of the phenomenon, and on the other hand side to the improvement of device performance.

The simplest approach to the FM-AFM interaction is obtained by using materials in the thin film form. Indeed the most recent studies have been performed on systems comprising of a thin FM layer in contact with a thin AFM layer. This allows to obtain a structure more controllable than, for example, small particles or inhomogeneous materials, in which the FM-AFM interaction has been studied in the past. The possibility to use thin film magnetic materials as a model system mainly derives on the one hand from the improvement and invention of characterization techniques for magnetic surfaces, and on the other hand from the powerful theoretical methods which have been introduced to model these structures. In spite of the use of thin film FM-AFM bilayers, and notwithstanding the large number of investigations, the phenomena arising from the magnetic interaction at an FM-AFM interface still remain somehow puzzling. The problem is indeed complex from several points of view. At first, one has to consider that AFM materials are involved. Their magnetic properties are generally less known than the ones of FM materials, since most of the techniques used to characterize magnetic properties probe the spontaneous magnetization of the sample. At second, the interaction is realized at the interface, which is a location hidden to an easy experimental investigation. This is also connected to the fact that not all the parameters involved in such an interaction, such as structural perfection, influence of magnetic anisotropy, spin configuration, or domain formation, are easily controlled at the same time. Finally the character of the interaction is such that not only the properties of the FM material are modified by the proximity of the AFM material, but also the properties of the latter are changed in a mutual interaction. Therefore to fully understand the problem one should look at both sides of the interface.

In order to study the magnetic interaction at an FM-AFM interface, $Co/Fe_{50}Mn_{50}$ bilayers have been used in this work, where Co represents the FM material, and $Fe_{50}Mn_{50}$ the AFM material. The epitaxial growth of this system on a Cu(001) single crystal, demonstrated in this thesis, assures that also the bilayers are in a single crystalline state. This is actually a good starting point with respect to the fundamental understanding of the magnetic interaction at the interface. Many previous investigations have in fact been focussed on polycrystalline films obtained by sputtering techniques. These systems, besides having a rougher interface, lead to the complication that the coupling between the ferromagnet and the antiferromagnet is influenced by the presence of different grains. The interface conditions of the epitaxial bilayers studied here are instead more controllable, and no grains of different crystallinity are present. Since the FM-AFM interaction is very sensitive to the interface conditions, epitaxial bilayers are therefore better candidates in order to understand the details of the magnetic coupling. The techniques used here to investigate the magnetic properties of these bilayers do not allow the direct detection of AFM order. The structure of the AFM order in $Fe_{50}Mn_{50}$ films has instead been deduced indirectly from the modifications of the Co magnetic properties, resulting from the interaction with the AFM layer.

The heart of the thesis relies on the observations of magnetic domains in the Co/Fe₅₀Mn₅₀ bilayers by using a photoelectron emission microscope (PEEM). X-ray magnetic circular dichroism (XMCD) has been used as contrast mechanism, where the excitation source was supplied by a synchrotron radiation source. One of the capabilities of this technique, which turned out to be particularly useful here, is its elemental specificity, and the possibility to access buried layers. It has been therefore possible to image the domain configuration for both the Co/Fe₅₀Mn₅₀ and the Fe₅₀Mn₅₀/Co bilayers on Cu(001). The formation of domains in an FM material is the result of the minimization of the different involved energy terms. When the FM material is brought into contact with the AFM material, an "extra" contribution may influence both the domain shape and the direction of the magnetization inside the domains. This has indeed been found here for a Co film coupled to an AFM Fe₅₀Mn₅₀ film. This observation not only shows how the properties of the FM Co layer are modified by the interaction with the antiferromagnet, but also leads to some considerations on the properties of the AFM material that induce these effects. In particular the presence of small domains, probably of topological origin, is indirectly deduced in the AFM ordered $Fe_{50}Mn_{50}$ film. These domains are considered to be one of the crucial points when describing the interaction at an FM-AFM interface. The other key to understand this interaction relies on the observation of ferrimagnetic moments in the $Fe_{50}Mn_{50}$ films. This is not due to a modification of the magnetic properties of $Fe_{50}Mn_{50}$ by the reduced dimensionality, but rather to induced moments by the proximity of the FM Co film. These induced moments are interpreted as being located at the interface between the "real" AFM $Fe_{50}Mn_{50}$ film and the FM Co film, thus mediating the magnetic interaction across the interface.

The thesis is organized as follows: chapter 1 describes the phenomenon exchange anisotropy from its discovery and first theoretical description to the most recent applications, with some emphasis on the observations that still remain without explanation. The system under investigation, i.e., $Co/Fe_{50}Mn_{50}$ bilayers deposited on Cu(001), is already introduce here. In chapter 2 the experimental techniques used are briefly described, together with the way the samples were obtained, in particular Fe_xMn_{1-x} films of different composition. Chapter 3 contains the results of the work: $Co/Fe_{50}Mn_{50}$ bilayers are characterized from a structural and magnetic point of view, and images of magnetic domains are shown from samples in different conditions. Finally in chapter 4 the results are discussed. At the end of the thesis some concluding remarks are given.

Chapter 1

Interaction between a ferromagnetic and an antiferromagnetic material

The magnetic properties of a ferromagnetic (FM) material are modified by the contact with an antiferromagnetic (AFM) material under some special preparation conditions. This has been discovered in 1956 by Meiklejohn and Bean in fine particles of Co (100-1000 Å) surface oxidized to form cobaltous oxide [1]. The resultant particles had a core of FM material (Co) and a shell of AFM material (CoO). As a result of the interaction a *unidirectional* anisotropy, dubbed *exchange anisotropy*, was established in the Co particles. The discovery of this new phenomenon and the first theoretical interpretation are the subject of section 1.1.

Especially in recent years the phenomena resulting from the interaction between an FM and an AFM material have found interesting applications. Some of them are considered in section 1.2. Special emphasis is given there to the use of an AFM material in a so-called *spin valve*. With the introduction of these devices, approximately ten years ago, the number of studies about exchange anisotropy increased in fact strongly. The scientific interest is here associated with the improvement of device performance. In spite of the flourishing of researches, the understanding of the FM-AFM interaction is still not completely established, as outlined in section 1.3. In particular it is not yet fully clear what the microstructural origin of the interaction is and no theory explaining all the experimental observations is available.

In order to study the FM-AFM interaction, the Co/Fe₅₀Mn₅₀ system has been used in this work. This is introduced in the last section of this chapter (1.4). The magnetic properties of the antiferromagnet Fe₅₀Mn₅₀, and in particular the bulk spin configuration as taken from literature, are described there. Co and Fe₅₀Mn₅₀ have been used in thin film form in the present work, in order to better control and characterize the FM-AFM interface. The mutual interaction between Co and Fe₅₀Mn₅₀ is in fact strongly related to the interface conditions.

1.1 Exchange anisotropy and related phenomena

Exchange anisotropy has been discovered [1] in fine Co particles having a CoO coating. CoO is an antiferromagnet with a Néel temperature (T_N) of 293 K. When cooling the particles from 300 K (cobaltous oxide in a paramagnetic state) to 77 K (cobaltous oxide in an AFM state) in a saturating magnetic field, a *unidirectional* anisotropy was observed. Since this anisotropy results from the exchange interaction between the FM and the AFM material, it was dubbed exchange anisotropy. Co particles without the CoO coating cooled down from room temperature to low temperature in a strong magnetic field exhibit a *uniaxial* anisotropy [2], that is two equivalent, opposite, easy directions of magnetization are present. When coated by CoO and cooled in a field, the direction of the field during cooling selects just *one* of these two directions, leading to the observed unidirectional anisotropy.

A manifestation of this unidirectional anisotropy is a displaced hysteresis loop which occurs after the sample is cooled in a magnetic field. The original loop of Meiklejohn and Bean from 1956 is shown in Fig. 1.1. The two hysteresis loops in this figure were taken at 77 K. The CoO



Figure 1.1: Hysteresis loops of CoO coated Co particles at 77 K after cooling from room temperature with no applied field (1) and under a saturating field (2). From Ref. [1].

coated Co particles were at first cooled from room temperature to 77 K in a zero external field. The corresponding hysteresis loop, labeled as (1) and drawn as a dashed line, is symmetric about the vertical axis. When the specimen is cooled to 77 K in a strong magnetic field (field cooling), the hysteresis loop (2), full line, is displaced to the left along the applied field axis. The value of the displacement, that is the distance between the origin of the axes and the center of the hysteresis loop, is usually called *exchange bias field* (H_{eb}). Together with the displaced hysteresis loop also an increase in coercive field is usually observed as a consequence of the interaction between an FM and an AFM material. Both effects are temperature dependent, disappearing when approaching the Néel temperature of the antiferromagnet. In particular the shift in the hysteresis loop disappears at a temperature usually called *blocking temperature* (T_B). In some cases T_B is much lower than T_N , while in other cases $T_B \approx T_N$ (see Tables 2-4 in Ref. [10]).

In order to understand the displaced hysteresis loop in the Co-CoO system from an intuitive point of view, the sketch reported in Fig. 1.2 can be useful [3]. It represents the first simple model put forward by Meiklejohn and Bean to explain the phenomenon. In (d) a hypothetical loop shifted to the left along the applied field axis is reported. The points (a), (b), and (c) on the loop correspond to the three states of the Co-CoO interface on the left of the picture. The arrows represent spins on Co atoms or ions, and the open circles are oxygen ions. When a strong field (H) is applied at 300 K along the vertical direction from the bottom to the top of the figure, the Co saturates in the direction of the field, but the paramagnetic CoO is little affected. However the spins of the first layer of the Co ions in the CoO are forced by exchange interaction to be parallel to the spins of the Co atoms in the metal. By cooling in the external field to below T_N , the antiferromagnet orders therefore as sketched in Fig. 1.2 (a), even after the field has been switched off (H = 0). The Co magnetization (M) is still along the direction imposed by the field. When a strong field is now applied in the downward (negative) direction (b), the spins in the Co will reverse, and the exchange coupling at the interface will also try to rotate the spins in the oxide. The crystal anisotropy in the antiferromagnet tries instead to avoid this spin rotation. As a result just the spins at the CoO interface are partially rotated as depicted in (b). Therefore, in order to rotate the Co magnetization, one has also to overcome the interaction exerted at the interface by the CoO, which tries to keep the spin direction in the original upward (positive) direction. The loop results therefore shifted to the left side along the applied field axis. When in fact a upward (positive) field is applied, the Co rotates at smaller fields, since now also the exchange interaction at the interface helps to restore the up (positive) spin direction as depicted in (c). The spins in the FM Co have therefore just one stable remanent configuration, when the spins are pointing upward. In other words the anisotropy is unidirectional.

From an analytical point of view the properties of the interaction at an FM-AFM interface may be derived by considering the energetic terms involved [4, 5]. Considering an FM layer of thickness t_{FM} and an antiferromagnetic layer of thickness t_{AFM} in contact with each other, and treating both layers as single spins, the energy per unit surface of the interface can be written



Figure 1.2: Mechanism of the shifted loop in the Co-CoO system. From Ref. [3].

as [6]:

$$E = -HMt_{FM}\cos\theta_{FM} + K_{FM}\sin^2\theta_{FM} + K_{AFM}\sin^2\theta_{AFM} - J\vec{S}_{FM}\vec{S}_{AFM}$$
(1.1)

For simplicity the anisotropy axes of the FM and AFM layers are considered to be the same and the anisotropy is uniaxial. The external field H is applied along this axis. \vec{S}_{FM} and \vec{S}_{AFM} are the spins in the FM and the AFM layers respectively. The angles θ_{FM} and θ_{AFM} are the angles of the FM and AFM materials magnetization with respect to the anisotropy axis. K_{FM} and K_{AFM} are the anisotropy constants of the FM and AFM layers respectively, and J is the exchange coupling constant of the interaction between the two layers. The first term in equation 1.1 describes the interaction between the external field and the magnetization of the FM layer (M). The second and the third term on the right hand side account for the anisotropy energy in the FM and the AFM layers respectively. Finally the forth term is the coupling energy between the two layers. If the antiferromagnetic anisotropy is considered to be large compared with J, the spins of the antiferromagnet are frozen along the easy axis direction [2] and equation 1.1 reduces to

$$E = -HMt_{FM}\cos\theta_{FM} + K_{FM}\sin^2\theta_{FM} - J\cos\theta_{FM}$$
(1.2)

From the first derivative of this expression one finds that the energy has extrema corresponding to saturation at $\theta = 0$ and $\theta = \pi$, for positive and negative applied field respectively. By calculating the second derivative, one finds that the $\theta = 0$ and $\theta = \pi$ configurations are minima for $J + H + 2K_{FM} > 0$ and $2K_{FM} - J - H > 0$ respectively [7]. From these conditions one obtains the right (H_{cR}) and left (H_{cL}) coercive fields as

$$H_{cR} = -\frac{2K_{FM} + J}{Mt_{FM}} \tag{1.3}$$

and

$$H_{cL} = \frac{2K_{FM} - J}{Mt_{FM}} \tag{1.4}$$

Since the coercive fields are not equal, the hysteresis loop is shifted along the applied field axis. The exchange bias field is the midpoint of the biased hysteresis loop, that is

$$H_{eb} = \frac{J}{Mt_{FM}} \tag{1.5}$$

This result is derived under the assumption of a large value of K_{AFM} . More generally one has always to consider an interplay between the anisotropy of the antiferromagnet and the exchange coupling energy J between the FM and the AFM materials. In this view from equation 1.1 one can obtain also a qualitative explanation for the increase in coercive field resulting from the FM-AFM interaction [6]. If the AFM anisotropy energy is large compared to J, as in the case considered above, the energy of the system will be minimized by keeping the angle θ_{AFM} low. The AFM sublattice magnetization will not rotate upon switching the FM layer, with the result that a displaced loop will appear. In the other case, when J is large compared to the anisotropy energy, it is energetically favorable to keep a low angle between the AFM and FM magnetization directions. In this case when reversing the FM layer also the AFM layer will reverse, and irreversible changes in the AFM material give rise to the increase in coercive field. Since an FM-AFM interface is usually believed to be characterized by lateral inhomogeneities, both effects could be present at the same time, with some AFM parts that switch together with the FM layer, and others that remain in the pinned direction.

1.2 Applications

The most promising application of systems exploiting the exchange bias phenomenology is in spin valve devices [8]. It was indeed the use of an antiferromagnetically exchange-biased bilayer

in a spin-valve [9] that induced a renewed interest and a flourishing of investigations on the exchange anisotropy phenomenon, both from a theoretical [7] and an experimental [10] point of view.

The working principle of a spin value is based on the giant magnetoresistance (GMR) effect [11, 12]. When two FM layers are separated by a non-FM spacer layer, the change in the mutual orientation of the magnetization in the two FM layers (parallel or antiparallel) is reflected in a change of the electrical resistance of the system. This variation of magnetoresistance can be up to one or more orders of magnitude bigger than the *anisotropic magnetoresistance* effect, which leads to a difference in resistance for current flowing parallel or perpendicular to the magnetization direction in a FM material. It was therefore dubbed "giant". The structure of a spin value is shown in Fig. 1.3 (a). Two FM layers are separated by a non-magnetic spacer



Figure 1.3: Multilayered structure of a spin-valve (a) and variation of magnetization (b) and magnetoresistance (c) as a function of applied field. From Ref. [10].

layer that mediates the magnetic interaction. One of the FM layers (labeled "Pinned" in the figure) has the magnetization direction "frozen" by the contact with an AFM layer. When the hysteresis loop of the spin value is measured (b) the "Free" layer switches its magnetization at values of external applied field around 0. Higher fields are needed to reverse the magnetization in the Pinned layer, the hysteresis loop of which is centered by the exchange anisotropy effect at an external field of about +400 Oe. As a consequence, the two FM layers will have a mutual magnetization direction that varies from parallel to antiparallel and to parallel again, giving rise to a change in the magnetoresistance (c) of the structure as a function of external field (GMR effect). In particular the resistance is "low" when the two FM layers have the same magnetization direction, and it is "high" when the FM layers have antiparallel magnetization direction. Therefore the external field, operating at values around zero, can set the low and the high value of magnetoresistance, which can be interpreted as the "0" and "1" value of a byte.

Such a device has been already proposed and used in several applications in the field of magnetic recording media. In particular a spin-valve is already in the market incorporated in read heads of hard disks [13]. One of the most exciting possible future applications of these devices is in non-volatile magnetic random access memory (MRAM) [14, 15]. Arrays of spin valves could be in fact used to build up memories for computers with the advantage to store data independently of the electric power supply.

1.3 Theoretical models and unsolved issues

Equation 1.2, which has been shown to explain the shifted loop, suffers actually from several simplifying approximations. One of these has already been mentioned, i.e., the infinitely large AFM anisotropy, that implies the frozen direction of the AFM spins. On the other hand in section 1.1 the intuitive explanation of the increased coercive field in exchange biased systems already involves an AFM spin rearrangement. Moreover equation 1.2 does not take into account domains in both the FM and the AFM materials. Finally it is valid, strictly speaking, just for *uncompensated* AFM interfaces, that is when the sum of the spins of the different sublattices at the AFM interface is not equal to zero. The exchange energy at the FM-AFM interface can be expressed as

$$E_{eb} = -J\vec{S}_{FM}\vec{S}_{AFM} \tag{1.6}$$

where \vec{S}_{FM} and \vec{S}_{AFM} are the magnetic moments at the FM and AFM interface, respectively. One immediately sees that E_{eb} , and therefore the loop shift, disappear if the AFM sublattices at the interface cancel each other to give a zero value of \vec{S}_{AFM} (compensated AFM interface). However in several systems comprising of a nominally compensated AFM surface, an exchange biased loop has been experimentally observed.

Moreover, the obtained theoretical value of H_{eb} , expressed by equation 1.5, results to be typically two orders of magnitude bigger than the experimental values, as it is for example discussed in Ref. [16]. In order to obtain a quantitative prediction of H_{eb} , some of the above mentioned approximations must be overcome. Mauri *et al.* [17], for example, hypothesized the formation of a kind of domain wall in the AFM material parallel to the interface. Upon rotating the FM material by an external field, the spins of the antiferromagnet remain fixed at the original pinning direction just in the bulk, far away from the FM-AFM interface. The interface AFM spins, in contrast, are dragged by the rotating FM spins. As a result the AFM spins form a spiraling structure along a direction perpendicular to the interface, that is a domain wall of energy proportional to $\sqrt{A_{AFM}K_{AFM}}$, where A_{AFM} and K_{AFM} are the exchange stiffness and the crystalline anisotropy of the AFM material, respectively. The calculations of Mauri *et al.* lead to the more realistic expression for the exchange bias field

$$H_{eb} = \frac{2\sqrt{A_{AFM}K_{AFM}}}{Mt_{FM}} \tag{1.7}$$

A similar expression for H_{eb} has been obtained by Malozemoff [16, 18, 19], starting from a different point of view. He proposed that the random field, induced by surface roughness or interfacial alloying, acting from the FM into the AFM layer, causes the latter to break up into small domains with boundaries perpendicular to the interface. Both models involve domain walls formation and therefore the analytic expression of H_{eb} is similar. Since the model proposed by Malozemoff involves lateral inhomogeneities at the FM-AFM interface, it can be applied also to compensated AFM surfaces, while for this case the model of Mauri *et al.* would predict a non-shifted loop. In order to explain the occurrence of exchange bias for compensated AFM interfaces, a different approach has been followed by Koon [20]. By using a micromagnetic numerical calculation he also obtained an expression for H_{eb} which leads to values comparable to the experimental ones. One of the results of his calculations is that a general occurrence in an exchange biased bilayer is a 90° angle between the spin direction in the ferromagnet and in the antiferromagnet. This so-called *spin-flop coupling* leads also to a canting of the interfacial AFM spins away from, or towards to, the FM magnetization direction, according to an antiferromagnetic or ferromagnetic coupling at the interface, respectively. In this case exchange bias results from the formation of parallel domain walls in the antiferromagnet, as in the model of Mauri *et al.*

Spin-flop coupling alone has been actually shown by Schulthess and Butler [21, 22] not to lead to an exchange biased hysteresis loop. Using a microscopic Heisenberg model, they solved the Landau-Lifshitz-Gilbert equation of motion obtaining that, for perfectly flat compensated AFM interface, a spin-flop coupling just leads to an increase in coercive field, as it is also encountered in exchange biased systems. The introduction of interfacial defects in the model, following Malozemoff's idea, generates uncompensated spins that can instead lead to a shifted loop with quantitatively reasonable values.

From an experimental point of view the magnetic behaviour of an FM-AFM interface has been characterized as a function of several parameters, such as FM and AFM layer thickness, temperature, different field cooling procedures, crystal orientation, influence of roughness (see Ref. [10] for a review). Yet several issues are still not completely understood. An example is the increase in coercive field, which has been comparatively less intensively studied than the exchange bias field, even if already early experiments recognized it as a typical feature of this interaction [23]. One of the reasons is that the coercivity is often also strongly related to the microstructure of the system under investigation. This is even more true in the case of thin ferromagnetic films. Despite of this, some systematic studies have been tried. For example higher order anisotropy [24] or interfacial magnetic frustration [25] have been claimed to account for the increase in coercive field. More in general the microscopic origin of the FM-AFM interaction is not yet completely clear. This is related to the interfacial nature of this interaction, which is usually justified by the fact that both the coercive field and H_{eb} , decrease linearly by increasing the FM layer thickness [6, 26]. It turns out that the interface conditions play a crucial role. These are actually not so easy to control. Especially in sputtered films, which are frequently used to investigate the FM-AFM interaction, roughness, crystallinity, and grain size may play a role [27]. On the other hand a technique would be needed to investigate the magnetic configuration at both sides of the interface (FM and AFM side) at the same time. In particular there is a lack of information about the spin configuration at the surface of an AFM material, and how this can be changed by interaction with an FM material. Just in recent years the observation of magnetic domains in AFM thin films has been reported [28, 29, 30], and there are indications that the magnetic properties of an AFM material may be modified by the contact with an FM material [31].

Each of the above mentioned theoretical models has had a partial experimental confirmation. A perpendicular coupling between the FM and the AFM axes of magnetization has for example been observed in Fe films grown on AFM FeF₂ single crystals [32], thus supporting Koon's idea. On the other hand a spiraling spin structure in an FeMn film sandwiched between permalloy and Co has been claimed [33], pointing therefore to the validity of the model of Mauri *et al.* Recently more attention has been actually brought to Malozemoff's approach. Uncompensated spins in an AFM material with a nominally compensated surface, induced by cooling under an applied field, have in fact been observed [34] with a temperature dependence consistent with the change of H_{eb} as a function of temperature. As it will be shown later in this work, uncompensated moments in an AFM material may also be induced just by the contact with an FM material, and this seems to be a general feature of FM-AFM interfaces [35, 36]. Moreover the finding that defects in the volume of the AFM material may induce the formation of domains in the antiferromagnet [37], has led to the formulation of a domain state model to explain the exchange bias effect [38]. The domains formed in the volume of the antiferromagnet carry a remanent surplus magnetization, which causes and controls exchange bias. These observations stress the importance of having domains in the AFM material and suggest them as a fundamental ingredient for the explanation of the magnetic behaviour of an FM-AFM interface.

1.4 The system under investigation: $Co/Fe_{50}Mn_{50}$ bilayers

 $Co/Fe_{50}Mn_{50}$ bilayers have been used in the present work in order to study the FM-AFM interaction. Experiments on this system are not so frequently encountered in literature [35, 39, 40], and always have been performed on structures obtained by sputtering techniques. Here instead the bilayers were grown by a molecular beam epitaxy technique, which ensures a better control of the interface conditions. Moreover the used samples are single crystalline thin films, as it will be demonstrated in chapter 3, which eliminates the complication of having grains of different crystalline orientation. In this way a structurally well characterized system can be obtained. This is indeed a prerequisite for a fundamental understanding of the magnetic coupling at the Co/Fe₅₀Mn₅₀ interface.

In bulk material $Fe_x Mn_{100-x}$ disordered alloys are antiferromagnetic with an fcc structure in a concentration range between x = 40 at.% and x = 80 at.% at room temperature [41, 42]. The Néel temperature varies with x and reaches a maximum of about 500 K at $x \approx 50$ at.%. The three energetically most likely spin structures for fcc γ bulk Fe_xMn_{100-x} alloy, identified by Umebayashi and Ishikawa [41], are shown in Fig. 1.4. For clarity in the figure the spin direction is displayed by arrows for just some atoms. In the so-called (001) spin structure (a) the spins are collinear along one axis, changing direction in successive planes. The $\langle 110 \rangle$ spin structure (b) realizes a net zero magnetization with a non-collinear spin arrangement: spins of different sublattices are located along two axes rotated by 90° in adjacent planes. Finally in the noncollinear (111) spin structure (c) the spins point along the (111) directions, with atoms on the $(0,0,0), (\frac{1}{2},\frac{1}{2},0), (0,\frac{1}{2},\frac{1}{2}), \text{ and } (\frac{1}{2},0,\frac{1}{2})$ positions, having moments directed to the center of the tetrahedron defined by these atomic positions. This last model has been at first hypothesized by Kouvel and Kasper as a possible spin arrangement in the AFM disordered FeNiMn alloy [43]. The neutron diffraction experiments of Umebayashi and Ishikawa [41] indicated the $\langle 111 \rangle$ structure as the most probable spin configuration. Consistent with this conclusion are also more recent Mössbauer experiments, where actually the two non-collinear (110) and (111) spin structures could not be distinguished [44]. At present no observations on possible modifications of this spin structure close to the surface or in an $Fe_x Mn_{100-x}$ alloy thin film are available. The $\langle 111 \rangle$ model for the spin structure has been recently confirmed by first principle calculations [45, 46] as the energetically most favourable. Actually other kinds of theories forecast, for the alloy in the equiatomic composition, even more complicated non-collinear spin structures [47] or the possibility that the substitutional disorder of the system could stabilize a collinear spin structure [48].

The relatively high T_N and the good crystallographic matching with the FM Ni₈₀Fe₂₀ alloy (Py) have made Py/Fe₅₀Mn₅₀ one of the most studied exchange biased systems [49, 50, 51]. This high interest is also connected to a technological point of view, because Py/Fe₅₀Mn₅₀ has been used until recently as the basis of many spin valve structures [8, 52, 53]. As a result this



Figure 1.4: Possible bulk $Fe_x Mn_{100-x}$ spin structures for x between 40 at.% and 80 at.%.

system has been fully characterized with respect to parameters for applications, for example the influence of the crystallographic orientation of the interface, AFM and FM thickness dependence, temperature dependence, and so on. Nevertheless the exact origin and the consequences of the FM-AFM interaction in this system are still under debate.

As it will become clear later, the technique used to image magnetic domains required to choose another material as FM layer in the system. One of the powerful capabilities of the mentioned technique is in fact its elemental selectivity. Therefore any possible ferromagnetic signal coming from the Fe in the $Fe_{50}Mn_{50}$ layer would be masked by the signal from the same element in Py. An ideal substitute of Py as FM material is Co. Even if usually less used in applications due to the better soft magnetic properties of Py, it does not present any disadvantage

for fundamental studies.

To preserve the AFM character of the $Fe_{50}Mn_{50}$ alloy, an fcc substrate is preferable. Since the structural, electronic and magnetic properties of Co/Cu(001) have been fully characterized [54], this system has the features fitting the present purposes. The lattice parameter of $Fe_{50}Mn_{50}$, a = 3.629 [42], is in fact fitting quite well with the one of Cu(001), a = 3.615 [55]. This lattice misfit of less than 1% should promote a pseudomorphic growth of $Fe_{50}Mn_{50}$ on Cu(001). The same holds also for the growth of $Fe_{50}Mn_{50}$ on Co/Cu(001), considering that Co acquires the same in-plane lattice parameter as Cu when deposited on it. Experimental evidence confirming these considerations will be given later in this work for both configurations, namely for Co/Fe₅₀Mn₅₀/Cu(001) and Fe₅₀Mn₅₀/Co/Cu(001) bilayers.

Chapter 2

Experimental aspects

The tools to characterize the structural and magnetic properties of the films are briefly described in the first section of this chapter. In particular Auger electron spectroscopy and electron diffraction techniques, namely low and medium energy electron diffraction, were used to characterize the structural and growth properties, while the magneto-optical Kerr effect was exploited to record magnetization curves. In section 2.2 it is explained how thickness and concentration of the Fe_xMn_{1-x} alloy films have been determined. The microscope used for magnetic domain imaging is then presented in section 2.3, with some emphasis on the way the images are practically obtained. This part contains three subsections, where the technical features of the microscope are briefly delineated at first, then the magnetic contrast mechanism, namely x-ray magnetic circular dichroism, is presented, and finally the way is described in which elemental images of magnetic domains are obtained. In order to investigate the thickness dependence of the domain patterns, micro-wedge-shaped samples were used. The method for growing these structures is described in the last section of the chapter.

The acronyms and abbreviations which are used are listed below:

UHV	ultra high vacuum
AES	Auger electron spectroscopy
ML	atomic monolayer
LEED	low energy electron diffraction
MEED	medium energy electron diffraction
MOKE	magneto-optical Kerr effect
PEEM	photoelectron emission microscope
XMCD	x-ray magnetic circular dichroism

2.1 Experimental techniques

The experiments were performed in a UHV chamber with a base pressure of about 1×10^{-8} Pa. The Cu substrate used for growing the films was a disk-shaped single crystal with the [100] direction normal to the surface. In order to obtain a clean and smooth surface the crystal was treated in the vacuum chamber by 1 keV Ar⁺ ion bombardment and subsequent heating up to about 800 K in 3 minutes, keeping then this temperature for 2 more minutes. The temperature was controlled by an N-type thermocouple attached to the sample holder. The AES [56] system, used to check the cleanliness of the substrate, comprises of a cylindrical mirror analyzer with an integral electron gun set to 3 keV energy during operation. A typical spectrum obtained from the clean Cu(001) is displayed in Fig. 2.1, where the three peaks of the *LMM* Auger transitions at 778 eV, 848 eV, and 922 eV are recognized. The presence of surface contaminants is below the detection limit of the Auger system ($\approx 2\%$ of a ML). The ordered surface structure has



Figure 2.1: AES spectrum from the clean Cu(001) substrate.

been checked by LEED [57], where a beam of electrons with primary energy up to $\approx 500 \text{ eV}$ is incident perpendicularly on the surface, and the elastically backscattered electrons produce a diffraction pattern imaged on a fluorescent screen. An example of the diffraction pattern of the Cu(001) clean substrate, recorded on a computer via a CCD camera, for 80 eV electron energy is displayed in Fig. 2.2. Besides the two-dimensional translational symmetry at the surface, from LEED one can gain information also about the average vertical interlayer distance. In fact, due



Figure 2.2: LEED pattern of the Cu(001) substrate for 80 eV electron energy.

to the interference of the electrons scattered by neighboring atoms along the direction normal to the surface, there exists a modulation of the LEED spot intensity as a function of the primary energy of electrons. Within the kinematic theory, based on the approximation that only single scattering processes occur, the vertical interlayer distance d can be expressed as

$$d = \frac{n\pi\hbar}{\sin\theta\sqrt{2m_e(E_p(n) - V_0)}}$$
(2.1)

where the integer n is the order of the corresponding interference peak, $E_p(n)$ the primary energy of the electrons of that peak, V_0 the additional energy shift due to the average inner potential in the crystal, m_e the electron mass, and θ the incident angle with respect to the sample surface. Using equation 2.1, with the experimental value $\theta = 85^{\circ}$, the average interlayer distance for the Cu(001) is $d = 1.81 \pm 0.02$ Å, in agreement with the bulk value [55].

The films studied in this work were grown on the clean substrate at room temperature by electron beam assisted thermal evaporation. The electron beam is focussed on the tip of the material held in a water-cooled evaporation source. Fe and Co were cut from high purity wires (99.99% purity) of 2 mm diameter and welded at one side to the body of the evaporation source. To evaporate Mn two ways were used: either a small irregular Mn piece, of dimension around 10 mm, held by a Ta wire, or a rod of 5 mm diameter fixed to the evaporator by a thin Ta foil wrapped around it. The second solution offered better results in term of stability of evaporation rate. In both cases the material had a purity of 99.99%. FeMn alloys were obtained by simultaneous evaporation of Fe and Mn from two different sources. During deposition the pressure in the chamber could be kept below 5×10^{-10} Pa.

The rate of deposition of the growing films, typically of 0.5-1 ML per minute, was checked

by counting the oscillations in the (00) spot MEED intensity recorded during evaporation. In the chamber set-up the AES system is mounted face-to-face to the LEED system. This allows diffraction experiments in a grazing incidence geometry with primary electron kinetic energy of 2 keV, using the electron gun of the AES system and the fluorescent screen of the LEED system. An example is shown in Fig. 2.3, where the growth of Fe on Cu(001) was monitored with MEED. The (00) spot intensity is plotted as a function of time at the bottom axis. The drop of the



Figure 2.3: MEED specular intensity oscillations recorded during the growth of Fe on Cu(001) as a function of time at the bottom axis. The thickness in ML is given at the upper axis.

intensity at time 0 corresponds to the opening of the shutter in front of the evaporation source. Besides some complicated characteristics of the growth of Fe on Cu(001), the intensity displays an oscillatory behaviour connected to the periodicity of the amount of roughness during the layer-by-layer growth of the film. By counting the oscillations one can extract the thickness of the film, as it is shown at the upper axis.

Magnetization curves have been measured by MOKE [58]. This technique is based on the fact that the polarization characteristics of a laser beam reflected from a sample depend on the magnetic state of the sample itself. The MOKE setup uses a UHV-compatible coil with a soft iron core driven by a computer controlled bipolar power supply. The maximum accessible field at the sample is about 200 Oe. The light from a He-Ne laser, operated at a wavelength of $\lambda = 633$ nm, passes through the bore of the coil impinging on the sample with an angle of $\approx 10^{\circ}$ from the sample surface. Before entering the coil, the laser beam is periodically modulated at

50 kHz between left and right circular polarization that allows a detection of the Kerr signal by a lock-in technique. The reflected beam is then analyzed by means of a polarization filter and an integrated photodiode/amplifier. An interference filter tuned to the wavelength of the laser covers the input aperture of the photodiode in order to reduce the influence of stray light. With this geometry one can measure the component of the magnetization parallel to the surface. Rotating the sample in a way that its rear side faces the coil, and using another laser beam incident at an angle of $\approx 70^{\circ}$ from the sample surface, also the component of the magnetization perpendicular to the sample surface could be measured.

2.2 Fe_xMn_{1-x} alloy composition

It has been shown in the previous section that from the oscillations in the MEED intensity it is possible to determine the evaporation rate of Fe (E_{Fe}) deposited on Cu(001). Also during the simultaneous evaporation of Fe and Mn, to obtain an Fe_xMn_{1-x} alloy, MEED oscillations are present, as will be discussed in the next chapter, giving the possibility to determine the evaporation rate (E_{FeMn}) of the alloy films. If Fe is evaporated at the same rate for deposition of the Fe film and of the Fe_xMn_{1-x} film, one can compare E_{Fe} and E_{FeMn} as determined by MEED. In this way one has already the possibility to calculate the composition in the Fe_xMn_{1-x} film, since x is obtained by the ratio of E_{Fe} and E_{FeMn} .

A complementary method to cross-check the composition is given by AES. In the general case of a film (F) growing on a substrate (S) in a layer-by-layer mode the Auger intensities of the substrate and the film of thickness d_F can be written as

$$I_S = I_0 S_S \exp^{-d_F/\Lambda_S} \tag{2.2}$$

$$I_F = I_0 S_F (1 - \exp^{-d_F/\Lambda_F}) \tag{2.3}$$

respectively, with the approximation that the attenuation of the Auger electrons within a material is determined by the mean distance the electrons can travel before an inelastic scattering event occurs. The subscripts S and F denote the substrate and the film, respectively. The quantities S_i (i = S, F) are the sensitivities of the Auger transition. The effective attenuation lengths $\Lambda_i = \lambda_i / \cos \alpha$, are correlated to the attenuation length of the Auger electron in the materials λ_i via the detection angle α of the Auger electron spectrometer. For the results discussed here $\alpha = 22.5^{\circ}$. Finally I_0 represents the intensity of the primary beam. From an Auger spectrum one can measure I_S and I_F , and determine the thickness of the film once the parameters S_i and λ_i are known.

In the case of a binary alloy the situation is more complicated, since now both the constituents of the alloy reduce the Auger signal from the substrate. Moreover, for Fe and Mn simultaneously growing on Cu, an additional complication arises because of energetic overlapping of some of the prominent peaks of the LMM transition. In Fig. 2.4 an Auger spectrum of a 6 ML Fe₅₀Mn₅₀ film grown on Cu(001) is shown. Thickness and concentration were determined by MEED. As



Figure 2.4: Auger spectrum from a 6 ML Fe₅₀Mn₅₀ film grown on Cu(001). I_{Mn} , I_{Fe} , and I_{Cu} are the Auger intensities for the three materials at 545 eV, 705 eV, and 922 eV, respectively.

one can see, from each of the three materials there is at least one Auger peak not overlapping with others. The only peaks not overlapping are marked by dashed lines at 545 eV, 705 eV, and 922 eV. The corresponding Auger electron intensities are defined as I_{Mn} , I_{Fe} , and I_{Cu} . Experimentally one can therefore obtain the ratio between the Cu and Mn (Fe) Auger intensities as $R_{Mn} = I_{Cu}/I_{Mn}$ ($R_{Fe} = I_{Cu}/I_{Fe}$), as a function of the thickness and the concentration of the alloy film.

In the case of $\text{Fe}_x \text{Mn}_{1-x}$ growing on Cu, the ratio between equations (2.2) and (2.3) can be written for Mn and for Fe respectively as

$$R_{Mn} = \frac{S_{Cu} \cdot \exp^{-d/\Lambda_{Cu}}}{S_{Mn} \cdot (1-x)(1-\exp^{-d/\Lambda_{Mn}})}$$
(2.4)

$$R_{Fe} = \frac{S_{Cu} \cdot \exp^{-d/\Lambda_{Cu}}}{S_{Fe} \cdot x \cdot (1 - \exp^{-d/\Lambda_{Fe}})}$$
(2.5)

where now d is the total film thickness in ML and x, the Fe concentration, can be statistically interpreted as the fraction of the substrate covered by Fe. To determine d and x from equations (2.4) and (2.5), the quantities S_i (i = Mn, Fe, and Cu) and Λ_i are needed. These can be obtained by graphically adjusting the theoretical curves (2.4) and (2.5) in a way to match the experimental values of R_{Mn} and R_{Fe} , whose corresponding thickness and concentration were determined from MEED oscillations. The result of this procedure is shown in Fig. 2.5, where the



Figure 2.5: Equations (2.4), dashed lines, and (2.5), full lines, for different values of R_{Mn} and R_{Fe} . The parameters S_i and Λ_i have been adjusted to match the experimental points, displayed as black dots.

black dots are the experimental points as determined by MEED. For each point an experimental value of R_{Mn} and R_{Fe} , as measured by AES, is given. Of course for pure Mn (Fe) just the parameter R_{Mn} (R_{Fe}) is measured. For the Mn experimental points the vertical error bar is bigger, since the thickness could not be determined directly by MEED oscillations. Equations (2.4) and (2.5) are displayed as dashed and full lines respectively, for different values of R_{Mn} and R_{Fe} as indicated at the upper and right axis respectively. By varying the values of S_i and Λ_i the lines were graphically adjusted in order to be consistent with the experimental points. In this way the parameters S_i and Λ_i can be used to solve the equations (2.4) and (2.5) as a function of d and x, and to determine thickness and concentration of the Fe_xMn_{1-x} alloy films on Cu with an error of 0.5 ML thickness and 5% concentration.

2.3 Imaging magnetic domains with PEEM

The use of PEEM to image magnetic structures [59] has been in recent times a quite broad field of research. In this technique the electrons emitted from a magnetic sample are used to obtain an image of magnetic domains. The various approaches differ by the primary excitation source and by the contrast mechanism. In the present work PEEM was used in connection with a synchrotron radiation source exploiting XMCD. In this way elemental selectivity is added to the surface sensitivity and the good lateral resolution of the microscope.

2.3.1 Basic features of the microscope

The schematic set-up of the PEEM used in these experiments [60] is shown in Fig. 2.6 [61, 62, 63]. The exciting light impinges on the sample at an angle of 30° from the surface. The sample stage forms an integral part with the electron optical column, to avoid any possible relative motion of sample and objective lenses, thus reducing the influence of vibrations on the image quality. A wobble-stick is used to transfer the sample between the manipulator, in which the films are deposited on the clean substrate, and the sample stage of the microscope, within the same UHV chamber. Once inserted in the sample stage, lateral positioning can be done by two orthogonal piezo-driven microslides. These permit to scan a range of approximately 5 mm \times 5 mm. The emitted low energy electrons (energy up to 10 eV), extracted by electrostatic objective lenses, pass through a contrast aperture, set for the present experiments to 150 μ m of diameter. This aperture determines the resolution of the instrument and the intensity of the image. The stigmator/deflector, located in the back focal plane of the objective, corrects non-spherical aberrations of the electron optic. In addition it may be used as an x/y deflector to shift the field of view without moving the sample. The size of the field of view can be limited by the iris aperture. The image is then magnified by a two-stage projective lens system, intensified by a microchannel plate, and converted into visible light by means of a scintillator crystal (YAG-Screen). For computer-recording a Peltier-cooled slow scan CCD camera is used.

The microscope can be operated in two imaging modes, namely survey mode and high resolution mode. The former works with extraction field around 120 V resulting in a field of view of 330 μ m in the used set-up. In the latter the microscope is operated with higher extraction voltage (typically 13 kV) obtaining a field of view of $\approx 90 \ \mu$ m. In this "zoom-in" mode, images of the sample topography can be obtained with a lateral resolution as good as 20 nm [64], which is the design limit set by chromatic and spherical aberrations.



Figure 2.6: Schematic set-up of the PEEM.

2.3.2 XMCD as a contrast mechanism

In order to obtain a lateral image of the domain configuration, the electrons, emitted from the sample and collected by the PEEM, must carry information about the magnetization state of the sample itself. This is obtained by exploiting the phenomenon of XMCD in photoabsorption [65]. Due to this effect the structure of a photoabsorption spectrum and the relative intensities of the spectral features vary upon changing the magnetization direction of the sample or the polarization state of the incident light. In this work the electronic transition from p to d states in the 3d metals Mn, Fe, and Co were used to obtain a magneto-dichroic signal, and hence a magnetic domain contrast. When circularly polarized light excites an electron from a p level, the excited electron will be spin polarized, due to the spin-orbit coupling in the core level.

The spin quantization axis is defined by the photon spin, and it will be mutually oppositely directed for right and left polarized light. The unoccupied states in the d band wherein the electron is excited is spin-splitted due to the exchange interaction. As a consequence there are more unoccupied minority spin states than majority spin states. The spin quantization axis is here given by the sample magnetization. This imbalance in the unoccupied states induces a different probability for the $p \rightarrow d$ transitions excited by oppositely circularly polarized light. The result is the XMCD spectrum shown in Fig. 2.7 (b). In Fig. 2.7 (a) two absorption spectra



Figure 2.7: (a) Intensity spectra around the L edges in Co for parallel (solid line) and antiparallel (dashed line) orientation of photon spin and sample magnetization; (b) XMCD signal obtained as the difference between the two spectra in (a). From Ref. [65].

are shown for parallel (solid line) and antiparallel orientation (dashed line) of photon spin and sample magnetization direction. These absorption spectra are usually obtained by measuring the secondary electron yield, the intensity of which is proportional to the absorption event. The same electrons are actually the ones which give rise to an image in PEEM, as will be discussed below. The difference between the two spectra, shown in (b), is the result of the different absorption intensity for opposite direction of photon spin or magnetization. The dichroic spectrum of Fig. 2.7 (b) has opposite sign at the L_2 ($2p_{1/2}$) and the L_3 ($2p_{3/2}$) edges, due to the opposite sign of the spin polarization of the electrons exited from these two spin-orbit split levels.

The magnetic dichroism is then transferred to the low-energy electron imaged by the PEEM in a two step mechanism. At first the hole created by the photoexcitation of the 2p electron

decays by an Auger process. At second the Auger electron on its way through the solid suffer inelastic scattering events, generating a cascade of low-energy electrons. This secondary electron yield carries finally the magneto-dichroic signal into the microscope. The escape depth is determined by the cascading process of the scattered Auger electrons and is material dependent. For example it has been demonstrated that magnetic dichroism, measured by total secondary electron yield, could be detected from a 300 Å Co film covered by a polymer cover layer as thick as 100 Å [66]. For the 3*d*-metals a typical detection length of 20 Å has been reported [67]. This rather high sampling depth, together with the elemental selectivity introduced via the photoexcitation step, make PEEM used with XMCD a unique instrument to access the magnetic state of buried layers.

The way in which the magnetic information is obtained influences the lateral resolution. This is mainly determined by the energy distribution of the collected low energy secondary electrons, which can be as large as 10 eV. With a proper choice of the size of the contrast aperture and the setting of the electrostatic fields in the microscope a typical lateral resolution obtained in the experiments presented here was 400 nm.

As pointed out before, XMCD in photoabsorption depends on the mutual orientation of excitation light and sample magnetization. In particular it is proportional to the scalar product of these two vectorial quantities. The same holds also for the magnetic signal in PEEM, that appears as bright/dark contrast for domains with a magnetization axis parallel or antiparallel to the light direction.

2.3.3 How to get magnetic domain images

The results presented in this thesis were obtained at the helical undulator beamline UE56-2 PGM2 of the synchrotron radiation facility BESSY II in Berlin [68, 69]. Typically the primary beam energy was set to the L_3 absorption edge of the desired material, and two images of the same area of the sample were obtained for opposite light helicity. As shown in the previous subsections the two images will have locally different intensity according to the magnetization direction in the sample. In Fig. 2.8 an example is given for a 6 ML Co film grown on Cu. The intensity images obtained at the L_3 edge for positive and negative helicity are labeled as $I_{L_3}(+)$ and $I_{L_3}(-)$, respectively. The light is coming from the top to the bottom of the figure, as shown by the arrow labeled $h\nu$ (in the figure the projection of the incoming x-rays direction on the sample surface is actually shown). Each image was exposed for a total time of 4 minutes with a 2×2 binning of camera pixels. The field of view is approximately 400×400 pixels² as displayed at the left and bottom axes of the images. This corresponds to an area of approximately 90×90 μ m² of the sample surface, as calibrated by imaging with the same parameters of the microscope structures of known dimensions. The micrometer scale is also displayed in the images at the right and top axes. Due to the properties of XMCD, the magnetic signal is obtained by subtracting



Figure 2.8: The asymmetry of two images obtained at the L_3 adsorption edge of Co for opposite helicity $I_{L_3}(+)$ and $I_{L_3}(-)$ results in an image with a strong magnetic contrast (Asy). Azimuth crystallographic axes and in-plane projection of incoming x-rays $(h\nu)$ are indicated.

the two images, whereas it is eliminated by summing then up. To obtain the magnetic contrast and to eliminate topological and illumination effects, the asymmetry (Asy) is calculated, which is defined as the difference of the images normalized to the sum:

$$Asy = \frac{I_{L_3}(+) - I_{L_3}(-)}{I_{L_3}(+) + I_{L_3}(-)}$$
(2.6)

The calculated asymmetry is shown on the bottom of Fig. 2.8, where magnetic domains are evident as different grey tones. A positive value of the asymmetry leads to a bright domain,

while a negative value to a dark one. To obtain quantitative information from the value of the asymmetry, and thus the direction of the magnetization in the different domains, the intensity at the edge was subtracted for each helicity to the intensity a few eV before the edge (pre-edge intensity). In this way the influence of the background is eliminated. By comparing images taken with different azimuth orientations, one can therefore determine the vectorial direction of the magnetization in the different domains. In the "Asy" image of Fig. 2.8 this is displayed by the black and white arrows printed in some domains. The in-plane crystallographic orientation of the sample with respect to the incoming x-rays direction is shown on the left of the Asy image. The [100] in plane direction was rotated by an angle $\theta = 7^{\circ}$ from the direction of the four easy directions of Co along the $\langle 110 \rangle$ axes. The maximum contrast obtained in Co, that is the algebric sum of the asymmetry of a domain magnetized along the incoming x-rays direction and a one opposite to it, was ≈ 32 %.

2.4 Double wedge deposition

The study of the thickness dependence of magnetic properties of thin films is better achieved by using wedge-shaped samples. This avoids the time consuming procedure of preparing different samples with different thicknesses. Micro-wedge-shaped samples were therefore prepared to be imaged by PEEM. To this aim an aperture of $2 \times 0.5 \text{ mm}^2$ was placed in front of the sample, with a distance of 1 mm to the sample surface [70]. During the evaporation of the films the sample was then rocked about the long axis of the aperture by typically $\pm 10^{\circ}$. In this way two wedge regions were obtained separated by a plateau of uniform thickness. The slope and width of the wedges are determined by the maximum evaporated thickness and the value of the rocking angle. Typical values for the width of the wedge were $\approx 200 \ \mu m$. By using successively two orthogonal apertures, crossed double wedge films were obtained. This was achieved as graphically explained in Fig. 2.9, where for displaying purposes the drawings are not to scale. At Step 1 "aperture 0" is placed in front of the sample and the first film is evaporated keeping the azimuth orientation of the sample/aperture at $\varphi = 0^{\circ}$, and rocking the whole around an axis perpendicular to the hook of the sample holder, as graphically depicted in the figure. After taking out "aperture 0", in Step 2 "aperture 90" is inserted. The sample/aperture is then rotated at an azimuth angle $\varphi = 90^{\circ}$ and the second evaporation takes place, rocking the sample/aperture about an axis parallel to the hook of the sample holder. Step 3 shows the completed sample in (a). The schematic three-dimensional picture of the Cu substrate with the two crossing double wedges (b) makes more clear how the final sample looks like.

The evaporation rates were calibrated before the preparation of the wedges for continous films deposited in identical conditions by counting MEED oscillations recorded during the growth. From the total evaporation time during a wedged film deposition one therefore knows the maxi-



Figure 2.9: Schematic representation of the evaporation of crossed double wedge samples.

mum thickness (plateau thickness). The thickness at each position on the wedge is then obtained by element selectively taking the ratio between the intensity at the L_3 absorption edge and the pre-edge intensity. This ratio displays the slope of the wedge. The accuracy of the film thickness obtained in this way is estimated as ≈ 10 %.

Chapter 3

Structural and magnetic characterization of Co/FeMn bilayers

Using the techniques introduced in the previous chapter, Co/FeMn bilayers have been studied, and the experimental results are presented here. In section 3.1 the growth of $Fe_x Mn_{100-x}$ alloy films on Cu(001) and on Co/Cu(001) has been characterized at first by recording the MEED intensity during film deposition. Oscillations in the MEED intensity, observed for a broad range of alloy film composition, indicate a layer-by-layer growth mode. The structure of $Co/Fe_{50}Mn_{50}$ bilayers has then been studied (section 3.2) by MEED and LEED in order to check the epitaxy of the system on Cu(001). Section 3.3 describes how magnetization curves, measured by MOKE, allow to obtain information on the magnetic transition in $Fe_{50}Mn_{50}$ films between a paramagnetic and an antiferromagnetic state. This transition is confirmed by the Co domain patterns obtained by PEEM from the bilayers in the as-grown state, presented in section 3.4. Exploiting the elemental selectivity of PEEM, domain images from Fe and Mn were obtained, as shown in section 3.5. This small ferromagnetic signal in Fe and Mn could be detected when the $Fe_{50}Mn_{50}$ alloy film was in contact with ferromagnetic Co. The consequences of thermal treatments on the domain patterns are then studied in section 3.6, and finally the influence of the application of external fields on the Co domain configuration is the subject of section 3.7.

3.1 Growth of Fe_xMn_{100-x} alloy films

As it has been shown in the previous chapter the presence of oscillations in the MEED intensity during the growth of Fe on Cu(001) allows determining the thickness of the deposited film. This is not the case for the evaporation at room temperature of Mn on Cu(001) as it is displayed in Fig. 3.1. In the figure the (00) spot MEED intensity is shown as a function of the evaporation



Figure 3.1: MEED intensity recorded during the growth of Mn on Cu(001).

time at the bottom axis, and the thickness in ML at the upper axis. The time has been scaled in a way that the "0" corresponds to the opening of the shutter in front of the evaporation source. The thickness has been determined after evaporation using AES, as explained in the previous chapter. The closing of the shutter is indicated by the arrow. One can see that at the opening of the shutter (time 0) the intensity drops down. This is a common feature of all the MEED intensity curves presented in this work and it is due to the initial increase of density of surface steps. Apart from a small shoulder at a thickness slightly lower than 1 ML, the intensity decreases up to a thickness of ≈ 3 ML, staying later almost constant.

During the simultaneous evaporation of Fe and Mn on Cu(001) and on Co/Cu(001), oscillations in the MEED intensity are observable. This is shown in Fig. 3.2 for a wide range of concentration between $x \approx 30$ at.% to x = 95 at.% for Fe_xMn_{100-x} films on Cu(001) (a) and on 6 ML Co/Cu(001) (b). In the figure the MEED intensity is displayed as a function of the FeMn thickness in ML. Each curve corresponds to a different film with a different concentration as indicated. The arrows present in some curves indicate the closing of the shutter in front of the evaporators. Apart from the Fe₂₅Mn₇₅ film on Cu(001), in all other cases the intensity presents an oscillatory behaviour related to the periodic variation of the average number of surface defects [71, 72]. This in turn is due to the layer-by-layer growth mode of the Fe_xMn_{100-x} alloy films on


Figure 3.2: MEED intensity recorded during the growth of $\text{Fe}_x \text{Mn}_{100-x}$ alloy films on Cu(001) (a) and on 6 ML Co/Cu(001) (b).

Cu(001) and on 6 ML Co/Cu(001), at least for the concentration range in which the oscillations are present. As pointed out in the previous chapter the periodicity of the oscillations gives a precise way to determine the film thickness in ML units.

3.2 Epitaxial $Co/Fe_{50}Mn_{50}$ bilayers on Cu(001)

From the results of the previous section, the pseudomorphic layer-by-layer growth mode of the Fe_xMn_{100-x} alloy films, in particular at the equiatomic composition, can be deduced. This is true when the $Fe_{50}Mn_{50}$ film is deposited on Cu(001) and on a thin Co layer on Cu(001). On the other hand from the presence of MEED oscillations during the growth of Co on Cu(001) it has been concluded that a thin Co film also grows in a layer-by-layer mode on Cu(001) [73]. More generally the same conclusion can be drawn for both the bilayers $Fe_{50}Mn_{50}/Co$ and $Co/Fe_{50}Mn_{50}$ growing on Cu(001) as it is demonstrated in Fig 3.3. In the upper part of the figure the MEED intensity as a function of time is shown for the growth of 6 ML Co on top on Cu(001), and the subsequent evaporation of 8 ML $Fe_{50}Mn_{50}$ on top of it. The order of evaporation is reversed in the lower part of the figure where the MEED intensity as a function of time is displayed for the growth of the 13 ML Co/8 ML $Fe_{50}Mn_{50}$ bilayer on Cu (001). In both cases the stop of the double-evaporation is indicated by the "shutter closed" arrow. Distinct oscillations are present



Figure 3.3: MEED intensity recorded during the growth of 8 ML $Fe_{50}Mn_{50}/6$ ML Co (top) and 13 ML Co/8 ML $Fe_{50}Mn_{50}$ (bottom) bilayers on Cu(001).

for all the four films evaporated.

To obtain further information about the crystallographic structure, LEED experiments have been performed. It is in particular important, as it will be pointed out in the next chapter, to check if the $Fe_{50}Mn_{50}$ film keeps the fcc structure also in the thin film regime. Selected LEED patterns are presented in Figs. 3.4 and 3.5. Fig. 3.4 shows the LEED pattern of the clean Cu substrate at 125 eV electron energy (a) and of a 26 ML $Fe_{50}Mn_{50}$ film on Cu(001) at 115 eV (b). The primary energy of the electrons is reported in every image. The LEED patterns appear to be quite similar for both cases. This indicates that the $Fe_{50}Mn_{50}$ film forms a p(1×1) overlayer with the substrate lattice, at least up to the maximum investigated thickness of 26 ML. A similar



Figure 3.4: LEED patterns from the clean Cu(001) substrate (a), and a 26 ML thick $Fe_{50}Mn_{50}$ film on Cu(001) (b). The primary energy of the electrons is indicated.



Figure 3.5: LEED patterns from the clean Cu(001) substrate (a), 6 ML Co/Cu(001) (b), 0.5 ML Fe₅₀Mn₅₀/6 ML Co/Cu(001) (c), and 2 ML Fe₅₀Mn₅₀/6 ML Co/Cu(001) (d). The primary energy of the electrons is indicated.

behaviour is expected also for $Fe_{50}Mn_{50}$ films growing on a Co/Cu(001) substrate, considering that Co acquires the same in-plane lattice parameter as Cu when deposited on top of it. In Fig. 3.5 the first stage of growth of an $Fe_{50}Mn_{50}$ film on 6 ML Co/Cu(001) is investigated. In particular LEED patterns are shown for the Cu(001) substrate (a), 6 ML Co/Cu(001) (b), 0.5 ML $Fe_{50}Mn_{50}/6$ ML Co/Cu(001) (c), and 2 ML $Fe_{50}Mn_{50}/6$ ML Co/Cu(001) (d). Again in all images a very similar diffraction pattern can be seen. As stated above, a thin Co film forms a $p(1\times1)$ overlayer on Cu(001), as one can see by comparing Figs. 3.5 (a) and (b). Moreover, one realizes that in the diffraction patterns of the initial stages of growth of $Fe_{50}Mn_{50}$ on Co/Cu(001), see Figs. 3.5 (c) and (d), no superstructures are formed, as it could be in the case of alloying with the underlying layer.

Besides the lateral arrangement of the atoms, from LEED one can also extract information about the vertical average interlayer distance. This has been obtained, as briefly explained in the previous chapter, by recording the intensity of the (00) diffracted spot as a function of the primary electron energy. The so called I(E) curves are reported in Fig. 3.6 for several $Fe_{50}Mn_{50}$ films grown on Cu(001), upper part of the figure, and on 6 ML Co/Cu(001), lower part. The



Figure 3.6: Intensity versus energy dependence of the LEED (00) beam for $Fe_{50}Mn_{50}$ films grown on Cu(001) (top) and on 6 ML Co/Cu(001) (bottom).

label at each curve indicates the layer thicknesses. The composition of the $Fe_{50}Mn_{50}$ films is omitted in the labels for brevity. For comparison also the I(E) curves for the Cu substrate and the 6 ML Co/Cu(001) are shown. The integer *n* labels the order of the single scattering interference Bragg maximum. The intensity of the maxima is somehow lower for the films than for the Cu(001) substrate, but the peaks are visible up to the maximum thickness reported. Within the kinematic theory, using equations (2.1), from the I(E) curves one can calculate the average value of the interlayer distance near the surface. The result is shown in Fig. 3.7 as a function of the Fe₅₀Mn₅₀ film thickness expressed in ML. In the figure the horizontal dashed line



Figure 3.7: Vertical interlayer distance as a function of $Fe_{50}Mn_{50}$ thickness, for films grown on Cu(001) (squares), and on 6 ML Co/Cu(001) (circles).

at 1.808 Å indicates the vertical interlayer distance in bulk Cu(001) [55], and the dotted line at 1.74 Å the vertical interlayer distance for a 6 ML Co on Cu(001) [73], as taken from literature. When grown on Cu(001), the Co lattice is in fact slightly tetragonally compressed because of the lattice mismatch between the fcc Co and Cu unit cells. The open square and the solid circle indicate the experimental values for the Cu(001) substrate and the 6 ML Co/Cu(001) film respectively, as indicated by the labels. The solid squares are the vertical interlayer distances for $Fe_{50}Mn_{50}$ films grown on Cu(001). One can see that within the experimental error all the points agree with the value of fcc Cu. The open circles are the values of interlayer distance for $Fe_{50}Mn_{50}$ films grown on 6 ML Co/Cu(001). The two points for low $Fe_{50}Mn_{50}$ thickness, at 1 and 2 ML, are still quite close to the value of the Co film. This is most likely due to the fact that what is measured here is an *average* value. For low $Fe_{50}Mn_{50}$ thickness the contribution of the Co film is still quite big. For higher $Fe_{50}Mn_{50}$ thickness the vertical interlayer distance is again identical to the one of the fcc Cu(001) substrate. The above results from MEED and LEED are therefore pointing to the conclusion of an epitaxial grown of the $Co/Fe_{50}Mn_{50}$ bilayers on Cu(001). Moreover the fcc crystalline structure of the substrate is preserved also in the grown bilayers.

3.3 Coercivity and hysteresis loops

As mentioned in the first chapter of this thesis two typical consequences of the interaction between a ferromagnetic (FM) material and an antiferromagnetic (AFM) material are a displaced hysteresis loop and an increased coercive field. In order to measure these two quantities, hysteresis loops have been obtained by using MOKE with the external applied field kept along the [110] azimuth direction of the substrate. This is the easy axis of magnetization of a thin Co film grown on Cu(001) [74, 75]. As pointed out in the first chapter of this thesis in bulk material an $Fe_{50}Mn_{50}$ alloy is an antiferromagnet with a $T_N \approx 500$ K [42]. Thin $Fe_{50}Mn_{50}$ films deposited on Cu(001), measured in this work, do not show any ferromagnetic signal at room temperature both in the longitudinal and in the polar MOKE geometry, up to an $Fe_{50}Mn_{50}$ thickness $t_{FeMn} = 20$ ML. This is in agreement with the not-FM state of the alloy also on the thin film region. To probe the AFM ordering, the change in the magnetic properties of an adjacent Co film has been measured. In a typical experiment, hysteresis loops were recorded at room temperature upon increasing t_{FeMn} by subsequent evaporation steps on top of 6 ML Co/Cu(001). From the hysteresis loops the coercivity (H_c) and the remanent magnetization of the Kerr signal (M_r) were measured. The result is shown in Fig. 3.8 where the open squares are M_r points (left axis) and the solid circles are values of H_c (right axis) as a function of t_{FeMn} . The lines are guides to the eyes. The inset displays for comparison the hysteresis loops (Kerr



Figure 3.8: Remanence M_r (left axis) and coercive field H_c (right axis) as a function of Fe₅₀Mn₅₀ film thickness. The inset displays the hysteresis loops for $t_{FeMn} = 0$ ML and $t_{FeMn} = 12$ ML.

intensity versus external applied field) for $t_{FeMn} = 0$, that is for the 6 ML Co/Cu(001) film, and

 $t_{FeMn} = 12 \text{ ML Fe}_{50}\text{Mn}_{50}$ on top of 6 ML Co/Cu(001). The pronounced increase of H_c starting at $t_{FeMn} \approx 10 \text{ ML}$ ($\approx 20 \text{ times from } t_{FeMn} = 9.5 \text{ ML}$ to $t_{FeMn} = 12 \text{ ML}$) is the indication that AFM ordering in the Fe}₅₀Mn₅₀ film is establishing at that thickness, i.e., Fe}₅₀Mn₅₀ films thicker than 10 ML are AFM at room temperature. Comparable results have also been obtained upon reversing the order of deposition, when the Co film is deposited on top of the Fe}₅₀Mn₅₀ film. Due to the interaction between the antiferromagnet and the Co film, the coercive field of the bilayer is strongly increased compared to that of the pure FM film. The increase in H_c due to the AFM ordering of Fe}₅₀Mn₅₀ has been measured at a similar thickness also for Fe}₅₀Mn₅₀/Ni₈₀Fe₂₀ bilayers on Cu(001) [78].

The variation of M_r in the displayed thickness range shows a quite complicated behaviour. A very similar thickness dependence has been found for the Kerr rotation or the Kerr intensity during the deposition of metallic non-magnetic overlayers on top of magnetic films [76, 77]. In those cases an oscillatory behaviour superimposed to a slow decrease as a function of the overlayer thickness was recognized. The oscillations were attributed to spin-polarized quantum well states in the metallic overlayers, which influence the magneto-optic properties of the system. Due to the likeness in the experimental observation, a similar conclusion could be attempted also for the present case of $Fe_{50}Mn_{50}$ on Co. One can furthermore notice that in Fig. 3.8 right at the increase of H_c a correlate decrease of M_r is observed, which may be related to the antiferromagnetism in the Fe₅₀Mn₅₀ layer. Indeed, as it will be shown later, a change in easy axis from the $\langle 110 \rangle$ to the (100) azimuth directions is observed in a Co film coupled to an Fe₅₀Mn₅₀ film thicker than 10 ML. In Fig. 3.8 a reduction in M_r of approximately a factor $1/\sqrt{2}$ from $t_{FeMn} = 9.5$ ML to $t_{FeMn} = 12$ ML can be seen. This can be explained by considering that the external field, applied along one of the $\langle 110 \rangle$ directions, is not able to fully saturate in this direction the magnetic film, which now has an easy magnetization axis along one of the $\langle 100 \rangle$ directions. In this case the measured remanence is just the projection of the value along one of the $\langle 100 \rangle$ directions on one of the $\langle 110 \rangle$ directions.

From the increase in coercive field one can measure the thickness dependence of the temperature T_{AFM} at which the AFM ordering disappears. Due to finite size scaling [79] one would expect in general a decrease of this temperature for decreasing film thickness. For thick enough films T_{AFM} should approach T_N , the uniquely defined value of the bulk material. Fig. 3.9 shows the temperature dependence of the coercive field of a bilayer of Fe₅₀Mn₅₀ films of three different thicknesses (9, 11, and 13 ML) deposited on top of 6 ML Co/Cu(001). The values of H_c have been obtained from the hysteresis loops recorded upon increasing the temperature. For displaying purpose they have been normalized to the value at low temperature for each film thickness. The lines superimposed to the experimental points in Fig. 3.9 are guides to the eyes. In all three curves one can see a step-like decrease of H_c upon increasing temperature. This corresponds to the crossing of T_{AFM} at that thickness. The ordering temperature is shifting towards higher values for higher t_{FeMn} . The coercive field above the ordering temperature for



Figure 3.9: Normalized coercive field as a function of temperature for t_{FeMn} Fe₅₀Mn₅₀/6 ML Co/Cu(001), with $t_{FeMn} = 9$, 11, and 13 ML.

all the films is very similar to the coercive field of pure 6 ML Co/Cu(001) at room temperature. From these experiments one can obtain an experimental measure of T_{AFM} . This is done, as graphically explained for the data set of $t_{FeMn} = 11$ ML, by taking the intersect of the tangents in the right side of the step-like curve. The obtained values of T_{AFM} are plotted in Fig. 3.10 in a three-dimensional way as a function of t_{FeMn} and of the Fe concentration x in the Fe_xMn_{100-x} alloy films. Here also values of T_{AFM} for films with concentration different than equiatomic composition are added. As it was already clear from Fig. 3.9, the ordering temperature is increasing upon increasing film thickness at a fixed concentration, as one realizes by looking at the points connected by the black dashed line. Moreover one can also see that the concentration has some influence on the value of T_{AFM} . When reducing the Fe concentration the ordering temperature is in fact increasing. This is for example easily seen for the three data points at fixed thickness of $t_{FeMn} = 11$ ML connected by the black dotted line.

As explained in the first chapter, the FM-AFM interaction is an interface effect. The whole Co film contributes to the Zeeman energy from the applied field. If the coercivity of the Co/Fe₅₀Mn₅₀ is caused by pinning at the interface, for $t_{FeMn} > 10$ ML, it should therefore decrease when increasing the FM layer thickness. In order to prove this statement for the measured enhancement of the coercive field, Co films of increasing thickness were deposited on top of 12.5 ML Fe₅₀Mn₅₀/Cu(001). Hysteresis loops were recorded at room temperature at each step of Co evaporation, and the measured values of H_c are displayed in Fig. 3.11 as a function of Co thickness (t_{Co}). The Fe₅₀Mn₅₀ film thickness was chosen in a way to have the alloy in an AFM state, as concluded by the experiments described in Fig. 3.8. From Fig. 3.11 one can see



Figure 3.10: T_{AFM} as a function of film thickness (t_{FeMn}) and Fe concentration (x) in Fe_xMn_{100-x} alloy films.



Figure 3.11: Variation of coercive field as a function of Co thickness for t_{Co} Co/12.5 ML Fe₅₀Mn₅₀/Cu(001) films.

that the coercive field is monotonically decreasing upon increasing t_{Co} . When one tries to fit the data with a function of the type $H_c = H_{c,\infty} + bt_{Co}^{-c}$, the exact value of the parameter c depends on the constraint one applies to the parameter $H_{c,\infty}$. If $H_{c,\infty}$ has the physical constraint to be positive, one obtains a best fit with $c = 1.1 \pm 0.2$. This curve fit is also displayed in the figure

by the dotted line. Therefore one approximately obtains a linear dependence of the coercive field on the inverse of the Co thickness, which is an indication of the interfacial origin of the measured effect.

At the beginning of this section a hysteresis loop with an enhanced coercive field has been emphasized as resulting from the FM-AFM interaction. In order to obtain a hysteresis loop shifted by H_{eb} along the applied field axis, a pinning direction must be set. This is usually obtained by growing the AFM material with an applied field, or by cooling through the Néel temperature under an applied field. The samples studied in the present work displayed typically low values of H_{eb} compared to the values of H_c . An example is shown in Fig. 3.12. Here a 15 ML



Figure 3.12: Hysteresis loops from a 15 ML Co/20 ML $Fe_{50}Mn_{50}/Cu(001)$ sample for the two opposite field cooling directions measured at 280 K. In the inset an hysteresis loop from the same sample measured at 255 K is displayed.

Co/20 ML Fe₅₀Mn₅₀/Cu(001) bilayer was evaporated at room temperature and then annealed at ≈ 420 K. During cooling down to 230 K a field of 1 kOe supplied by a permanent magnet was applied along the [110] azimuth direction. The positive direction of the field of the permanent magnet coincides with the positive direction of the field applied during recording the MOKE hysteresis loops. The hysteresis loop displayed in the figure by the dotted line has been then recorded at 280 K after field cooling in the negative direction. The loop results shifted towards positive values of external field as a usual exchange biased loop, termed "negative exchange bias" (see Fig. 1.1). By repeating the field cooling procedure upon reversing the field of the permanent magnet, the hysteresis loop, recorded again at 280 K, is shifted towards the negative direction. This is displayed by the full line loop. In this second loop the left and right coercive fields are indicated as H_{c1} and H_{c2} , respectively. The exchange bias and coercive fields measured from the loop are around 15 Oe and 70 Oe, respectively. By decreasing the temperature both quantities are increasing as one can see in the inset of Fig. 3.12, where a hysteresis loop measured at 255 K after field cooling in the positive direction is shown. Actually this loop is already not completely saturated for negative values of applied field with the maximum available field of 200 Oe.

3.4 Co domain configuration in the as-grown state

In the previous section it has been deduced that the transition from paramagnetic (PM) to AFM in thin $Fe_{50}Mn_{50}$ films is thickness dependent. When a Co film is in contact with an $Fe_{50}Mn_{50}$ film of varying thickness, one could expect that the Co domain configuration presented some differences for coupling to the PM or AFM state of the $Fe_{50}Mn_{50}$ film. This is indeed the case as shown in Fig. 3.13. In (a) the domain configuration of Co in a Co/Fe₅₀Mn₅₀/Cu(001) sample



Figure 3.13: (a) Co domain configuration in a Co/Fe₅₀Mn₅₀/Cu(001) double wedge sample; (b) enlarged view of the region around the transition at $t_{FeMn} \approx$ 10 ML. Thicknesses are indicated at the axes. Incident x-rays direction ($h\nu$) and crystallographic axes are reported in the rectangular box below the images.

is shown. As explained in the previous chapter this has been obtained with the PEEM from the asymmetry of the Co L_3 absorption edge. Co and Fe₅₀Mn₅₀ films were grown as crossed double wedges. In the imaged area the Fe₅₀Mn₅₀ thickness increases from bottom to top as indicated at the left axis, and the Co thickness from left to right, as indicated at the bottom axis. In the rectangular box the crystallographic axes are indicated and the direction of the incoming x-rays $(h\nu)$ is displayed by the big arrow at 52° from the [100] axis. Note that in this and in the next PEEM images the direction of the incoming x-rays is already the projection on the

plane of the sample surface. When Co becomes FM at room temperature for a thickness bigger than $t_{Co} \approx 2$ ML, it presents distinct domain patterns as a function of Fe₅₀Mn₅₀ film thickness. For $t_{FeMn} < 10$ ML two big domains are visible. The magnetization direction, displayed by the arrows inside the domains, is along (110) azimuth directions. The values of the asymmetry in the domains are in fact the same as in the not shown region where Co is just in contact with the Cu(001) substrate. As mentioned previously, a thin Co film grown on Cu(001) has a four-fold anisotropy along the (110) azimuth directions [74, 75]. For $t_{FeMn} > 10$ ML the features of the Co domain image are drastically changed. Now the domains are very small, close to the limit of the instrumental resolution of 400 nm. In Fig. 3.13 (b) an enlarged view of the region around the transition at $t_{FeMn} \approx 10$ ML is displayed. Here the Fe₅₀Mn₅₀ thickness is indicated at the right axis, increasing from bottom to top, and the Co thickness again at the bottom axis. One can see that at $t_{FeMn} \approx 10$ ML suddenly in the Co film small domains are present that are getting even smaller for increasing t_{FeMn} , until they are not any more resolved above approximately 13 ML. This change in Co domain configuration happens at the same $Fe_{50}Mn_{50}$ thickness at which the coercive field starts to increase, as reported in the previous section. Both observations are therefore attributed to the same phenomenon, that is the interaction of Co with antiferromagnetically ordered Fe₅₀Mn₅₀ film.

A different situation can be expected when first Co is grown on Cu(001), and then the $Fe_{50}Mn_{50}$ film on top of it. In this case in fact the Co film is not forced to acquire a specific domain pattern induced by the underlayer, which is now non magnetic. Yet the interaction with the AFM overlayer induces a modification in the Co magnetic properties, as one can see in Fig. 3.14. Here an $Fe_{50}Mn_{50}/Co/Cu(001)$ double wedge sample was imaged for two different directions of light incidence, obtained after rotation of the sample by 90° abount the surface normal. Inside the rectangular box the direction of the incoming x-rays, together with the crystallographic axes, are shown. Here the $h\nu$ (a) arrow indicates the incoming x-rays direction for image (a), while the $h\nu$ (b) arrow is the incoming x-rays direction for image (b). The $Fe_{50}Mn_{50}$ and Co thicknesses are indicated at the left and bottom axes of images (a) and (b), respectively. Looking at image (a), one notices that for $t_{FeMn} < 10$ ML a domain configuration comparable to the case of Co grown on top of $Fe_{50}Mn_{50}$ is present, i.e., big domains oriented along the $\langle 110 \rangle$ azimuth directions. At the PM-AFM transition of the Fe₅₀Mn₅₀ overlayer (at $t_{FeMn} > 10$ ML), no small domains are present in the Co film, but now a change in grey scale contrast is visible. The change in grey scale for $t_{FeMn} > 10$ ML is consistent with a magnetization directed along the [100] direction, as displayed by the arrow. The local magnetization directions can be determined by comparing asymmetry values of image (a) with the corresponding domains in image (b), after sample rotation. For example the dark grey domain in (a), with magnetization oriented along the [110] direction, corresponds to the black domain in (b). Its shape is slightly different in the two images, due to some thermally activated domain wall movement during the time elapsed between the recording of (a) and (b). As stated above, the grey scale in every



Figure 3.14: (a) Co domain configuration in a $Fe_{50}Mn_{50}/Co/Cu(001)$ sample and (b) after a 90° sample rotation. In (c) and (d) histograms of the asymmetry values in the different domains in (a) and (b), respectively, are shown.

domain in (b) is changed with respect to image (a) according to the different projection onto the light incident direction. By comparing the values of asymmetry in the two measurements one confirms the magnetization directions as displayed by the arrows in the domains. The asymmetry values in each domain are extracted by means of histograms of the domain images. These are displayed in (c) for image (a), and in (d) for image (b). Each peak in the histogram has already been labeled with the respective magnetization direction. The asymmetry values have actually to be corrected by a possible shift in the zero value. The amount of this shift can be fixed by knowing the maximum contrast of a typical Co film. This value, dependent on the actual setting of the PEEM and the beam line, has been determined to be about 32%. In this way the asymmetry values are 0.5%, 12.5%, and 16% for the $[1\overline{1}0]$, [100], and [110] domains in image (a), and -16%, -10%, and 0.5% for the same domains in (b). Since these values are just the in-plane projection through an angle θ of the magnetization on the incoming x-rays direction, geometric considerations give the searched values of the angle θ . The error in determining θ , and from it the magnetization direction, can be estimated to be about $\pm 2^{\circ}$. These results are valid on the hypothesis that the magnetization is always in the film plane, as it should be for a Co film of the considered thickness. As a result of the two independent measurements one obtains therefore that for $t_{FeMn} > 10$ ML the magnetization is indeed oriented along the [100] azimuth direction. This implies the surprising conclusion that the interaction of a thin Co film

with an AFM $Fe_{50}Mn_{50}$ film induces a change in the Co easy axes of magnetization from the $\langle 110 \rangle$ crystallographic directions to the $\langle 100 \rangle$ crystallographic directions.

The deposition of an Fe₅₀Mn₅₀ film on top of Co seems to induce this change in the Co easy of magnetization, for t_{FeMn} thicker than a certain value, without modifying the Co domain shape. This can be seen in Fig. 3.15, where the Co domain configuration is shown for a Fe₅₀Mn₅₀/Co



Figure 3.15: Co domain configuration in a Fe₅₀Mn₅₀/Co/Cu(001) sample. t_{FeMn} and t_{Co} are indicated at the left and bottom axes of the image, respectively. The crystallographic axes and the direction of the incoming x-rays ($h\nu$) are shown. The arrows in some domains display the magnetization direction.

double wedge deposited on Cu(001). The image was taken at room temperature in the asgrown state. t_{FeMn} and t_{Co} are indicated at the left axis, increasing from bottom to top, and at the bottom axis, increasing from left to right, respectively. The crystallographic axes are indicated at the right hand side of the figure. The in-plane direction of the incoming x-rays $(h\nu)$, depicted by the big arrow, has an angle of 24° from the [100] direction. The arrows in the domains show the magnetization direction, calculated by considering the projection of the asymmetry value on the direction of the incoming x-rays. Four different grey scale contrasts are visible, corresponding to two of the $\langle 110 \rangle$ directions for $t_{FeMn} < 10$ ML, and two of the $\langle 100 \rangle$ directions for $t_{FeMn} > 10$ ML. A change in magnetization direction in the Co film, occurring at $t_{FeMn} \approx 10$ ML, is therefore again present. One can in particular notice that a domain wall, running almost vertical in the image, is present in both thickness regions. This would indicate that the deposition of Fe₅₀Mn₅₀ on the Co film has not changed the domain pattern of Co. The Co domains in contact with an Fe₅₀Mn₅₀ film thicker than 10 ML have just changed their magnetization direction.

The change in Co easy axis of magnetization presented in the previous two figures has not been observed for thicker Co film. In particular a 20 ML Co film covered by an Fe₅₀Mn₅₀ film in the AFM state, shows an in-plane easy magnetization axis again along the $\langle 110 \rangle$ azimuth directions. This further magnetic easy axis rotation has not been investigated in details, but there are indications that it takes place around 15 ML. In particular it is not clear at present by which mechanism the natural Co magnetic easy axis is restored. Two scenarios may exist: either the easy axis of magnetization in Co rotates continuously from the $\langle 100 \rangle$ to the $\langle 110 \rangle$ azimuth directions when increasing Co thickness, or there is a coexistence of easy directions in different sample areas at the same thickness.

3.5 Ferromagnetic signal from Fe and Mn

As explained in the previous chapter, one of the unique features of the PEEM is its elemental selectivity and the possibility to access buried layers. Besides the Co domain configuration, presented in the previous section, one is therefore tempted to search for a magnetic signal coming from Fe and Mn. In principle one would not necessarily expect to succeed in this task by using the PEEM as it has been exploited in this work, because the working principle involves magnetic circular dichroism that is sensitive to *ferromagnetic* order only. From the MOKE experiments on single $Fe_{50}Mn_{50}$ alloy films on Cu(001) no magnetic signal was obtained for the studied thickness range, and correspondingly also no magnetic domain images could be detected by the PEEM for these films.

The situation is different when the alloy is in contact with a ferromagnetic Co film, that is for $t_{Co} > 2$ ML. In this case Fe and Mn were found in a partially ferromagnetic state, as it is shown in Fig. 3.16. The three images show the domain patterns of Co, Fe, and Mn from the same area of a $Co/Fe_{50}Mn_{50}/Cu(001)$ sample. The elemental selectivity has been obtained, as explained in the previous chapter, by excitation with radiation tuned to the L_3 edge of the respective element. The Co and $Fe_{50}Mn_{50}$ films were grown as crossed double wedges. The Co thickness of the imaged area, indicated at the bottom axis, increases from left to right. The $Fe_{50}Mn_{50}$ thickness, increasing from bottom to top, is indicated for all three images at the left axis of the Co image. Below the images, inside the rectangular box, the direction of the crystallographic axes is shown. The big arrow at 52° from the [100] direction displays the direction of the incoming x-rays $(h\nu)$. In the Co image one can see a behavior similar to the one described in Fig. 3.13. The Co film forms small domains for $t_{FeMn} > 10$ ML. For $t_{FeMn} < 10$ the domains are much bigger and oriented along two of the $\langle 110 \rangle$ azimuth directions. In the Fe and Mn images the same domain pattern can be recognized however with a lower contrast. In particular the typical maximum contrast in Fe and Mn is approximately just 5% of the contrast in Co. The total time for acquisition of the Co and Fe images was around 10 minutes each,



Figure 3.16: Co, Fe, and Mn domain patterns of the same area of a $Co/Fe_{50}Mn_{50}/Cu(001)$ sample. Co and $Fe_{50}Mn_{50}$ thicknesses are indicated at the left and bottom axes of the images, respectively. The box below the images contains the direction of the incoming x-rays $(h\nu)$ and the crystallographic axes.

while it was twice that time for Mn. The resulting different statistic is the reason why the Mn image appears sharper than the Fe one.

A small ferromagnetic signal has been also obtained from Fe and Mn for the reversed order of deposition, when the $Fe_{50}Mn_{50}$ film is deposited on top of Co/Cu(001). This is shown in Fig. 3.17 where the same area of a $Fe_{50}Mn_{50}/Co/Cu(001)$ sample is imaged by tuning the x-ray photon energy to the Co, Fe, and Mn L_3 adsorption edges. The thicknesses are indicated at the left axes of the images for the $Fe_{50}Mn_{50}$ film, increasing from bottom to top, and at the bottom axis of the Mn image for the Co film, increasing from left to right. The rectangular box below the images contains the indication of the crystallographic axes and the incoming x-rays direction $(h\nu)$. The Co image is qualitatively comparable with the one shown in Fig. 3.14. For $t_{FeMn} > 10$ ML the magnetization direction, indicated by the arrows in the domains, is rotated from the $\langle 110 \rangle$ crystallographic axes to the $\langle 100 \rangle$ crystallographic axes. A small ferromagnetic signal is again detectable also for Fe, as displayed by the domain pattern of the Fe image. From this domain pattern one would conclude that Fe is coupled ferromagnetically to Co. Interestingly the contrast is reversed in the Mn image. For example the black domain in Co, oriented along the [010] azimuth direction, becomes bright in the Mn image. The most likely conclusion is therefore that Mn has a small net ferromagnetic moment, which is coupled antiferromagnetically to Co and Fe. In order to prove these considerations, an asymmetry scan along a line indicated in each image has been taken. The asymmetry values of the line scans are displayed in Fig. 3.17



Figure 3.17: On the left column Co, Fe, and Mn domain patterns of the same area of a Fe₅₀Mn₅₀/Co/Cu(001) sample are shown. Thicknesses are indicated at the images axes. The direction of the incoming x-rays $(h\nu)$ together with the crystallographic axes is contained in the box below the images. In the right column the result of scans, along the black line printed in each image in the left column, is reported.

on the right hand side of the corresponding image. The pixel scale at the right axis of each line scan graph is the same as for the corresponding domain image. The top axis of the Co

scan graph indicates the asymmetry scale, intended to serve also for the Fe scan graph. For the Mn scan graph the asymmetry is indicated at the bottom axis. In the Co scan one can see three levels of asymmetry, marked by the three vertical lines. These correspond to the three domains crossed by the line scan in the Co image. The scans in the Fe and Mn images have been multiplied by the reported factor in order to be in the same scale as the Co one. One can see that the three asymmetry levels in the Fe and Mn scans are spaced exactly as in the Co scan. This demonstrates that while Fe is coupled ferromagnetically to Co, having the same magnetization orientation in the different domains, Mn is antiferromagnetically coupled to Co and Fe. Moreover, one can realize from the Fe and Mn images of Fig. 3.17 that a ferromagnetic signal is detectable for both $t_{FeMn} < 10$ ML and $t_{FeMn} > 10$ ML. It means that whatever the magnetic state of the Fe₅₀Mn₅₀ alloy, PM or AFM, when in contact with a Co film it acquires an induced ferromagnetic moment. This was not so clear from the images of Fig. 3.16. The small domains present there for $t_{FeMn} > 10$ ML are not fully resolved for Fe and Mn. Therefore one could be tempted to think that no magnetic moment is induced in Fe and Mn when the Fe₅₀Mn₅₀ film is in an AFM state. This is proven to be not true by the images in Fig. 3.17.

Besides the induced magnetic moments, another effect present in some of the Mn images is shown in Fig. 3.18. The Co (a) and Mn (b) images printed in the figure were acquired from the same area of a $Co/Fe_{50}Mn_{50}/Cu(001)$ sample. The thicknesses of the two films, grown as crossed double wedges, are indicated at the left axis for the $Fe_{50}Mn_{50}$ film, increasing from bottom to top, and at the bottom axis for the Co one, increasing from left to right. After deposition of the bilayer, the sample was annealed in the preparation chamber up to ≈ 420 K and then cooled down under an external applied field of 200 Oe. At room temperature the field was at first reduced to 0, and subsequently a pulse of -30 Oe was applied. In both cases the sample was aligned in a way that the field, supplied by the coil usually used for MOKE, had its positive direction along the [110] axis. The positive direction of the field is indicated as a grey arrow (+H) in the rectangular box between the images, along with the crystallographic axes and the direction of the incoming x-rays $(h\nu)$. The arrows in the domains of the Co image display the magnetization direction. One can see that in most part of the Co film for $t_{FeMn} < 10$ ML the magnetization is along the $[\bar{1}\bar{1}0]$ azimuth direction (black domain), that is the direction imposed by the small field applied at room temperature. This field was in fact able to reverse the magnetization here except in the region of lower Co thickness, and it had no influence on the Co film coupled to the Fe₅₀Mn₅₀ film for $t_{FeMn} > 10$ ML. This reflects the high coercive field, as measured by MOKE, in that $Fe_{50}Mn_{50}$ thickness region. One can further notice that for $t_{FeMn} > 10$ ML the Co film is still oriented along one of the (100) directions. The Mn image displays the same domain pattern as the Co image, from the beginning of the $Fe_{50}Mn_{50}$ wedge at $t_{FeMn} = 0$. Superimposed to the magnetic domain contrast, some stripes of alternating contrast are also visible. The stripes are perpendicular to the gradient of total thickness t, that is to the sum of the increasing Co and $Fe_{50}Mn_{50}$ films thicknesses. Some values of t are indicated at one



Figure 3.18: Co (a) and Mn (b) domain patterns of the same area of a Co/FeMn/Cu(001) sample. Fe₅₀Mn₅₀ and Co thicknesses are indicated at the left and bottom axes, respectively. Crystallographic axes, and incoming x-rays ($h\nu$) and applied field (H) directions are displayed in the rectangular box between the images. In (c) the result of a scan along the long axis of the rectangle printed in (b), averaged on the asymmetry values of the short axis of the rectangle, is shown.

of the long axes of the rectangle printed in (b). The result of a line scan along the long axis of the rectangle, averaging the asymmetry values across the width of the rectangle, is shown in (c), where the asymmetry is plotted as a function of the total films thickness t. The oscillations correspond to the alternating contrast in the stripes. One sees that the period of the oscillations is 1 ML in the total thickness. A close look to image (b) in fact indicates that the period of the stripes is 1 ML in both the vertical (Fe₅₀Mn₅₀ thickness) and the horizontal (Co thickness) directions.

It is worth to mention that the observation of these stripes is quite sensitive to the preparation conditions. In general the intensity of the stripes has been found to be higher when the alloy wedge film is deposited on top of Co, especially when the Mn concentration is bigger than 50% in the Fe_xMn_{100-x} film. Moreover the time elapsed between the recording of the images and the preparation of the sample could induce a further increase in the stripes intensity.

3.6 Thermal treatments

As it has been shown in Fig. 3.13, a Co film that grows on an AFM $Fe_{50}Mn_{50}$ film presents small domains due to the magnetic interaction of the underlayer. These domains, frozen-in during the growth of the Co film, are metastable. By annealing at a temperature higher than the AFM ordering temperature they have in fact the possibility to rearrange in a way to minimize the energy configuration. This is for example shown in Fig. 3.19. Here Co, Fe, and Mn are imaged



Figure 3.19: Co, Fe, and Mn domain patterns, recorded at room temperature, of the same area of a Co/Fe₅₀Mn₅₀/Cu(001) sample annealed at ≈ 420 K before imaging. t_{FeMn} and t_{Co} are indicated at the left and bottom axes, respectively. Crystallographic axes and incoming x-rays direction $(h\nu)$ are found in the rectangular box below the images.

at room temperature from the same area of a Co/Fe₅₀Mn₅₀/Cu(001) sample annealed at ≈ 420 K after deposition and then transferred to the PEEM chamber. The Fe₅₀Mn₅₀ thickness is indicated at the left axis of the Co image, increasing from bottom to top, and the Co film thickness at the bottom axes of all the three images, increasing from left to right. The rectangular box below the images contains the indication of the crystallographic axes and of the direction of the incoming x-rays ($h\nu$). In the Co image, for $t_{FeMn} > 10$ ML, the small domains characteristic of the as-grown sample have disappeared. The annealing process creates bigger domains, which, once cooled down the sample to room temperature, are oriented along the $\langle 100 \rangle$ azimuth directions, as displayed in the figure by the arrows in the domains. For $t_{FeMn} < 10$ ML the magnetization is again found along one of the $\langle 110 \rangle$ azimuth directions. The result depends on the temperature at which the sample is annealed after film deposition. From Fig. 3.10 one can see that a 13 ML Fe₅₀Mn₅₀ film looses the AFM order for a temperature higher than 390 K. Therefore the Co film imaged in Fig. 3.19, at the annealing temperature of ≈ 420 K, was not any more in contact with an AFM material. On the other hand at this temperature the Co film is still FM, at least for $t_{Co} > 2.5$ ML [80]. In order to minimize the total magnetic energy, the small domains originally present in the as-grown state (see Fig. 3.13) merge together into the bigger domains present in Fig. 3.19. Each domain wall adds in fact an energy term to the total magnetic energy and at ≈ 420 K they are not anymore forced to exist by the interaction with the AFM underlayer. Upon cooling down one crosses T_{AFM} of the Fe₅₀Mn₅₀ film from above. The domains formed in Co do not change drastically their shape during this process, but, once at room temperature a change in the magnetic easy axis is observed. This change in easy axis of the Co magnetization is the same as observed for a Co film covered by an AFM Fe₅₀Mn₅₀ film, shown in Fig. 3.14. In the Fe and Mn images of Fig. 3.19 one can see an identical domain pattern as in the Co image. This ferromagnetic moment in the Fe₅₀Mn₅₀ alloy is again induced by the contact with the Co film. In particular here, one realizes that it is also present in the AFM part of the Fe₅₀Mn₅₀ alloy when the Co film is deposited on it.

The evolution of the Co domains during an annealing process from the as-grown state can be directly imaged by the PEEM, as it is presented in Fig. 3.20. Here a sample consisting of 6 ML



Figure 3.20: Co domain evolution imaged in a 6 ML Co/Fe₅₀Mn₅₀/Cu(001) sample during annealing and subsequent cooling. t_{FeMn} is indicated at the left axis of some images. Incoming x-rays direction $(h\nu)$ and crystallographic axes are indicated inside the rectangular box at the top left corner of the figure.

Co deposited on a wedge shaped $Fe_{50}Mn_{50}$ film on Cu(001) is imaged in the as-grown state in (a).

By annealing the sample directly in the PEEM the domain pattern in the same sample area is then recorded at different temperatures in images (b) to (g). The $Fe_{50}Mn_{50}$ thickness, indicated at the left axes of (a) and (d), increases from bottom to top of each image. The rectangular box on the upper left side of the figure contains the information about the crystallographic axes and the incoming x-rays direction $(h\nu)$. The only shortcoming here is that the exact value of the temperature is not known, since the sample stage of the PEEM is not equipped by a thermocouple. The value of temperature reported in the upper right corner of each image has been deduced by the results of the MOKE experiments as reported in Fig. 3.10. The sample in the as-grown state (a) presents the usual domain pattern for Co grown on $Fe_{50}Mn_{50}$ (see for example Fig. 3.13). At $t_{FeMn} < 10$ ML a single domain oriented along the [110] direction is present. For $t_{FeMn} > 10$ ML the Co film is characterized by small domains, which appear to reduce their size by increasing $Fe_{50}Mn_{50}$ thickness. Upon increasing the temperature at 400 K (b) bigger domains are formed at lower $Fe_{50}Mn_{50}$ thickness, while the small domains are still present at higher thickness. Since the Co domain pattern reflects the magnetization state of the underlayer, one can deduce that now the $Fe_{50}Mn_{50}$ alloy is AFM for higher t_{FeMn} than in image (a). In the region of small domains, Co is still coupled to AFM $Fe_{50}Mn_{50}$ while in the region where the domains are now bigger, $Fe_{50}Mn_{50}$ is in a PM state. Actually the $Fe_{50}Mn_{50}$ thickness at which this transition occurs is now less sharp than in the as-grown state. On the other hand, by comparing the domain sizes in (a) and (b), one would conclude that in (b) the $Fe_{50}Mn_{50}$ film is AFM for thicknesses bigger than ≈ 13 ML. At 430 K (c) the transition line between small and big domains is further moved towards higher Fe₅₀Mn₅₀ thicknesses ($t_{FeMn} \approx 16$ ML). In the region of big domains one can now recognize three different grey scales that correspond to three different magnetization directions along the $\langle 110 \rangle$ axes, as indicated by the arrows in the domains. The transition line is further moved towards higher t_{FeMn} upon increasing the temperature to 460 K (d), until at 480 K in almost all of the imaged area just big domains are present (e). The magnetization direction is now in all domains along $\langle 110 \rangle$ axes. By reducing the temperature to 430 K (f) the domain pattern is almost unchanged compared with image (e). Yet some differences are present in the grey scale contrast of the domains. Above the white dashed line the dark domains are less dark and the bright domains less bright than below the same line. This change in grey scale is consistent with the magnetization being directed along the (100) axes for Fe₅₀Mn₅₀ thicknesses bigger than the white dashed line. This change is comparable with what has been observed in figures 3.14 and 3.19. Moreover image (f) can be compared with image (c): the change in easy axis of magnetization and the presence of small domains before annealing, respectively, are indications of having the $Fe_{50}Mn_{50}$ film in an AFM state. When the sample is finally imaged again at room temperature (g) also the domains below the dashed white line of (f) have now the magnetization directed along the $\langle 100 \rangle$ axes. One can further notice that the domains directed along the $[\bar{1}10]$ azimuth direction in image (f), rotate their magnetization in image (g) either along the [010] or the $[\overline{100}]$ direction. The change in easy axis, with respect to a Co film grown on Cu(001), is therefore again evident for the Co film coupled to an $Fe_{50}Mn_{50}$ film thicker than 10 ML. Just for the lowest $Fe_{50}Mn_{50}$ thickness the magnetization is still directed along one of the $\langle 110 \rangle$ axes as it is in image (a).

Once the bigger domains in the Co layer are formed, the easy axis change observed when comparing images (e) and (g) in Fig. 3.20 is reversible, as it can be tested by repetitive heating and cooling. This is shown in Fig. 3.21, where the Co domain configuration at room temperature



Figure 3.21: Co domain configuration at room temperature in a 9 ML Co/ t_{FeMn} Fe₅₀Mn₅₀/Cu(001) sample in the as-grown state (a), after a first annealing at \approx 500 K (b), and after a second annealing at \approx 500 K (c). All three images are taken at room temperature. In the rectangular box the direction of the incoming x-rays ($h\nu$) and the crystallographic axes are indicated. t_{FeMn} is indicated at the left axis of some of the images.

in a 9 ML Co/ t_{FeMn} Fe₅₀Mn₅₀/Cu(001) sample is imaged in the as grown state (a), after a first annealing at ≈ 500 K (b), and after a second annealing at the same temperature (c). t_{FeMn} is indicated at the left axes of images (a) and (c). In the rectangular box the crystallographic axes are indicated by the small arrows, together with the direction of the incoming x-rays displayed by the fat arrow ($h\nu$) at 52° from the [010] azimuth direction. Arrows in some domains indicate the direction of the magnetization. In Fig. 3.21 (a) the usual domain pattern consisting of small domains is shown for the Co film grown on AFM Fe₅₀Mn₅₀. After annealing at ≈ 500 K and successive cooling down to room temperature the same area of the sample is imaged in (b). This image is similar to Fig. 3.20 (g): For $t_{FeMn} > 10$ ML big domains oriented along the $\langle 100 \rangle$ axes are now present in place of the small domains. The same situation is encountered after a second annealing at ≈ 500 K (c). With respect to Fig. 3.21 (b), one notices a domain coarsening, which leads to a further annihilation of small features. Apart from this, Fig. 3.21 (b) and Fig. 3.21 (c) lead to the identical observation that the easy axis of magnetization is along the $\langle 100 \rangle$ axes for Co coupled to the AFM Fe₅₀Mn₅₀ film.

3.7 Response to an external applied field

As explained in the previous chapter the local magnetic information is carried to the PEEM by low energy secondary electrons. Due to their low energy the trajectories of these electrons are quite sensitive to a magnetic field. Therefore the image one obtains during the application of an external magnetic field is too much distorted. Nevertheless it is still possible to study the influence of a magnetic field on a domain configuration by recording the image after the external field has been applied. In the experiments presented here, a pulsed magnetic field parallel to the sample surface was obtained by discharging a capacitor through a core-less solenoid placed close to the sample stage of the PEEM. The measurements were carried out always at zero field.

In section 3.3 a steep raise in the coercive field of $Co/Fe_{50}Mn_{50}$ bilayers has been measured when the $Fe_{50}Mn_{50}$ films exceeds ≈ 10 ML. At the same $Fe_{50}Mn_{50}$ thickness an abrupt change in the Co domain configuration has been imaged by the PEEM. By recording images after application of an external field one can fully correlated these two observations. The result of this experiment is shown in Fig. 3.22. A $Co/Fe_{50}Mn_{50}$ crossed double wedge sample deposited on Cu(001) is imaged in the as grown state in (a), and after application of field pulses in (b) to (d). The $Fe_{50}Mn_{50}$ thickness increases from bottom to top in the images as indicated at the left axes, and the Co thickness from left to right as indicated at the bottom axes. In the rectangular box on the left hand side the crystallographic axes and the direction of the incoming x-rays $(h\nu)$ are indicated. The grey double arrow displays the positive (+H) and the negative (-H)directions of the applied field. In image (a) one can notice the characteristic domain pattern for Co grown on an $Fe_{50}Mn_{50}$ wedge, as described for example in connection with Fig. 3.13. The same sample area as in (a) has been imaged in (b) after the application of a -15 Oe field pulse. For $t_{FeMn} < 10$ ML the magnetization has been rotated from the [110] direction to the $[\overline{110}]$ direction. The region of the Co film coupled to $Fe_{50}Mn_{50}$ thicker than 10 ML remains unaffected by the field pulse. This reflects the behaviour of the coercive field as measured by MOKE in these two $Fe_{50}Mn_{50}$ thickness regions. The situation is reversible, as demonstrated in image (c). Here a field pulse of +15 Oe has been applied before recording the image, and one can see that the magnetization at $t_{FeMn} < 10$ ML has reverted back to the same direction as in image (a). Pulsed magnetic fields up to a maximum of 250 Oe have then been applied before recording image (d). The domain configuration has just changed for some domains at $Fe_{50}Mn_{50}$ thicknesses between 10 and 13 ML. Where now the bright area is present in place of the small domains the magnetization could be directed along the [010] azimuth direction. Actually, since



Figure 3.22: Change in the Co domain configuration in a Co/Fe₅₀Mn₅₀/Cu(001) sample by application of an external field. Thicknesses are indicated at the axes of the images. Field (*H*) and incoming x-rays ($h\nu$) directions, together with the crystallographic axes, are indicated in the box at the left of the images.

the area is so small, the statistics is not good enough to drawn definite conclusions. One can furthermore notice that the changes in image (d) with respect to image (c) are located at the higher Co thickness side. This is due to the Co thickness dependence of the coercive field as reported in Fig. 3.11. Moreover it is a hint on the decrease in coercive field upon increasing t_{Co} for a Co thickness region that was not included in Fig. 3.11.

Since the maximum available external field at the PEEM sample stage is around 300 Oe, Co films thicker than the one in Fig. 3.22 must be used to further study the dependence on an applied field. As just mentioned, by increasing the Co thickness the coercivity of the Co/Fe₅₀Mn₅₀ bilayer decreases, and one has a chance to change the domain configuration of Co coupled to thicker Fe₅₀Mn₅₀ films, as it is reported in Fig. 3.23. An Fe₅₀Mn₅₀/Co double wedge has been deposited on top of Cu(001) with thickness up to 20 ML Fe₅₀Mn₅₀ and 15 ML Co. The Fe₅₀Mn₅₀ thickness is indicated at the left axes of images (a) and (c), increasing from bottom to top, and the Co thickness at the bottom axes of images (c) and (d), increasing from left to right. All four images display the same sample area. The usual indication about crystallographic axes, and incoming x-rays ($h\nu$) and external magnetic field (H) directions are given in the rectangular box at the left hand side of the figure. The sample in the as-grown state, imaged in (a), displays a big grey



Figure 3.23: Dependence of the Co domain pattern on Fe₅₀Mn₅₀ film thickness upon application of an external field in an Fe₅₀Mn₅₀/Co double wedge sample on Cu(001). Thicknesses are indicated at the axes. The rectangular box on the left contains the incoming x-rays ($h\nu$) and the external applied field (H) directions, together with the crystallographic axes.

domain for $t_{FeMn} < 10$ ML with magnetization oriented along the [110] direction. For Fe₅₀Mn₅₀ thicknesses bigger than 10 ML, a dark region on the left of the image and a bright region on the right are present. Both of them are not single domain, as for example observed in Fig. 3.14, but showing areas of different grey scale. This observation can be interpreted by considering that in the present case domains oriented along different $\langle 100 \rangle$ easy axes are present in each of the two regions. On the other hand the Co film thickness is here maybe already big enough that the natural Co anisotropy favors an easy magnetization direction along the $\langle 110 \rangle$ axes, as discussed in section 3.4. Following this second hypothesis the different grey scale in each of the two regions could be ascribed to a coexistence of both easy axes of magnetization. A definitive decision between the two interpretations is not possible at present. After a field pulse of -110 Oe the same area of the sample as in (a) is imaged in (b). The grey domain at the bottom of the image has reversed its magnetization into the [110] direction. Some changes in the domain configuration are also visible for 10 ML < $t_{FeMn} < 14$ ML. Here in fact the previously bright region has reversed into the dark region. This dark region extends to higher Fe₅₀Mn₅₀ thickness after a field of -140 Oe (c). The white dashed line here is the average maximum position of the

dark region in the formerly bright region in image (b). The domain configuration is therefore reversed from bright to dark approximately along a line perpendicular to the Fe₅₀Mn₅₀ slope. A pulse of -275 Oe (d) further pushes the dark region up to higher t_{FeMn} . Again the white dashed line here indicates the average Fe₅₀Mn₅₀ thickness value of the dark region in (c). This behavior can be explained by considering that the coercive field of the Co/FeMn bilayers depends of the Fe₅₀Mn₅₀ thickness, even in the thickness range presented in Fig. 3.23. Therefore the dark region moves up towards higher Fe₅₀Mn₅₀ thicknesses along the Fe₅₀Mn₅₀ slope. The horizontal value that it can reach is the Fe₅₀Mn₅₀ thickness at which the applied field pulse equals the coercivity.

The area of the sample displayed in Fig. 3.23 is also particularly well suited to investigate the presence of a local exchange bias effect. The bright and dark region in image (a) were in fact most likely oppositely magnetized domains before the deposition of $Fe_{50}Mn_{50}$ on top of the Co film. Since each of these two domains was saturated in an opposite direction, the AFM $Fe_{50}Mn_{50}$ film grown on them acquires an opposite orientation of the exchange bias in the two domains. For analogy one could think of an experiment in which an antiferromagnet is grown in an external field in order to set the so-called *bias direction*. In the present case the field is supplied by the Co magnetization, and the bias direction is opposite in the two domains. Locally, that is in each of the two domains, an opposite asymmetric behavior upon applying the external field can therefore be expected. This is what can be called local exchange bias effect. In Fig. 3.24 (a) the image of the sample in the as-grown state is again reported, identical to Fig. 3.23 (a). The crystallographic axes, incoming x-rays and field directions are the same as in Fig. 3.23. In images (b)–(f) of Fig. 3.24 a contour plot of the domain pattern of image (a) is superimposed, displayed by white lines. The contrast of the contour plot has been set in a way that just the difference between the dark and bright regions can be recognized. The grey domain for $t_{FeMn} < 10$ ML is therefore ignored since it does not contribute to any local exchange bias. Considerations done in the previous sections lead in fact to the conclusion that, for that thickness range, the $Fe_{50}Mn_{50}$ film is not AFM at room temperature. A field of -330Oe is at first applied. Most part of the sample area, Fig. 3.24 (b), is now dark. A field of +110Oe (c) reverts the bottom part of the image to bright. In particular one can see, with the help of the contour plot of the as-grown state, that the bright region has extended to higher $Fe_{50}Mn_{50}$ thickness in the right side of the image than in the left side. This is consistent with the domain pattern in image (a). Due to local exchange bias the bright region can be more easily reversed where it was already present in the as-grown state. After a pulse of +330 Oe (d) the opposite domain configuration as in (b) is obtained: now the imaged sample area is almost totally bright. The subsequent field of -110 Oe (e) leads to a configuration symmetric compared with the one obtained in (c). Now it is the dark domain that extends to higher $Fe_{50}Mn_{50}$ thickness on the left side of the image, where the dark region was present in the as-grown state. Actually the effect is here less clear than in (c). In order to confirm the local exchange bias, the sum of images (c) and (e) has been calculated. The result is shown in image (f). Since a dark contrast corresponds



Figure 3.24: Observation of local exchange bias effect in a Co film coupled to an $Fe_{50}Mn_{50}$ film.

to negative values of the asymmetry and a bright contrast to positive ones, the image has an intermediate grey scale contrast (values of asymmetry around 0) for those areas that exhibit opposite contrast in images (c) and (e). The upper part of image (f) is bright on the right side and dark on the left side. These are the areas unchanged with respect to the as-grown state, because the coercivity is higher than 330 Oe. The interesting area is the one enclosed by the black rectangular box where a kind of horizontal stripe is visible. This stripe is mostly dark on the left of the contour plot line, and mostly bright on the right side. The result is therefore consistent with the bias directions imposed by the dark and bright regions of the as-grown state, and the present effect can be explained as arising from a local exchange bias.

Chapter 4

Discussion

This last part of the thesis is devoted to the discussion of the experimental results described in the previous chapter. In section 4.1 some comments are made on the possible conditions which favour the epitaxial growth of the $\text{Fe}_x \text{Mn}_{100-x}$ alloy films on Cu(001) and on Co/Cu(001). In the alloy at equiatomic concentration the fcc structure of the bulk is kept, and so the antiferromagnetic γ structure of FeMn should also be stabilized in thin films. The magnetic properties of a thin Co film coupled to an $\text{Fe}_x \text{Mn}_{100-x}$ film, as measured in the MOKE experiments, give indications also on the properties of the AFM material. In particular the temperature at which the AFM order is established in the thin films could be estimated and compared with the Néel temperature of the bulk material.

In section 4.2 the domain pattern of a Co film grown on a wedge-shaped $Fe_{50}Mn_{50}$ film is discussed. For a certain $Fe_{50}Mn_{50}$ thickness the Co presents small domains due to the interaction with an AFM ordered underlayer. In this region the Co film replicates the domain pattern of the AFM $Fe_{50}Mn_{50}$ film. Possible configurations of these domains are presented and their influence on the establishment of the exchange bias phenomenon is discussed. These domains are also responsible for the change in the Co easy axis (section 4.3), observed when a thin Co film is in direct contact with an AFM $Fe_{50}Mn_{50}$ film. Possible mechanisms leading to the rotation of the Co easy axis of magnetization from the $\langle 110 \rangle$ to the $\langle 100 \rangle$ in-plane directions are presented.

Finally section 4.4 is devoted to the discussion of the magnetic domain patterns imaged at the Fe and Mn edges in the $Fe_{50}Mn_{50}$ alloy. This observation is explained as induced interfacial moments due to the direct contact with the FM Co film. Part of these "extra" spins could be responsible for the pinning of the FM layer in an exchange biased system. Some comments are then made on the induced moments in Mn, which were observed either parallel or antiparallel to the Co and Fe magnetization, depending on the preparation conditions. This behaviour can be explained by considering that the magnetism of the Mn atoms is very sensitive to the local microstructural and magnetic environment in which they are embedded.

4.1 Properties of $Fe_x Mn_{100-x}$ thin films

 $Fe_x Mn_{100-x}$ thin films have been shown in the previous chapter to present MEED oscillations when growing on Cu(001) and on Co/Cu(001), in a range between $x \approx 30$ at.% to x = 100 at.% at room temperature. This is an indication that the alloy films are growing in a pseudomorphic layer-by-layer mode. This forced epitaxial growth mode is favoured by the small lattice misfit (f) between the Cu(001) substrate and the $Fe_x Mn_{100-x}$ alloy. The lattice misfit is defined by the expression

$$f = \frac{a-s}{s} \tag{4.1}$$

where a and s are the lattice parameters of the growing film and the substrate, respectively.

In Fig. 4.1 the lattice misfit between Cu and $\text{Fe}_x \text{Mn}_{100-x}$ bulk alloy is shown as a function of the Fe concentration x. The line connecting the points is a guide to the eyes. The lattice



Figure 4.1: Lattice misfit (f) between Cu and $\operatorname{Fe}_{x}\operatorname{Mn}_{100-x}$ alloy in the γ phase as a function of Fe concentration (x).

parameter of the Fe_xMn_{100-x} alloy has been taken from Ref. [42], where the γ phase was stabilized in the full range of composition by adding a small amount of Cu (5 at.%) for x < 40at.% and of C (4 at.%) for x > 80 at.%. The two vertical dashed lines in the figure delimit the concentration range in which MEED oscillations have been observed, that is the region in which the Fe_xMn_{100-x} alloy films grow in a layer-by-layer fashion on Cu(001) and on Co/Cu(001). As outlined by the grey rectangular box, the MEED oscillations are observed when the lattice misfit is approximately in the range -1% < f < +1%. One therefore can conclude that a small lattice misfit is a prerequisite for the epitaxial growth of the system under investigation.

This analysis is actually just qualitative for several points of view. At first, having a lattice misfit in the range $f = \pm 1$ % is not a general limit for epitaxial growth. Indicative is for example

the case of a Co thin film grown on Cu(001). This system has a lattice misfit of f = 1.8 %, and still the layer-by-layer growth mode has been deduced from the presence of MEED oscillations [74]. At second the fcc structure of the studied Fe_xMn_{100-x} thin films has not been proven for the full range of composition, but just for the alloy at the equiatomic concentration. On the other hand an fcc structure has been reported for pure Fe grown on Cu(001) at least for a thickness range between 4 and 10 ML [81]. Intuitively one could therefore expect an fcc structure for the Fe_xMn_{100-x} alloy films at least in a range of composition between x = 100 at.% and x = 50 at.%. When x becomes much smaller than 40 at.% (limit for the fcc structure in bulk Fe_xMn_{100-x}), a structural change can indeed be expected in the alloy. For example an hcp phase has been reported in bulk Fe_xMn_{100-x} alloys at room temperature between x = 12 and x = 30 at.% [82]. These observations, namely a departure from the fcc phase for low Fe content in the alloy, are consistent with the present results of the absence of MEED oscillations at room temperature for x < 30 at.%. A change in the structure of the growing film can in fact lead to a growth mode more complicated than a simple layer-by-layer growth.

The fcc structure of the Fe₅₀Mn₅₀ alloy film deserves some further comments. Polycrystalline sputtered Fe₅₀Mn₅₀ films have been stabilized in the fcc structure on substrates promoting the fcc growth, as Cu [26, 49] or Ni₈₀Fe₂₀ [26, 83]. In the previous chapter the fcc structure of epitaxially grown Fe₅₀Mn₅₀ films has been deduced from the results of the LEED experiments. The ordered LEED pattern up to the maximum thickness investigated (26 ML Fe₅₀Mn₅₀) indicates that the film is a single crystal in registry with the substrate. This is in contradiction to Ref. [78], where a polycrystalline growth of Fe₅₀Mn₅₀ on Cu was suggested. The stabilization of the antiferromagnetic fcc γ phase is a particularly important point especially in view of possible applications in spin valves. The bcc α phase and the hcp ϵ phase, both present in the phase diagram of Fe_xMn_{100-x} alloys, would lower the AFM ordering temperature of the Fe₅₀Mn₅₀ layer, thus reducing the thermal stability of the spin valve. It has been indeed observed that in polycrystalline Fe₅₀Mn₅₀/Py bilayer a highly textured fcc phase induces a higher value of both the exchange bias field and the blocking temperature [84].

Generally the lack of a net magnetization renders difficult the study of the magnetic properties of AFM materials. On the other hand an FM material, in an exchange interacting FM-AFM bilayer, can be used as a probe of the magnetic order of the AFM material [85]. This approach has been used in the previous chapter to identify the thickness dependent transition from PM to AFM of an Fe₅₀Mn₅₀ thin film. The coercive field of a thin Co layer increases in fact steeply when the adjacent Fe₅₀Mn₅₀ layer thickness overcomes ≈ 10 ML (see Fig. 3.8). This effect is therefore related to a thickness dependent change in the properties of the Fe₅₀Mn₅₀ film. Since no structural change has been found by LEED in the investigated thickness range, it has been concluded that the increase in coercive field for $t_{FeMn} > 10$ ML is due to the interaction of Co with an AFM ordered Fe₅₀Mn₅₀ film. Indeed a coercivity enhancement is one of the fingerprints of the FM-AFM interaction. In other words a 10 ML thick Fe₅₀Mn₅₀ film would have an AFM ordering temperature around room temperature. This value is lower than the Néel temperature of bulk $Fe_{50}Mn_{50}$ (≈ 500 K [42]). This difference can be attributed to finite size effects [79]. A decrease of the AFM ordering temperature by reducing the film thickness has been reported for other AFM materials such as CoO [86] and NiO [87]. One could object that the increase in coercive field is a somehow indirect way to measure the AFM ordering temperature, especially considering that often in thin films the coercivity is related to microstructural parameters. On the other hand the change in the Co domain configuration at the same $Fe_{50}Mn_{50}$ thickness at which the increase in H_c has been measured, supports this view. Moreover, the measured AFM ordering temperature has been shown to shift towards higher values for increasing $Fe_{50}Mn_{50}$ thickness, an observation that is consistent with a finite size effect argument. In addition the coercive field of the $Fe_{50}Mn_{50}/Co$ bilayer has been shown (see Fig. 3.11) to scale linearly with the inverse of the Co thickness. This behaviour, besides being an indication of the interfacial nature of the interaction, has been attributed to the presence of losses in the AFM material in a recent model by Stiles and McMichael [88]. In this model the coercivity of an exchange biased FM-AFM bilayer stems from two mechanisms: inhomogeneous magnetization reversal, dominant at low temperature and high FM thicknesses, which leads to a decrease of the coercive field proportional to t_{FM}^{-2} (where t_{FM} is the FM thickness); instabilities in the AFM material, more relevant at high temperature, which cause a decrease of the coercive field like t_{FM}^{-1} . By increasing the $Fe_{50}Mn_{50}$ thickness on top of a FM Co thin film, the coercivity of the system must increase at that thickness at which the $Fe_{50}Mn_{50}$ layer becomes AFM. In fact when the AFM order is established, the spins in the antiferromagnet will couple to the FM material and are dragged during a magnetization loop by the Co magnetization, inducing the increased coercivity. At this $Fe_{50}Mn_{50}$ thickness in fact the AFM ordering temperature is still close to room temperature, and one is in the regime where the losses are predominantly due to instabilities in the AFM material.

By analogy with the Fe_xMn_{100-x} alloy at equiatomic concentration, the increase in coercive field has been used to estimate the AFM ordering temperature also for other composition values. As a result (see Fig. 3.10) the AFM ordering temperature has been found to increase monotonically with increasing Mn content. This is not fully in agreement with the variation of the Néel temperature as a function of concentration in bulk alloys [42]. In particular a maximum for T_N was found at $x \approx 50$ at.%. Instead in Fig. 3.10 the AFM ordering temperature still increases, at a fixed thickness, for x < 50 at.%. This difference could be attributed to a different magnetic behaviour of the alloy in the thin film form. On the other hand, what has been displayed in Fig. 3.10 is a transition temperature which is thickness dependent. In principle one can not exclude that films with different concentrations present different finite size effects. Additionally one has to consider that in Ref. [42] the fcc structure for the high Mn concentration range was stabilized by adding a small amount of Cu. Taking into account the influence of the added Cu, it has been shown [89] that the Néel temperature of bulk Fe_xMn_{100-x} alloy increases almost monotonically by increasing the Mn content. In particular the maximum at x = 50 at.% was not observed. This would be more in line with the present results on thin films.

4.2 Antiferromagnetic domains in $Fe_{50}Mn_{50}$ thin films?

As it has been shown in section 3.4 an FM Co film deposited on a wedge shaped $Fe_{50}Mn_{50}$ film displays a characteristic domain pattern. In particular for $t_{FeMn} < 10$ ML large domains with the magnetization oriented along the $\langle 110 \rangle$ in-plane directions are present. A similar domain configuration is encountered also when the Co film is deposited merely on Cu(001). When t_{FeMn} exceeds ≈ 10 ML the Co film presents much smaller domains with shrinking size for increasing $Fe_{50}Mn_{50}$ thickness. $t_{FeMn} \approx 10$ ML is exactly the thickness at which the sudden increase in coercive field has been measured. As discussed in the previous section this is related to the AFM ordering of the $Fe_{50}Mn_{50}$ film, when the AFM ordering temperature overcomes room temperature. The same mechanism is therefore considered here for the Co domain configuration: the small domains in Co observed for $t_{FeMn} > 10$ ML are induced by the interaction with the AFM ordered underlayer. These small domains are generally not favourable from an energetic point of view, as one can see from the Co domain configuration for $t_{FeMn} < 10$ ML. Every domain wall in fact adds an energy contribution to the sum of the total energy. The most likely hypothesis is therefore that the small domain configuration is frozen in by contact with domains in the AFM ordered $Fe_{50}Mn_{50}$ film when the Co grows on it. In this framework the Co film "senses" and replicates the AFM domain configuration of the $Fe_{50}Mn_{50}$ film. This implies also that these domains must be present at the surface of the AFM film. From Fig. 3.13 (b) one can estimate the dimension of these domains to be $\approx 1 \ \mu m$ around $t_{FeMn} \approx 10$ ML. For increasing $Fe_{50}Mn_{50}$ film thickness the domain dimension is even reducing, until around $t_{FeMn} \approx 13$ ML they are smaller than the instrumental resolution (≈ 400 nm). The small domains are not present when the Co film, deposited on Cu(001), is covered by the $Fe_{50}Mn_{50}$ film. In this case in fact the Co domain pattern is already arranged before the $Fe_{50}Mn_{50}$ deposition. This does not exclude, however, that domains are still present in the AFM ordered $Fe_{50}Mn_{50}$ film. Some indirect hint to this will be given in the next section.

By annealing the Co/Fe₅₀Mn₅₀ bilayer one realizes that the small domains visible in Co are metastable and that they are induced by the contact with a magnetic ordered Fe₅₀Mn₅₀ film. In fact, see Fig. 3.20, upon increasing the temperature the small domains merge together to form bigger domains. The annealing temperature is such that the Fe₅₀Mn₅₀ film is not anymore in an AFM state. In this case the Co domains are not pinned by the underlying antiferromagnetically ordered film, and can rearrange in order to minimize the total magnetic energy. Without the interaction of the AFM underlayer, for the Co film it is energetically more favourable to form big domains. Upon cooling down one crosses again the AFM ordering temperature of the Fe₅₀Mn₅₀ film from the high temperature side. Likely many small domains are again present in the Fe₅₀Mn₅₀ film, but the Co domain pattern formed at high temperature is now stable. Therefore at room temperature big domains, and not many small domains, are still present in the Co film. Yet the influence of the AFM ordered underlayer is present in the Co magnetic properties, since the big domains are now directed along the $\langle 100 \rangle$ in-plane directions. This implies a rotation of 45° of the Co easy axis of magnetization when the Co film is in contact with an AFM ordered film instead of a PM film. This easy axis change will be discussed in more detail in the next section.

The above interpretation, that the Co film replicates the domain structure of the underlying AFM $Fe_{50}Mn_{50}$ film, is supported by experiments where the domain configuration of an exchange interacting FM/AFM bilayer was detected simultaneously at both sides of the interface [90]. In particular a direct link was observed between the domain configuration in a Co film and in the underlying AFM LaFeO₃ film. Domain images where obtained by using a PEEM exploiting XMCD in order to image the FM layer, and x-ray magnetic *linear* dichroism (XMLD) [87, 91, 92] for the AFM layer. The XMLD signal is in fact proportional to the square of the magnetization, contrary to the XMCD signal which is linearly proportional to the magnetization. By using a PEEM in combination with XMLD one could therefore obtain magnetic domain images also from an AFM material where the net magnetization is zero. Indeed this has been done, besides the cited work on LaFeO₃, also for NiO [29, 31]. As a matter of fact such an experiment has been tried presently on the Fe₅₀Mn₅₀ films, but it results unsuccessful. A reason for this can be found in the crystal field splitting of the absorption peaks, which is present in LaFeO₃ and NiO oxides, but not in the metallic Fe₅₀Mn₅₀ films.

The possible configuration of the domains in the AFM $Fe_{50}Mn_{50}$ film must take into account its AFM spin structure. In chapter 1 it has been shown that the the non-collinear (110) and $\langle 111 \rangle$ spin configurations may be used to model the spin structure in bulk Fe₅₀Mn₅₀. As an approximation the bulk spin structure is considered to be conserved also in the thin films. By comparing the two spin-structure models one sees that the $\langle 111 \rangle$ model reduces to the $\langle 110 \rangle$ model if the spins are just projected into the film plane. In the $Co/Fe_{50}Mn_{50}$ system both the shape anisotropy and the strong in plane anisotropy of Co could indeed force the AFM spins to lie in the plane. Moreover, the difference in total energy between the two spin structures has been calculated to be rather small [45, 46], so that the imbalance given by the mentioned anisotropy argument could favour the (110) spin structure. It seems therefore a good approximation to consider that the $\langle 110 \rangle$ spin structure is realized in the present Fe₅₀Mn₅₀ films. In Fig. 4.2 (a) the spin structure of an Fe₅₀Mn₅₀ fcc unit cell, realizing the $\langle 110 \rangle$ spin model, is shown. The arrow on each site indicates the direction of the magnetic moment of that atom. In every other plane the moments have the same direction, as one can see by comparing Plane I and Plane III, where the atoms and the magnetic moment direction are depicted in black. The moments have a different direction in Plane II, where the grey has been used to colour the balls and the arrows. The top views of Plane I (b) and Plane II (c) reveal that in adjacent planes



Figure 4.2: (a) $Fe_{50}Mn_{50}$ bulk spin structure according to the $\langle 110 \rangle$ model. (b) and (c) are the top view of two successive planes. Arrows indicate the magnetic moment direction of the atom in that site.

the axis of the moments is rotated by 90° . These two terminations, for example, may induce different magnetic domains, as it is shown schematically in Fig. 4.3. Here just the domains at the surface of the $Fe_{50}Mn_{50}$ film are considered, and the domain walls are not depicted. The balls of different colours are atoms in different domains. Again the arrows indicate the direction of the magnetic moments. The straight lines connecting some of the atoms are just the squares of the fcc lattice. The in plane crystallographic axes are indicated. In Fig. 4.3 (a) a white and a grey domain are present in a flat $Fe_{50}Mn_{50}$ film. One recognizes that in the grey domain the structure of Fig. 4.2 (b) is realized, while in the white domain the moment direction of Fig. 4.2 (c) is repeated. The same two domains at the surface are realized also in the presence of a 1 ML high island, as depicted in Fig. 4.3 (b). There is a substantial difference between the domain configurations of Fig. 4.3 (a) and Fig. 4.3 (b). In order to understand this difference one has to consider that in an FM material the formation of magnetic domains is generally due to the reduction in energy by having magnetic flux closure patterns. Domains are therefore created until the energy gain is comparable to the added domain wall energy. An AFM material does not exhibit a magnetostatic stray field. The formation of domains is therefore unfavorable, and, in principle, an AFM material would have a stable configuration in a single domain state. The domains like the ones shown in Fig. 4.3 (a), extending maybe through the full thickness of the film, appear therefore unfavourable from an energetic point of view. This is even more true when considering that the number of the domains in the AFM $Fe_{50}Mn_{50}$ film must be at least as high



Figure 4.3: Some of the possible domain configurations in an AFM $Fe_{50}Mn_{50}$ film compatible with the bulk spin structure.

as the one of the domains visible in the Co film coupled to it. They therefore require a large amount of energy stored in each domain wall. In the case of Fig. 4.3 (b) a topological feature of the sample, namely a step, creates instead the two domains at the surface. The topological origin of the domains appear therefore more likely, since the domains are located only at the surface and no domain walls are required.

Up to now the domains at the AFM surface have been considered to be present before the Co film comes in contact with the $Fe_{50}Mn_{50}$ film. Actually one can not exclude a priori that, following Malozemoff's idea [16, 18, 19], the random field acting from the ferromagnet causes the antiferromagnet to break up into domain-like regions, following an argument originally made by Imry and Ma [93]. Here the randomness of the field arises for example from interface roughness or local difference in AFM alloy composition. By applying this model to the present observed domains in the Co/Fe₅₀Mn₅₀ system one has to postulate a mutual interaction between the two layers. The tendency of Co is to form big domains, each saturated along one of the $\langle 110 \rangle$ directions. In each of these domains the random field induces the underlying AFM film to break up in small domains. Since the Co film is actually in a growing process it minimizes the magnetic energy by replicating the small domains configuration of the AFM layer. Thus the small domains result frozen by exchange interaction also in the Co film.
As observed before, the small domains present in Co coupled to an AFM $Fe_{50}Mn_{50}$ film are shrinking by increasing t_{FeMn} . This can be explained by considering that for t_{FeMn} close to 10 ML the Co film during growth could already annihilate some of the smaller domains that had been present in the AFM $Fe_{50}Mn_{50}$ film before Co deposition. By increasing the $Fe_{50}Mn_{50}$ film thickness also the AFM ordering temperature increases, as it has been shown in section 3.3, leading to a more stable configuration of the AFM domains. In this case higher activation energy is required to annihilate some domains, and therefore the Co domains show a smaller size, closer to the original $Fe_{50}Mn_{50}$ domain size. Moreover one has to consider that in general the size of the domains in Co must not be the same as in $Fe_{50}Mn_{50}$, but they can not be smaller of them. On the other hand the smaller size of the Co domains for increasing t_{FeMn} can also be due to a corresponding shrinking of the AFM domains. If these domains are considered as in the Malozemoff's approach [18], they should scale as the inverse square root of the anisotropy constant of the antiferromagnet (K_{AFM}) . If this anisotropy increases by increasing t_{AFM} , the domain size (L) will in turn decrease. Since the exchange bias field is inversely proportional to L[18], one expects that the amount of shift in the hysteresis loop will decrease by decreasing t_{AFM} . This is indeed frequently observed experimentally [10]. Moreover the naive direct proportionality between the antiferromagnetic anisotropy and t_{AFM} could explain the observation of a shifted loop just for antiferromagnetic thickness above a critical value. As intuitively discussed in section 1.1, a high value of K_{AFM} compared to the exchange interaction between the FM and the AFM layer is required to obtain a shifted hysteresis loop. If this condition is not fulfilled, the hysteresis loop will not be shifted. However, the coercivity of the bilayer is still enhanced due to instabilities in the antiferromagnet. This is indeed the situation encountered in the MOKE experiments performed here. An 11 ML $Fe_{50}Mn_{50}/6$ ML Co bilayer on Cu(001) displays an increased coercivity, but not a shifted loop even after a field cooling procedure in a field of 1 kOe down to liquid nitrogen temperature. A finite value of H_{eb} has instead been obtained, after a field cooling procedure, just for higher $Fe_{50}Mn_{50}$ thickness (namely 20 ML), as shown in Fig. 3.12.

4.3 On the change in Co easy axis

The small domains present in a Co film grown on an AFM $Fe_{50}Mn_{50}$ film merge together to give larger domains upon annealing the sample. This has been demonstrated in Fig. 3.20, and it has been related to the crossing of the thickness dependent AFM ordering temperature of the $Fe_{50}Mn_{50}$ film. At high temperature the Co domains are oriented along the $\langle 110 \rangle$ azimuth directions, easy magnetization axes for a Co film grown on Cu(001) and on a PM $Fe_{50}Mn_{50}$ film. Upon cooling down to room temperature, the Co film coupled to the AFM $Fe_{50}Mn_{50}$ film has instead an easy axis of magnetization along the $\langle 100 \rangle$ azimuth directions. This effect is therefore related to the magnetic state of the $Fe_{50}Mn_{50}$ film. One has actually to consider that the change in Co easy magnetization direction observed after the thermal treatment is not *induced* by the thermal treatment. Most likely also in the as-grown state a Co film deposited on an AFM Fe₅₀Mn₅₀ film has an easy magnetization axis along the $\langle 100 \rangle$ directions. But, since the domains are very small, it just becomes difficult to be observed. Repetitive heating and cooling processes have been performed on the Co/Fe₅₀Mn₅₀ bilayers. The result, shown in Fig. 3.21, is that, once at room temperature, the Co film coupled to an AFM Fe₅₀Mn₅₀ has a magnetic easy axis along the $\langle 100 \rangle$ directions. The reversibility of this easy axis change implies that the effect is not due to a dramatic intermixing at the interface between the constituents of the films, but is related to the crossing of the AFM ordering temperature. Indeed the interaction at a Co/Fe₅₀Mn₅₀ interface seems quite stable with respect to thermal treatments. It has been shown that only annealing at a temperature as high as ≈ 570 K induces the formation of a FM FeMnCo alloy at the interface [94].

A close look at Fig. 3.21 reveals that the $Fe_{50}Mn_{50}$ thickness at which the transition from PM to AFM state takes place, as deduced from the Co domain pattern, is not the same in the three images. In order to clarify this point, image (c) of Fig. 3.21 is again reported in Fig. 4.4. In the upper left corner of the image the crystallographic axes are indicated. The



Figure 4.4: Domain pattern of a 9 ML Co film on top of a wedged $Fe_{50}Mn_{50}$ film. The $Fe_{50}Mn_{50}$ thickness is indicated at the left axis. The black dashed line indicates the thickness at which the Co film presents small domains in the as-grown state, as taken from Fig. 3.21 (a). The white dashed line is the transition between regions of different easy axis of magnetization.

black dashed line indicates the thickness at which in the as-grown state the Co film starts to present small domains, as taken from Fig. 3.21 (a). As discussed in the previous section this is the thickness at which the $Fe_{50}Mn_{50}$ film orders antiferromagnetically at room temperature. This thickness is around 10 ML. After an annealing process and a subsequent cooling back to room temperature the big newly formed domains in Co are oriented along the (100) azimuth directions. The change in easy axis is due to the AFM ordered $Fe_{50}Mn_{50}$ film. One therefore expects that it appears again around $t_{AFM} \approx 10$ ML. This is in fact the case for Fig. 3.19 where the sample was annealed in the preparation chamber, cooled down to room temperature and then transferred into the PEEM chamber. It is instead not realized in Fig. 4.4. The white dashed line in this figure indicates approximately the thickness at which the change in the Co easy axis appears. Evidently this line does not coincide with the black dashed line, but it is shifted towards higher $Fe_{50}Mn_{50}$ thickness of more than 1 ML. One could try to explain this shift by considering a formation of some alloy at the interface. As a matter of fact the film imaged in Fig. 4.4 has been annealed at a temperature that can be estimated to be 100 K higher than the one of Fig. 3.19. The supposed formed alloy layer, if it is antiferromagnetic, should have a Néel temperature lower than $Fe_{50}Mn_{50}$, at least under the assumption that no changes in the structure have occurred, and that the change in easy axis is merely due to the AFM order of the underlayer. In this case in fact one could expect that for lower T_N the AFM ordering temperature equals room temperature at a higher thickness. In this view a possibility is that some Co atoms have migrated into the $Fe_{50}Mn_{50}$ film to form an FeMnCo alloy at the interface. If this alloy is in an AFM γ phase, rather than in an FM state as in Ref. [94], it would have an AFM ordering temperature lower than $Fe_{50}Mn_{50}$. In fact the addition of Co to a γ -FeMn alloy reduces the Néel temperature [95]. If the formed FeMnCo alloy is FM, also a shift of the transition line would be expected. In this case the formation of the alloy reduces the effective thickness of the AFM $Fe_{50}Mn_{50}$ film, with the apparent result of a shift of the AFM ordering temperature towards lower values.

Actually a simpler and easier explanation of the shift of the transition line outlined in Fig. 4.4 can be found. As mentioned in the previous chapter the PEEM sample stage is not equipped with a thermocouple, therefore the temperature was just roughly estimated by consistency with the MOKE experiments. In the present case in particular the evaluation of the time to reach room temperature after stopping annealing could have been not correct. In this case the temperature at which Fig. 4.4 was taken could have been still relaxing towards room temperature. This of course would explain the shift in the transition line. An indication of the validity of this interpretation comes from the area enclosed by the white circle in Fig. 4.4. Here, in a sea of light grey scale, a dark grey domain is visible in the lower part inside the circle. This domain seems approaching the black domain just on top of the white circle, but its boundaries become faint when increasing thickness. This thickness region is just the one around the white dashed line, where the change in easy axis is now taking place.

dark grey domain, at least in the lower part inside the circle, looks the same as the domains directed along the $[\bar{1}10]$ axis for example in Fig. 3.20 (e). These intermediate domains, created upon annealing the sample, decay in some of the $\langle 100 \rangle$ directions once at room temperature, as seen in Fig. 3.20 (g). Indeed some of these intermediate grey scale domains are still present in Fig. 4.4 in the region between the white and the black dashed lines. Moreover, the dark grey domain enclosed in the white circle seems still decaying in the [010] direction. Indeed it is the only domain without well defined boundaries. This is an indication that the relaxation of the temperature towards room temperature was not yet complete. The shift between the white and the dashed line in Fig. 4.4 would have in this case not a structural origin. One has of course also to consider that the transition between the $\langle 110 \rangle$ to the $\langle 100 \rangle$ easy magnetization axis, for increasing $Fe_{50}Mn_{50}$ thickness, must not necessarily be sharp. In particular, as it will be discussed below, for increasing Co thickness the total Co anisotropy per unit area also increases, leading to a smearing out of this transition, until for rather thick Co films (more than ≈ 20 ML) the original easy magnetization direction along the $\langle 110 \rangle$ is resumed. By comparing Figs. 3.19 and 4.4 one realizes that in the imaged area the Co film in the latter is thicker than in the former. In both cases the small domains frozen in Co during growth on top of the AFM $Fe_{50}Mn_{50}$ film are observable at the same $Fe_{50}Mn_{50}$ thickness (above ≈ 10 ML), when the AFM ordering temperature overcomes room temperature. After the annealing process a direction of magnetization along the $\langle 110 \rangle$ directions may be favourable for the 9 ML thick film of Fig. 4.4, at least in the region slightly above $t_{FeMn} \approx 10$ ML, where the AFM ordering temperature is still close to room temperature. Here, for that Co thickness, a frustration of the AFM spins directions could occur rather than a change in the Co easy axis. This mechanism could therefore also lead to the shift in the transition line as observed in Fig. 4.4.

The same change in easy axis visible in the Co film after a thermal treatment of the $Co/Fe_{50}Mn_{50}/Cu(001)$ system, is also present when the order of deposition is inverted. When a $Fe_{50}Mn_{50}$ film is grown on a thin Co layer, the Co magnetization is along the $\langle 100 \rangle$ directions for $t_{FeMn} > 10$ ML. These two observations have not only the same origin, that is the coupling of Co to the AFM $Fe_{50}Mn_{50}$, but they are realized also in the same way. In both cases the pattern of the Co domains is generated when the Co film is *not* in contact with an AFM $Fe_{50}Mn_{50}$ film. The change in easy axis, in contrast, is realized when this contact takes place. For the $Co/Fe_{50}Mn_{50}$ configuration this can be understood by comparing Fig. 3.20 (e) and (g). At 480 K (e) the Co film interacts with a PM $Fe_{50}Mn_{50}$ film, and the Co domain pattern is arranged in order to minimize the Co "internal" magnetic energy. By cooling to room temperature (g) Co is brought into contact with an AFM material. The interaction with the AFM $Fe_{50}Mn_{50}$ film is evident by the change in easy axis. Likewise when Co is grown on Cu(001), a domain pattern is established that stays similar also after the subsequent deposition of $Fe_{50}Mn_{50}$, but, as before, for $t_{FeMn} > 10$ ML these domains display an easy axis of magnetization along the $\langle 100 \rangle$ directions. The evidence of this last statement has been given in Fig. 3.15.

The change in Co easy axis, due to the AFM ordering of the Fe₅₀Mn₅₀ film, can be explained by exploiting the hypothesis of domains in the AFM material "sensed" by the Co overlayer in the as-grown state. One has to consider that these domains must induce a preferred direction of the Co magnetization along the $\langle 100 \rangle$ axes. A first possibility is to consider an AFM domain structure analogous to the one of Fig. 4.3 (a), but with the AFM spins oriented along the $\langle 100 \rangle$ axes. This domain configuration, shown in Fig. 4.5 (b), is actually compatible with both an inplane non-collinear structure and with a collinear structure. In this second case each columnar



Figure 4.5: Schematic representation of a Co film (a) on top of an AFM ordered $Fe_{50}Mn_{50}$ film (b), where different domains are characterized by different directions of the AFM spins along the $\langle 100 \rangle$ directions.

domain would realize a collinear spin structure with the spins directed along one $\langle 100 \rangle$ axis, and with a rotation of this axis by 90° in the neighbour domain. The dimension of these domains must be smaller than the one of the Co domains in the as-grown state on top of AFM Fe₅₀Mn₅₀, therefore they may be as small as ≈ 100 nm. In Fig. 4.5 (a) a schematic representation of a Co film deposited on top of this domain structure is shown. In order to simplify the discussion, the Co film is considered to replicate exactly the domains in Fe₅₀Mn₅₀. The magnetization of the Co domains is forced by the underlying AFM domains along the $\langle 100 \rangle$ axes. In fact, for example, the AFM spins in the dark domain of Fig. 4.5 (b) are pointing along the [010] and the [010] directions. In this case, in analogy with Koon's model [20], a direction of the Co magnetization 90° from the AFM axis is energetically favourable. Therefore in that domain the magnetization in Co will point for example along the [100] direction, as depicted in Fig. 4.5 (a). In analogy the Co coupled to the white AFM domain finds a stable configuration by pointing, for example, along the [010] direction. When the Co/Fe₅₀Mn₅₀ bilayer is annealed and then cooled down to room temperature, or when the Fe₅₀Mn₅₀ film is grown on top of the Co film, the change in easy axis is present in much bigger Co domains. One could expect in this case that the Co domains have annihilated the small domains in Fe₅₀Mn₅₀. The latter, on the other hand, still maintain a spin configuration, in each domain, similar to the one displayed in Fig. 4.5 (b), leading therefore to a preferred orientation of the Co magnetization along the $\langle 100 \rangle$ directions.

An argument against this hypothesis can be found for the same reason which led, in the previous section, to discharge the possibility of AFM domains as the ones displayed in Fig. 4.3 (a). Indeed also the domain configuration shown in Fig. 4.5 (b), due to the energy stored in the domain wall, may not be favourable from an energetic point of view. An alternative explanation of the change in the Co easy axis, which does not suffer this limitation, involves the topological domains presented in the previous section. One can consider for simplicity the domain structure realized by the presence of a 1 ML step, displayed in Fig. 4.3 (b). The AFM spins are there directed along one of the two in-plane (110) axes in each domain, with a rotation of 90° from one domain to the next. For example in the grey domain of Fig. 4.3 (b) the spins are directed along either the [110] or the $[\bar{1}\bar{1}0]$ direction, while in the white domain they are along the $[1\bar{1}0]$ or the $[\bar{1}10]$ directions. When a Co film is brought into contact with many such domains, it would feel an exchange interaction statistically directed along the four (110) directions. This is true if the size of these topological AFM domains is smaller than the Co exchange length (around 3 nm). As a result of this exchange averaging over the four equivalent spin directions, the Co magnetization finds a stable configuration at 45° from the $\langle 110 \rangle$ directions, that is along the $\langle 100 \rangle$ axes. A simple energy minimization argument, following the sketch reported in Fig. 4.6, leads in fact to this conclusion. The four perpendicular directions are the four $\langle 110 \rangle$ axes of the AFM spins. The Co magnetization (M_{Co}) is directed along an azimuth angle φ , where $\varphi = 0$ corresponds to the [110] direction. For energy minimization a small twist $\delta \varphi \to 0$ of the AFM spins into the FM direction is allowed. The problem is to find the angle φ at which the energy of the system is minimized. One can assume that the Co interacts with each AFM spin leading to an energy of the form

$$E = J\cos\psi \tag{4.2}$$

Therefore the energy of the system of Fig. 4.6, when the Co magnetization lies in the first quadrant, can be expressed, in unit of J, as the sum of the interaction between the Co magnetization and the four Fe₅₀Mn₅₀ spin directions:

$$E = \cos(\varphi - \delta\varphi) + \cos(\pi - \varphi - \delta\varphi) + \cos(\frac{\pi}{2} - \varphi - \delta\varphi) + \cos(\frac{\pi}{2} + \varphi - \delta\varphi), \qquad (4.3)$$



Figure 4.6: Schematic configuration where the four perpendicular directions are the four $\langle 110 \rangle$ directions of the AFM spins. The Co magnetization (M_{Co}) lies along the solid arrow at an angle φ from the [110] direction. A small twist $\delta \varphi \to 0$ of the AFM spins (dashed arrows) towards the Co magnetization direction is allowed.

which may be written as

$$E = \cos(\varphi - \delta\varphi) - \cos(\varphi + \delta\varphi) + \sin(\varphi + \delta\varphi) - \sin(\varphi - \delta\varphi)$$
(4.4)

Analogous expressions can be found when M_{Co} is in the other three quadrants. By taking the first derivative of these expressions, extrema are found when the condition

$$\sin(\varphi + \delta\varphi) - \sin(\varphi - \delta\varphi) = \cos(\varphi \mp \delta\varphi) - \cos(\varphi \pm \delta\varphi)$$
(4.5)

is satisfied. Here the \pm signs at the respective $\delta \varphi$ are valid for the first and third quadrant, the upper sign, and for the second and forth quadrant, the lower sign. Applying the formula of addition and subtraction of sin and cos, equation 4.5 becomes

$$\sin\varphi\cos\delta\varphi + \cos\varphi\sin\delta\varphi - \sin\varphi\cos\delta\varphi + \cos\varphi\sin\delta\varphi = = \cos\varphi\cos\delta\varphi \pm \sin\varphi\sin\delta\varphi - \cos\varphi\cos\delta\varphi \pm \sin\varphi\sin\delta\varphi,$$
(4.6)

which reduces to

$$\cos\varphi = \pm \sin\varphi,\tag{4.7}$$

and is satisfied for $\varphi = 45^{\circ}$, 135° , 225° , and 315° , that is when the Co magnetization is directed along the $\langle 100 \rangle$ axes, at 45° from the $\langle 110 \rangle$ directions. From this result one therefore expects that the Co anisotropy has still a fourfold symmetry when the Co film is coupled to AFM Fe₅₀Mn₅₀. In the obtained images actually the Co film never showed four different grey scales for $t_{AFM} > 10$ ML. However at least in some cases, see for example Fig. 3.17, a uniaxial anisotropy can be excluded for the Co film coupled to AFM Fe₅₀Mn₅₀, since two domains magnetized along two perpendicular $\langle 100 \rangle$ directions are present.

The suggested model for the change in Co easy axis, when Co is coupled to an AFM $Fe_{50}Mn_{50}$ film, can be compared with the mechanism proposed by Slonczewski for explaining the biquadratic exchange coupling in trilayers consisting of FM layers separated by a non-FM spacer layer [97]. In his model spatial fluctuations of the spacer layer thickness, leading to alternating 0° and 180° coupling between two FM layers, are shown to induce an effective 90° coupling. Similarly in the present case an exchange averaging over 90° domains, induced by mono-atomic steps as the one shown in Fig. 4.3 (b), leads to a 45° coupling between the FM and AFM axes. Therefore one would expect the interface of an $Fe_{50}Mn_{50}$ film to be characterized by 90° domains generated by mono-atomic steps. The exchange averaging over these topological domains depends on the size of the domains, and such does the magnetic coupling. Indeed an investigation by scanning tunneling microscopy supports this interpretation [98]. The surface of an $Fe_{50}Mn_{50}$ film grown on Cu(001) displays a large number of small, ≈ 1 nm wide islands and holes, 1 ML high, in otherwise atomically flat terraces. Since an $Fe_{50}Mn_{50}$ film grows in a layer-by-layer mode on Cu(001) and on Co/Cu(001), the number of surface steps oscillates also with a ML period. Therefore also the coupling between the FM and the AFM layer should oscillate as a function of the $Fe_{50}Mn_{50}$ thickness, if the above considerations were true. This is indeed the case as shown in Fig. 4.7, where the Co domain pattern of a $Co/Fe_{50}Mn_{50}$ crossed double wedge deposited on Cu(001) is displayed in the as-grown state (a) and after application of a pulsed field of 330 Oe (b). The direction of the applied field (H) is displayed by the grey arrow in the rectangular box in the upper right corner of the figure, together with the crystallographic azimuth axes and the incoming x-rays direction $(h\nu)$. The Fe₅₀Mn₅₀ thickness is indicated at the left axes of images (a) and (b), increasing from bottom to top, while t_{Co} is indicated just at the bottom axis of image (b), increasing from left to right. The arrows in some domains display the magnetization direction. In image (a) one can see that, as usual, the Co film displays small domains when t_{FeMn} overcomes ≈ 10 ML. After a field pulse of 330 Oe (b) the Co film is saturated along the $[\bar{1}\bar{1}0]$ direction just in the bottom part of the image. For higher Fe₅₀Mn₅₀ thickness still the small domains are present, even if now many of them have switched to give a darker contrast. In the upper left side of the image however some stripes where the small domains have not switched are visible. In these stripes the domain pattern is still similar to image (a), before the application of the external field. In Fig. 4.7 (c) a vertical linescan of the asymmetry values, horizontally averaged over the rectangle printed in Fig. 4.7 (b), is shown. One can see that the stripes appear with a period of 1 ML on t_{FeMn} . One can attribute the stripes to a modulation of the coercive field as a function of the $Fe_{50}Mn_{50}$ thickness. As it has been shown in section 4.1, the coercivity of a Co/Fe₅₀Mn₅₀ bilayer mainly depends, for $t_{FeMn} > 10$ ML,



Figure 4.7: Co domain pattern of the same area of a Co/Fe₅₀Mn₅₀ bilayer on Cu(001) in the as-grown state (a) and after application of a field pulse of 330 Oe (b). Thicknesses are indicated at the axes. The rectangular box in the upper right side contains information about crystallographic axes and applied field (H) and x-rays ($h\nu$) directions. In (c) a vertical linescan of the XMCD asymmetry, averaged on the rectangular area shown in (b), is displayed.

on the interaction between the Co film and the AFM $Fe_{50}Mn_{50}$ film. This means also that it depends on the strength of the interface coupling between the two films. Therefore the coupling strength between the Co and the $Fe_{50}Mn_{50}$ layer oscillates with a 1 ML period, as a consequence of the oscillation of the number of step edge atoms related to the layer-by-layer growth mode. This indeed indirectly proves the importance of mono-atomic steps for the interaction at the Co-Fe₅₀Mn₅₀ interface.

The change in the Co easy axis has been explained by having a pinning exerted from the AFM Fe₅₀Mn₅₀ on the Co along the $\langle 100 \rangle$ directions. This pinning direction, being a property of the Fe₅₀Mn₅₀ layer, must still be present when the Co film, for $t_{Co} > 15$ ML, switches the easy axis of magnetization back to the original $\langle 110 \rangle$ directions, as mentioned in section 3.4. Some indication of this is given in Fig. 4.8. In (a) the Co domain pattern of a 20 ML Co/15 ML Fe₅₀Mn₅₀/Cu(001) is imaged at room temperature after heating to 480 K and subsequent



Figure 4.8: (a) Co domains pattern of a 20 ML Co/15 ML Fe₅₀Mn₅₀/Cu(001) bilayer. The same sample area is imaged after a field of 145 Oe (b), 165 Oe (c), and 220 Oe (d) applied along the indicated +H direction (grey arrow). The crystallographic axes and the direction of the incoming x-rays $(h\nu)$ are displayed. Arrows in some domains indicate the magnetization direction.

application of a field H of -220 Oe at room temperature. The positive and the negative direction of the field are shown by the grey arrows. The crystallographic axes are indicated in the figure, along with the incoming x-rays direction $(h\nu)$ displayed by the fat black arrow. One can see that in Fig. 4.8 (a) the magnetization direction in the domains, displayed by the arrows, is along the [110] and [110]. For this high Co thickness the original anisotropy direction of a Co film is therefore restored. The same sample area has been imaged after the application of pulsed fields of increasing strength in the positive field direction, namely 145 Oe (b), 165 Oe (c), and 220 Oe (d). One sees that the magnetization in each of the original domains is just rotated by 90°, and even the highest field is not enough to rotate the magnetization by 180°. In particular a field of 220 Oe has rotated the magnetization of the original dark grey domain into the [110] direction (black domain) and the original white domain into the [110] direction (light grey domain). A possible explanation of this behaviour can be found by assuming a pinning direction exerted by the Fe₅₀Mn₅₀ layer along the [010] and [010] directions, as sketched in the center of the figure. Even if the Co anisotropy now favours an orientation along the $\langle 110 \rangle$ axes, still, for certain field range, the Co magnetization flips just by 90° between two adjacent $\langle 110 \rangle$ axes. In fact a rotation of 180° would induce a rearrangement of the spins in the antiferromagnet, leading to the opposite pinning direction, which probably would require a higher field.

The fact that Co for t_{Co} above ≈ 15 ML is found to be magnetized along the $\langle 110 \rangle$ directions can be explained by considering that the total anisotropy energy per unit area in the Co film increases when increasing the Co thickness [96]. This also indirectly suggests that the change in easy axis due to the interaction with the AFM layer is an interface effect. The restoring of the original Co anisotropy has not been studied in details. It seems to happen in a range of several Co ML, but it is not clear if it is realized as a continuous canting of the magnetization direction or by a coexistence of both easy axes. The change in easy axis from the $\langle 100 \rangle$ directions to the $\langle 110 \rangle$ directions allows one to estimate the strength of the interface coupling energy between the FM and the AFM layer. In fact at the Co thickness, where this change takes place, the coupling energy should be comparable with the Co anisotropy energy. The fourfold in-plane anisotropy energy of a 15 ML Co/Cu(001) has been found to be around 2×10^{-4} J/m² [96]. If this value is similar also for a Co film with the same thickness in a $Co/Fe_{50}Mn_{50}$ system, the value of $2 \times 10^{-4} \text{ J/m}^2$ is an estimate of the interface coupling strength between Co and Fe₅₀Mn₅₀. The interface energy of the interaction between an FM material and $Fe_{50}Mn_{50}$ reported in literature spans actually quite a big range, from $1 \times 10^{-5} \text{ J/m}^2$ to $4.7 \times 10^{-4} \text{ J/m}^2$ (see Ref. [10]). The above value for the present $Co/Fe_{50}Mn_{50}$ single crystal bilayers falls inside this range.

The change of magnetization direction of a ferromagnet by interaction with an AFM material has also been observed in Ni₈₀Fe₂₀/Fe₅₀Mn₅₀ bilayers epitaxially grown on Cu(110) [99, 100]. One of the results of the interaction is that a strong in-plane uniaxial anisotropy develops along the [110] axis, while a mere Ni₈₀Fe₂₀ layer had a preferred easy magnetization axis along the \pm [001] directions. Thus a rotation of 90° of the magnetic easy axis is observed in this system. A "mesa and valleys" argument has been used to explain this change in easy axis [51]. Considering for the Fe₅₀Mn₅₀(110) film a spin structure of the $\langle 111 \rangle$ type, every plane in the direction perpendicular to the surface is in fact uncompensated, with the spins pointing alternatively along the [001] and the [001] directions. Adjacent mesa and valleys of one atomic layer would have therefore antiparallel orientation of the surface spins. If the lateral dimension of these mesa and valleys is smaller than some characteristic length in the Ni₈₀Fe₂₀ layer, this would lead to the observed rotation of the FM magnetization of 90° to the AFM spins. This model is qualitatively identical to the one proposed above, in order to explain the change in easy axis in the Co film, considering the presence of topological domains.

4.4 Ferrimagnetic moments in $Fe_{50}Mn_{50}$ alloy

In section 3.5 domain images of Fe and Mn in the $Fe_{50}Mn_{50}$ alloy film have been shown. Since the imaging technique is sensitive to the average magnetic moment, this immediately suggests that the $Fe_{50}Mn_{50}$ film is not anymore an antiferromagnet, as it is in the bulk, but rather a *ferrimagnetic* material. Actually images of domains in Fe and Mn could just be detected when $Fe_{50}Mn_{50}$ was in contact with an FM Co film. The ferrimagnetic state of the alloy film is therefore induced by the Co film. This also suggests that the magnetic moments are induced in Fe and Mn mainly at the interface, i.e., close to the Co film. This can not be tested definitively, since the way the magnetic images are obtained gives an average contribution along the direction perpendicular to the surface. An indication that the induced moments are mainly located at the interface with the Co film is suggested by the the increasing contrast in the Fe and Mn domain images by decreasing $Fe_{50}Mn_{50}$ thickness. This is shown, for example in the Fe case, in Fig. 4.9. In (a) the domain pattern of Co from an $Fe_{50}Mn_{50}/Co$ double wedge grown on Cu(001) is shown. The $Fe_{50}Mn_{50}$ and Co thicknesses of the imaged sample area are indicated at the left axis, increasing from bottom to top, and at the bottom axis, increasing from left to right, respectively. The arrows in some domains indicate the magnetization direction. In the box at the bottom of the figure the crystallographic axes are indicated, along with the incoming x-rays direction $(h\nu)$ at 52° from the [100] axis. A linescan of the asymmetry (Asy) value along the $Fe_{50}Mn_{50}$ slope is extracted from the domain along the [110] direction in image (a), horizontally averaging over the black rectangle printed in the image. The linescan is shown in Fig. 4.9 (b), where t_{FeMn} is indicated at the left axis, and the value of the asymmetry at the bottom axis. One sees that the asymmetry of Co is almost constant along the $Fe_{50}Mn_{50}$ slope. The same sample area as in (a) was imaged again by tuning the x-rays energy to the Fe L_3 absorption edge. In this way the Fe domain pattern shown in Fig. 4.9 (c) is obtained. The linescan extracted from the rectangle printed in the image is shown in (d). The asymmetry displays a strong dependence on the Fe₅₀Mn₅₀ thickness. It increases up to $t_{FeMn} \approx 1$ ML, it stays almost constant for the next ML, and then starts to decrease, reaching a constant value at ≈ 6 ML. This last value of asymmetry is then almost the same as for higher $Fe_{50}Mn_{50}$ thicknesses, and in particular when the Fe₅₀Mn₅₀ film is in an AFM state at room temperature ($t_{AFM} > 10$ ML). The thickness dependent behaviour of the Fe asymmetry suggests that in the first stage of growth the Fe atoms are fully magnetic. Indeed the value of asymmetry measured in this region (≈ 16 %) is similar to the value of a thick Fe film grown on W(001), which can be considered as a standard value of bulk material for the present geometry and degree of polarization [101]. By increasing thickness, non ferromagnetic material is added, thus decreasing the amount of the average magnetic signal. This suggests that the induced magnetic moments in Fe are located at the interface with Co. If this interpretation is correct, one could try to quantify the thickness (t_M) of the induced FM material present at the $Fe_{50}Mn_{50}$ side of the interface. In fact the ratio R between the value of asymmetry of Fe in the $Fe_{50}Mn_{50}$ film and the one of the bulk can be expressed in continuous thickness model as

$$R = \frac{\int_0^{t_M} \exp^{-x/\lambda} dx}{\int_0^\infty \exp^{-x/\lambda} dx}$$
(4.8)



Figure 4.9: Co (a) and Fe (c) domain patterns from the same area of an $Fe_{50}Mn_{50}/Co$ double wedge on Cu(001). $Fe_{50}Mn_{50}$ and Co thicknesses are indicated at the left and bottom axes of the images, respectively. Crystallographic axes and incoming x-rays direction $(h\nu)$ are indicated in the box below the images. In (b) and (d) linescans along the vertical direction of the images, horizontally averaging over the rectangles printed in the Co and Fe images, respectively, are shown.

where λ is the effective escape depth of the secondary electrons. The value of maximum contrast between oppositely magnetized domains measured in the Fe images, for $t_{FeMn} > 7$ ML, is about 1.5 %, while it amounts to 32 % for a thick Fe standard. This leads to a value of $R \approx 0.05$. From equation 4.8 the thickness of the induced magnetic layer at the interface can be calculated as

$$t_M = -\lambda \ln(1 - R) \tag{4.9}$$

Considering a value of $\lambda = 20$ Å [67], and taking the lattice spacing of Fe₅₀Mn₅₀ layers as 1.81 Å, one obtains $t_M \approx 0.5$ ML. This would mean that half of the Fe atoms in the interfacial ML are in an FM state. A similar estimation is not possible for the induced magnetic moment in Mn, since no value of the asymmetry for FM bulk Mn is available. In general, the measured Mn asymmetry in different domain images is not as reproducible as for Fe. Moreover whereas the Fe magnetic domains are always found in the same direction as the Co ones, Mn is sometimes coupled antiparallel to Co and Fe. These particular features of the induced moment in Mn will be discussed in more detail below.

An induced moment in a non-FM material by the proximity of an FM material is actually a general observation in thin film magnetism, dating back to the magnetization of Pd films by interaction with FM Ni films [102]. Indeed small magnetic moments, induced by proximity of FM materials, have been found in antiferromagnets as Cr [63] and also in non-magnetic metallic layers as Cu in contact with Co [103]. Hybridization effects of the *d*-electrons are generally claimed to give rise to such observations. The partial FM state of an antiferromagnet in contact with a ferromagnet could be important in order to understand exchange bias. For example the original model proposed by Meiklejohn and Bean [2] and the more recent calculations of Schulthess and Butler [22] forecast a non shifted hysteresis loop when the AFM interface is compensated. Uncompensated interfacial moments in an AFM material were at first observed in CoO, induced by a field cooling procedure [34]. Uncompensated moments have also been claimed in Fe₅₀Mn₅₀ sputtered film sandwiched between Co and Py [35] and uncompensated Ni spins were found in NiO/Co bilayers [36], where however the exact origin of them was not specified.

The present experiments have provided evidence that the contact with the Co film induces ferromagnetic moments in Fe and Mn, no matter whether the Fe₅₀Mn₅₀ is in a PM state (for $t_{FeMn} < 10$ ML) or in an AFM state (for $t_{FeMn} > 10$ ML). In particular when the alloy is in an AFM state one is tempted to consider the measured induced moments as *uncompensated* moments. This is actually not completely true. Uncompensated moments are, by definition, moments of atoms in an AFM lattice which sum up to give a non vanishing net magnetization. The induced moments in Fe and Mn, however, have been measured also when the Fe₅₀Mn₅₀ film is PM, where they fully participate to the FM phase of Co. When Fe₅₀Mn₅₀ is AFM, *a small part* of the measured induced moments is given by "real" AFM uncompensated moments. While most of the induced moments still participate to the FM phase of Co, switching their magnetization direction together with the Co film, the small fraction of uncompensated moments should be pinned to the direction imposed by the AFM spins in order to give rise to the shift in the hysteresis loop. These uncompensated moments are then in the direction of the Co magnetization just when the $Fe_{50}Mn_{50}$ film is deposited on top of Co, or after a field or remanent cooling procedure. Once this direction is established they will not rotate upon switching the Co magnetization, thus leading to exchange bias.

From an intuitive point of view, the origin of the uncompensated moments in the AFM $Fe_{50}Mn_{50}$ alloy can be explained by referring to Fig. 4.6 and the related discussion. A small twist of the AFM axes towards the magnetization of Co was allowed there, in order to explain analytically the rotation of the Co easy axis. This distortion in the AFM spin structure would give rise to a small uncompensated magnetic moment in the $Fe_{50}Mn_{50}$ film. Uncompensated moments may also be induced at the topological domains introduced in the previous sections in order to explain the domain configuration of a Co film coupled to an AFM $Fe_{50}Mn_{50}$ layer. An example is given in Fig. 4.10. The top view of a portion of the $Fe_{50}Mn_{50}$ surface, where



Figure 4.10: Schematic representation of the top view of a topological domain, created by a 1 ML step, at the $Fe_{50}Mn_{50}$ surface. The spins in the region marked by the dashed line are uncompensated. The in-plane crystallographic axes are indicated.

a topological domain created by a 1 ML high step is present, is schematically displayed. The surface of the grey domain is, for example, 1 ML above the white domain. The in-plane crystallographic axes are indicated. The direction of the AFM spins, represented by the arrows, is along the $\langle 110 \rangle$ in plane directions. If the step runs along one of the $\langle 100 \rangle$ axes the spins at the step are uncompensated, as one can see in the region marked by the black dashed line, where

they sum up to a net component along $[0\bar{1}0]$. In order to explain the change in easy axis of a thin Co film coupled to an AFM Fe₅₀Mn₅₀ film, no specific orientation is required for the steps. Statistically it is possible that some of them are directed along the $\langle 100 \rangle$ axes, thus originating uncompensated spins.

The effective influence of the uncompensated $Fe_{50}Mn_{50}$ moments on the coupling at the interface with Co is actually difficult to quantify from the images of the induced moments in Fe and Mn. This is related to the fact that the induced moments signal is already quite low, and that, as mentioned above, the uncompensated moments are just a small fraction of the induced moment. One can see this by considering that the induced moments do not change appreciably when $Fe_{50}Mn_{50}$ is PM or AFM, and that uncompensated moments are not present when the alloy film is paramagnetic. Moreover the uncompensated moments should mediate the coupling between the FM and the AFM layer in the way explained above. In the previous section (see Fig. 4.7) an oscillation in the coupling strength has been deduced by the 1 ML oscillation in the coercivity. If this interpretation is correct, also the value of the uncompensated moments should oscillate with a period of 1 ML. Actually in the Fe and Mn images such a behaviour in the asymmetry can not be seen. This can be explained by considering that most part of the obtained asymmetry is not given by the uncompensated moments, whose oscillating behaviour as a function of the Fe₅₀Mn₅₀ thickness is therefore hidden.

The induced moments in Mn deserve some more comments. While the asymmetry in Fe (and therefore the value of the magnetic moment) has always been found similar in all the samples imaged with the PEEM, a different behaviour has been observed for Mn. In fact the asymmetry value, being usually slightly smaller than the one measured in Fe, in some samples has been found higher than the Fe one, in some others such small that domain images could not be detected. Moreover while the induced moments in Fe were always found parallel to the Co film magnetization, the domains in Mn have been found in some cases magnetized in the same direction as the Co and Fe ones (see Fig. 3.16), in some other cases in the opposite direction, as in Fig. 3.17. In this respect the experiment shown in Fig. 4.11 is symptomatic. Six images are present in the figure, displaying the Co (upper row) and the Mn (lower row) domain configuration from a 20 ML Co/16 ML $Fe_{50}Mn_{50}$ bilayer grown on Cu(001) and annealed at ≈ 420 K after deposition. The crystallographic axes and the direction of the incoming x-rays $(h\nu)$ are displayed by the arrows in the figure. In the images of column (a) Co and Mn are imaged at room temperature in the as-prepared state. The Co domain pattern can be compared with the one of Fig. 3.19, except that now the magnetization is directed along the (110) azimuth axes, as displayed by the arrows in some domains. This is expected considering that for a 20 ML thick Co film the anisotropy energy favours again these directions, as discussed in the previous section. Mn shows an inverted contrast with respect to Co, indicating that the magnetization direction in Mn is antiparallel to the Co one. In column (b) Co and Mn are imaged at ≈ 500 K, after the application of field pulses up to H = 110 Oe in both the directions of the grey double



Figure 4.11: Images in column (a) display Co (upper row) and Mn (lower row) domain patterns at room temperature from a 20 ML Co/16 ML Fe₅₀Mn₅₀/Cu(001) sample, annealed at ≈ 420 K after deposition. Co and Mn are imaged in column (b) at ≈ 500 K after different field pulses in the directions indicated by the grey double arrow (H), and in column (c) after cooling down to room temperature. The direction of the incoming x-rays ($h\nu$) and the crystallographic axes are indicated. Note that the sample position is slightly changed between (b) and (c) by thermal drift.

arrow $(\pm H)$ in the figure. One can see that now the Mn domains are magnetized in the same direction as the Co ones. The situation does not change once the sample is imaged again at room temperature, as one realizes by comparing the Co and Mn images of column (c).

These somehow puzzling observations about the induced moments in Mn can be understood considering that the magnetic state of the Mn atoms is very sensitive to oxidization and to the magnetic environment in which they are located. Contradictory reports have been indeed published about the orientation of the magnetization in Mn films on top of FM substrates, either parallel or antiparallel with respect to the substrate [104]. For example submonolayer Mn films are found to be FM on top of Fe at least for a certain thickness range [105, 106]. The Mn magnetization reduces to zero or changes from parallel to antiparallel with respect to the underlying Fe by oxygen absorption [107]. Another mechanism which can influence the amount and the direction of the induced moment in Mn in the $Fe_{50}Mn_{50}$ films is the local interaction of Mn atoms with the Fe and Co atoms. XMCD experiments in absorption reveal that in FM FeMn and CoMn alloys the Mn magnetization is found aligned antiparallel to Fe in $Fe_{90}Mn_{10}$ and parallel to Co in $Co_{90}Mn_{10}$ [108]. This would suggest that preferentially the Mn atoms tend to couple antiferromagnetically to Fe and ferromagnetically to Co, at least when alloying. From these considerations one can therefore expect that local differences in composition or in amount of roughness in nominally identical films may induce a locally different environment in the Mn atoms. For example the number of Fe or Co atoms surrounding the Mn atoms may change. Considering the different interaction that comes into to play, that is Mn-Mn, Mn-Fe, and Mn-Co, one could therefore expect that also the amount and the direction of the induced magnetic moment may change.

Finally an interesting effect present in some of the Mn images is the superposition of regular stripes of alternating contrast on the domain pattern. These are shown in the Mn image of Fig. 3.18. From this figure one sees that the stripes are perpendicular to the gradient of total thickness. Indeed the period of the stripes in the vertical and horizontal directions corresponds to 1 ML in the $Fe_{50}Mn_{50}$ and Co thickness, respectively. In order to clarify the origin of these stripes, an $Fe_{50}Mn_{50}/Co$ double wedge sample, in which stripes in the Mn images were present, was illuminated by a Hg lamp. The resultant image is shown in Fig. 4.12 (a). The $Fe_{50}Mn_{50}$ and Co thickness of the imaged area are indicated at the left axis, increasing from bottom to top, and at the bottom axis, increasing from left to right, respectively. Since the energy of the Hg lamp is around 4.9 eV, the emitted electrons are the ones with a final energy just slightly above the work function of the material. The contrast of Fig. 4.12 (a) therefore arises from local differences in the work function due to structural and topological features of the sample surface, and it does not contain magnetic information. Still the stripes of alternating grey scale contrast are present, which are the same as the ones observed in some of the Mn L_3 images. Obviously now the stripes are not superimposed to the magnetic domains, and the intensity difference of adjacent stripes is higher than in the Mn images. One can therefore perform a scan along the vertical and the horizontal directions in the image, in order to check the stripes' period and compare it to the film thickness. These line scans have been performed along the horizontal and vertical white lines printed in Fig. 4.12 (a), and are reported in Fig. 4.12 (b) and Fig. 4.12 (c), respectively. In (b) the intensity (left axis) is plotted as a function of the Co thickness at the upper axis. In (c) the intensity is at the bottom axis, and the $Fe_{50}Mn_{50}$ thickness at the right axis. One can see that the oscillations in the intensity, corresponding to different grey scale contrast in the stripes, have a period of 1 ML in both the Co and the $Fe_{50}Mn_{50}$ thickness. Actually one can not identify whether a maximum or a minimum in the intensity oscillations corresponds to the completion of an integer number of monolayers. In fact while the 10% error in the thickness calibration is negligible in the determination of the period of the oscillations, it amounts to almost 1 ML in the absolute thickness, therefore impeding to obtain definitive conclusions about the phase of oscillations. Considering the topological origin of the contrast in Fig. 4.12, a possible explanation of stripes of different intensity having a period of 1 ML is the periodic variation of the number of surface steps. During the layer-by-layer growth of the $Co/Fe_{50}Mn_{50}$ sample on Cu(001) the number of surface steps oscillates indeed with monolayer



Figure 4.12: (a) PEEM image obtained by shining with a Hg lamp a $Fe_{50}Mn_{50}/Co$ double wedge bilayer on Cu(001). (b) and (c) display a linescan of the intensity measured along the horizontal and vertical white lines in image (a), respectively.

period. Therefore the intensity oscillations observed in Fig. 4.12 (b) and Fig. 4.12 (c) can have the same origin as the MEED oscillations presented in the previous chapter, and confirm once again the layer-by-layer growth mode for the $Co/Fe_{50}Mn_{50}$ bilayers on Cu(001). The fact that these stripes were observed also in the Mn domain images, and not in the Fe and Co images, can be explained by considering that the topological origin of the stripes may induce an intensity difference between adjacent stripes. In this case a slight difference in intensity between the right and left polarized light can give rise to a non vanishing term when one calculates the asymmetry. Moreover Mn, among the three materials considered here, is the one with lower binding energy. Therefore the edge intensity of Mn from a $Co/Fe_{50}Mn_{50}$ sample has the lowest background, thus possibly enhancing this effect.

Conclusions

The interaction at the interface between a ferromagnetic (FM) and an antiferromagnetic (AFM) material has been studied in this work at $Co/Fe_{50}Mn_{50}$ bilayers, epitaxially grown on Cu(001) single crystal. A layer-by-layer growth mode of the bilayers has been deduced from the presence of oscillations in the medium energy electron diffraction intensity recorded during deposition. The AFM fcc γ phase of the bulk $Fe_{50}Mn_{50}$ alloy is conserved also in thin films at least up to the maximum thickness investigated of 26 ML, as demonstrated by the low energy electron diffraction patterns obtained after deposition of the alloy on Cu(001) and on Co/Cu(001). The magnetic behaviour of the bilayers has been studied by recording magnetization curves, exploiting the magneto-optic Kerr effect, and by imaging magnetic domains, using a photoelectron emission microscope (PEEM) excited by synchrotron radiation with x-ray magnetic circular dichroism (XMCD) as magnetic contrast mechanism. The following main results have been obtained:

- The coercive field of the Co/Fe₅₀Mn₅₀ bilayers displays, at room temperature, a large increase of more than one order of magnitude, when the Fe₅₀Mn₅₀ thickness (t_{FeMn}) overcomes approximately 10 ML. This increase in coercivity is attributed to the coupling of the FM Co film to the AFM Fe₅₀Mn₅₀ film. A 10 ML thick Fe₅₀Mn₅₀ film has therefore an AFM ordering temperature around room temperature. This value is lower than the Néel temperature of the bulk material (≈ 500 K). The temperature and Fe₅₀Mn₅₀ thickness dependence of the increase in coercive field suggest that this is due to the reduced dimensionality of the film with respect to the bulk.
- The magnetic domain configuration of a Co film at room temperature in the as-grown state does not present substantial differences when the Co is either grown on Cu(001) or on top of a Fe₅₀Mn₅₀ film thinner than 10 ML. For $t_{FeMn} > 10$ ML Co presents a totally different domain pattern: the size of the domains is reduced by several orders of magnitude, and it decreases further for increasing Fe₅₀Mn₅₀ thickness until it becomes much smaller than the instrumental resolution (≈ 400 nm). $t_{FeMn} \approx 10$ ML is the thickness at which the large increase in coercive field has been measured at room temperature, due to the coupling of Co with an AFM Fe₅₀Mn₅₀ film. The small domains in Co are therefore frozen during the growth on top of an AFM ordered underlayer. These Co domains reflect the presence

of AFM domains at the surface of the $Fe_{50}Mn_{50}$ film, probably induced by topological features of the sample, such as mono-atomic steps.

- The small domains in Co, frozen during the growth, are metastable: upon annealing at a temperature higher than the AFM ordering temperature, they merge together to form magnetic domains orders of magnitude bigger. After cooling back to a temperature lower than the AFM ordering temperature, the magnetization of the newly formed domains is along the (100) azimuth axes, while a Co film deposited on Cu(001) or coupled to a paramagnetic $Fe_{50}Mn_{50}$ film has an easy axis of magnetization along the (110) in plane directions (this change in easy axis was probably already present in the small domains of the as-grown state, but not evident because of their small size). The change in easy axis, for thin Co films, is visible at room temperature for $t_{FeMn} > 10$ ML, that is when the $Fe_{50}Mn_{50}$ film is in an AFM state. The same observation has been made when the Co film is covered by $Fe_{50}Mn_{50}$. In this case the domain pattern of Co is decided by the growth on top of Cu(001), leading to formation of big domains, which, when coupled to AFM $Fe_{50}Mn_{50}$, have a magnetization directed along the $\langle 100 \rangle$ axes. This change in easy axis can be explained by considering topological domains in the AFM ordered $Fe_{50}Mn_{50}$ film, which has a non-collinear spin structure, in which the spins are pointing along the $\langle 110 \rangle$ in-plane directions. If these domains are smaller than the Co exchange length, an effective 45° coupling between Co and Fe₅₀Mn₅₀ spins is realized, by exchange averaging over monoatomic steps in the $Fe_{50}Mn_{50}$ film. This leads to an easy magnetization direction of Co along the $\langle 100 \rangle$ axes.
- Images of magnetic domains could be detected also from Fe and Mn when the Co film was in contact with Fe₅₀Mn₅₀, both in the PM and the AFM state. In the case of Fe, this induced magnetic moment, if confined at the interface with Co, would amount to approximately half of the interfacial ML. Mn domain images could be detected in which the magnetization was either parallel or antiparallel to Co and Fe. This is related to the sensitivity of the magnetic state of the Mn atoms to the structural and magnetic environment in which they are embedded.

These results suggest that the clue to understand the interaction at an FM-AFM interface is the presence of domains in the AFM material. These domains, even if not observed directly in this work, appear as a natural explanation of the Co domain configuration when coupled to an AFM $Fe_{50}Mn_{50}$ film. The topological origin of the AFM domains requires only little energy for domain formation. An indirect evidence of their importance for the FM-AFM coupling has been given. Statistically the topological domains give rise to locally uncompensated AFM spins at the interface, which are necessary in order to set an exchange bias direction. Part of the FM moments measured in Fe and Mn must indeed consist of these uncompensated spins. However, due to the rather low magnetic signal, it is quite difficult to distinguish the part strongly coupled to the AFM lattice spins, giving rise to the exchange bias effect, and the part rotating upon switching the magnetization of the Co layer. In general, therefore, a domain model seems the more appropriate in order to come to a fundamental understanding of the exchange anisotropy phenomenology, if one takes also into account structural inhomogeneities even at a very small lateral lengthscale.

The results have been obtained mainly thanks to the capability of PEEM, used with XMCD as a contrast mechanism, for element-resolved imaging of magnetic domains and for access to buried layers. In this way domain images could be obtained from all the three elements in the studied bilayers, leading to the observation of the induced moments in Fe and Mn. Moreover, the domain configuration of the Co film, both on top and below the $Fe_{50}Mn_{50}$ layer, could be detected. An explanation that is able to take into account both observations had therefore to be found. A limitation still present in the technique is the impossibility to image directly the domains in the AFM $Fe_{50}Mn_{50}$ film. Efforts could be made in this direction by using PEEM with x-ray linear magnetic dichroism (XMLD) in photoemission. XMLD in photoemission could be used also to check if a general preferred magnetic direction is present in thin AFM $Fe_{50}Mn_{50}$ films. Finally a magnetic domain imaging technique with a much higher lateral resolution, such as scanning electron microscopy with polarization analysis, or spin polarized scanning tunneling microscopy, could be used to better define the dimensions of the Co domains in the as-grown state on top of AFM $Fe_{50}Mn_{50}$. Since these domains are related to the underlying AFM domains, some further considerations on the latter could be possible.

Zusammenfassung

Die Manipulation von Systemen, die ein ferromagnetisches (FM) Material enthalten, kann zur Entdeckung unerwarteter magnetischen Eigenschaften führen, die oft neue interessante Anwendungen ermöglichen. Vor über 40 Jahren wurde mit dem sogennanten Exchange-Bias-Effekt ein Verfahren gefunden, das eine gezielte Veränderung des magnetischen Verhaltens eines FM-Materials ermöglichte: Während die Hystereseschleife eines FM-Materials normalerweise symmetrisch zum Nullpunkt des externen Feldes liegt, weist ein FM Material in Kontakt mit einem antiferromagnetischen (AFM)-Material eine vom Nullpunkt verschobene Hystereseschleife auf. Bei hinreichend großer Verschiebung bewirkt die magnetische Wechselwirkung mit dem AFM-Material, dass im FM-Material nur noch eine einzige im Nullfeld stabile Magnetisierungsrichtung übrigbleibt. Wenn man versucht, die Magnetisierung aus dieser stabilen Richtung heraus zu invertieren, wird mehr Energie benötigt als für die Drehung in die ursprüngliche Richtung. Da diese Anisotropie in der Magnetisierungsrichtung des FM-Materials durch die Austauschwechselwirkung mit dem AFM-Material induziert wird, wird der Energieunterschied zwischen beiden Konfigurationen als Austausch-Anisotropie bezeichnet. Die seitliche Verschiebung der Hystereseschleife, die nur einer der Effekte der FM-AFM-Wechselwirkung ist, wird bereits seit ein paar Jahren in technologischen Anwendungen, unter anderem im Bereich der Datenspeicherung, verwendet. Aufgrund des technologischen Interesses kam es zu verstärkten experimentellen und theoretischen Forschungsaktivitäten, die zum einen ein besseres Verständnis der Grundlagen der beobachteten Phänomene und zum anderen die Verbesserung der Leistungen der Bauelemente anstrebten. Die große Anzahl von Forschungsarbeiten hat jedoch bisher nicht dazu geführt, die Kopplung zwischen einem FM- und einem AFM-Material vollständig zu verstehen.

Um den Exchange-Bias-Effekt zu untersuchen, wurden in dieser Arbeit Co/Fe₅₀Mn₅₀-Doppelschichten epitaktisch auf einem Cu(001)-Einkristall aufgewachsen. Die während des Wachstums aufgezeichneten Oszillationen in der Intensität der Beugung von Elektronen mittlerer Energie (MEED), ließen auf ein van der Merwe-Wachstum der Doppelschichten schließen. Mittels Beugung langsamer Elektronen (LEED) wurde strukturell die fcc- γ -Phase von dünnen AFM Fe₅₀Mn₅₀-Schichten auf Cu(001) und auf Co/Cu(001) verifiziert.

Das magnetische Verhalten der Doppelschichten wurde durch Messungen von Hystereseschleifen analysiert, die mit Hilfe des magneto-optischen Kerr-Effekts aufgenommen wurden. Bei Raumtemperatur zeigt die Koerzitivfeldstärke von Co/Fe₅₀Mn₅₀-Doppelschichten einen Anstieg um mehr als eine Größenordnung, wenn die Fe₅₀Mn₅₀-Dicke (t_{FeMn}) ungefähr 10 atomare Monolagen (ML) überschreitet. Diese Erhöhung der Koerzitivfeldstärke ist auf die Kopplung zwischen der FM-Co- und der AFM-Fe₅₀Mn₅₀-Schicht zurückzuführen. Daraus wird geschlossen, dass die AFM-Ordnungstemperatur eines Fe₅₀Mn₅₀-Films von 10 ML Dicke gerade bei Raumtemperatur liegt. Dieser Wert ist niedriger als die Néel-Temperatur des Volumen-Materials (≈ 500 K). Die gefundene Abhängigkeit der Erhöhung der Koerzitivfeldstärke von der Temperatur und der Fe₅₀Mn₅₀-Dicke weist darauf hin, dass diese Erhöhung eine Folge der reduzierten Dimensionalität des Films im Vergleich zum Volumenkristall ist.

Elementselektive Abbildungen von magnetischen Domänen wurden mittels eines Photoelektronen-Emissionsmikroskops (PEEM) erstellt, das den Zirkulardichroismus bei der Absorption mit zirkular polarisierter Röntgenstrahlung (XMCD) ausnutzt. Die ursprüngliche Domänenstruktur eines bei Raumtemperatur direkt auf Cu(001) aufgewachsenen Co-Films unterscheidet sich nicht merklich von der eines Co-Films, der auf einen weniger als 10 ML dicker Fe₅₀Mn₅₀-Film aufgebracht wurde. Ist $t_{FeMn} > 10$ ML, weist Co eine völlig unterschiedliche Domänenstruktur auf: Die Domänen sind um mehrere Größenordnungen kleiner und werden mit zunehmender Schichtdicke kleiner als die instrumentelle Auflösung von etwa 400 nm. Da bei $t_{FeMn} \approx 10$ ML bei Raumtemperatur auch die Erhöhung der Koerzitivfeldstärke gemessen wurde, lässt sich das so interpretieren, dass die kleinen Co-Domänen während des Wachstums auf der AFMgeordneten Unterlage eingefroren werden. Die Co-Domänen zeichnen die Anwesenheit von AFM-Domänen an der Oberfläche des Fe₅₀Mn₅₀-Films nach, die ihrerseits wahrscheinlich von topologischen Merkmalen der Probe (z.B. monoatomaren Stufen) hervorgerufen werden.

Die kleinen, während des Wachstums eingefrorenen Co-Domänen sind metastabil: Beim Heizen über die AFM-Ordnungstemperatur hinaus verschmelzen sie wieder zu wesentlich größeren magnetischen Domänen. Beim anschließenden Abkühlen unter die AFM-Ordnungstemperatur findet man eine Magnetisierungsrichtung der neu entstehenden Domänen entlang (100)-Azimutachsen, während ein direkt auf Cu(001) aufgewachsener Co-Film oder ein mit einem paramagnetischen (PM) $Fe_{50}Mn_{50}$ -Film gekoppelter Film eine magnetische leichte Richtung entlang (110)-Achsen aufweist. Bei Raumtemperatur findet dieser Wechsel der magnetisch leichten Richtung dann statt, wenn $t_{FeMn} > 10$ ML ist, d.h. wenn sich der Fe₅₀Mn₅₀-Film im AFM-Zustand befindet. Das gleiche wurde festgestellt, wenn der Co-Film mit Fe₅₀Mn₅₀ bedeckt ist. In diesem Fall wird die Domänenstruktur durch das Wachstum auf Cu(001) bestimmt: Wenn der Co-Film mit einem AFM Fe₅₀Mn₅₀-Film bedeckt ist, entstehen Große Domänen, die eine Magnetisierungsrichtung entlang $\langle 100 \rangle$ -Azimutachsen haben. Den Wechsel der magnetisch leichten Richtung kann man erklären, wenn die topologischen (durch monoatomare Stufen induzierten) Domänen im AFM-Fe₅₀Mn₅₀-Film in Betrachtung gezogen werden: Die AFM-Ordnung in $Fe_{50}Mn_{50}$ wird durch eine nicht kollinearen Struktur erreicht, bei der die Spins entlang $\langle 110 \rangle$ -Azimutachsen liegen. Ein Co-Film in Kontakt mit einer monoatomaren Stufe der

Fe₅₀Mn₅₀-Oberfläche spürt die Austauschwechselwirkung entlang der vier $\langle 110 \rangle$ -Azimutachsen. Dies verursacht eine effektive 45°-Kopplung zwischen Co und Fe₅₀Mn₅₀, d.h. eine magnetisch leichte Richtung entlang $\langle 100 \rangle$ -Azimutachsen.

Elementselektive Abbildungen der magnetischen Domänen in diesem System wurden auch für Fe und Mn erzielt, und zwar sowohl im PM- als auch im AFM-Zustand des Fe₅₀Mn₅₀. Das dabei im Fall des Eisens gefundene induzierte magnetische Moment entspricht etwa dem einer halben Grenzflächenlage Fe, wenn man annimmt, dass es komplett an der Grenzfläche zum Co lokalisiert ist. Für die Domänen des Mn wurde sowohl eine parallele, als auch eine antiparallele Magnetisierung zu Fe und Co gefunden. Dies wird mit der Empfindlichkeit des magnetischen Zustands der Mn-Atome gegenüber ihrem strukturellen und magnetischen Umfeld erklärt.

Die Ergebnisse zeigen, dass die Existenz von Domänen im AFM-Material von wesentlicher Bedeutung ist, um die Kopplung zwischen einem FM- und einem AFM-Material erklären zu können. Die AFM-Domänen, obwohl sie in dieser Arbeit nicht direkt abgebildet werden konnten, liefern eine einfache Erklärung für die Co-Domänenstruktur, wenn das Co mit einem AFM-Fe₅₀Mn₅₀-Film gekoppelt ist. In einer statistischen Weise liefern die topologisch bedingten AFM Domänen an der Grenzfläche unkompensierte Spins, die für die Festlegung der Richtung des Exchange Bias verantwortlich sind. Ein Teil des FM-Moments, das in Fe und Mn gemessen wurde, ist als Folge solcher unkompensierter Spins zu verstehen.

Bibliography

- [1] W. H. Meiklejohn and C. P. Bean, Phys. Rev. 102, 1413 (1956).
- [2] W. H. Meiklejohn and C. P. Bean, Phys. Rev. 105, 904 (1957).
- [3] B. D. Cullity, Introduction to magnetic materials, Addison-Wesley, Reading, 1972.
- [4] W. H. Meiklejohn, J. Appl. Phys. 33, 1328 (1962).
- [5] A. Yelon, in *Physics of Thin Films*, vol. 6, edited by M. H. Francombe and R. W. Hoffman, Academic Press, New York, 1971.
- [6] D. Mauri, E. Kay, D. Scholl, and J. K. Howard, J. Appl. Phys. 62, 2929 (1987).
- [7] R. L. Stamps, J. Phys. D: Appl. Phys. 33, R247 (2000).
- [8] J. C. S. Kools, IEEE Trans. Magn. 32, 3165 (1996).
- [9] B. Dieny, V. S. Speriosu, S. S. P. Parkin, B. A. Gurney, D. R. Wilhoit, and D. Mauri, Phys. Rev. B 43, 1297 (1991).
- [10] J. Nogués and I. K. Schuller, J. Magn. Magn. Mater. 192, 203 (1999).
- [11] M. N. Baibich, J. M. Broto, A. Fert, F. Nguyen Van Dau, F. Petroff, P. Eitenne, G. Creuzet, A. Friederich, and J. Chazelas, Phys. Rev. Lett. 61, 2472 (1988).
- [12] G. Binasch, P. Grünberg, F. Saurenbach, and W. Zinn, Phys. Rev. B 39, 4828 (1989).
- [13] C. H. Tsang, R. E. Fontana Jr., T. Lin, D. E. Heim, B. A. Gurney, and M. L. Williams, IBM J. Res. Develop. 42, 103 (1998).
- [14] J. de Boeck and G. Borghs, Physics World, April 1999, pp 27-32.
- [15] S. S. P. Parkin, K. P. Roche, M. G. Samant, P. M. Rice, R. B. Beyers, R. E. Scheuerlein, E. J. O'Sullivan, S. L. Brown, J. Bucchigano, D. W. Abraham, Y. Lu, M. Rooks, P. L. Trouilloud, R. A. Wanner, and W. J. Gallagher, J. Appl. Phys. 85, 5828 (1999).
- [16] A. P. Malozemoff, Phys. Rev. B 35, 3679 (1987).
- [17] D. Mauri, H. C. Siegmann, P. S. Bagus, and E. Kay, J. Appl. Phys. 62, 3047 (1987).
- [18] A. P. Malozemoff, Phys. Rev. B 37, 7673 (1988).

- [19] A. P. Malozemoff, J. Appl. Phys. 63, 3874 (1988).
- [20] N. C. Koon, Phys. Rev. Lett. 78, 4865 (1997).
- [21] T. C. Schulthess and W. H. Butler, Phys. Rev. Lett. 81, 4516 (1998).
- [22] T. C. Schulthess and W. H. Butler, J. Appl. Phys. 85, 5510 (1999).
- [23] A. A. Glazer, A. P. Potapov, R. I. Tagirov, and Ya. S. Shur, phys. stat. sol. 16, 745 (1966).
- [24] Y. J. Tang, B. Roos, T. Mewes, S. O. Demokritov, B. Hillebrands, and Y. J. Wang, Appl. Phys. Lett. 75, 707 (1999).
- [25] C. Leighton, J. Nogués, B. J. Jönsson-Åkerman, and I. K. Schuller, Phys. Rev. Lett. 84, 3466 (2000).
- [26] C. Tsang, N. Heiman, and K. Lee, J. Appl. Phys. 52, 2471 (1981).
- [27] A. E. Berkowitz and K. Takano, J. Magn. Magn. Mater. 200, 552 (1999).
- [28] D. Spanke, V. Solinus, D. Knabben, F. U. Hillebrecht, F. Ciccacci, L. Gregoratti, and M. Marsi, Phys. Rev. B 58, 5201 (1998).
- [29] J. Stöhr, A. Scholl, T. J. Regan, S. Anders, J. Lüning, M. R. Scheinfein, H. A. Padmore, and R. L. White, Phys. Rev. Lett. 83, 1862 (1999).
- [30] A. Scholl, J. Stöhr, J. Lüning, J. W. Seo, J. Fompeyrine, H. Siegwart, J.-P. Locquet, F. Nolting, S. Anders, E. E. Fullerton, M. R. Scheinfein, and H. A. Padmore, Science 287, 1014 (2000).
- [31] H. Ohldag, A. Scholl, F. Nolting, S. Anders, F. U. Hillebrecht, and J. Stöhr, Phys. Rev. Lett. 86, 2878 (2001).
- [32] T. J. Moran, J. Nogués, D. Lederman, and I. K. Schuller, Appl. Phys. Lett. 72, 617 (1998).
- [33] F. Y. Yang and C. L. Chien, Phys. Rev. Lett. 85, 2597 (2000).
- [34] K. Takano, R. H. Kodama, A. E. Berkowitz, W. Cao, and G. Thomas, Phys. Rev. Lett. 79, 1130 (1997).
- [35] W. J. Antel Jr., F. Perjeru, and G. R. Harp, Phys. Rev. Lett. 83, 1439 (1999).
- [36] C. Sánchez-Hanke and C.-C. Kao, J. Magn. Magn. Mater. 226-230, 1803 (2001).
- [37] P. Miltényi, M. Gierlings, J. Keller, B. Beschoten, G. Güntherodt, U. Nowak, and K. D. Usadel, Phys. Rev. Lett. 84, 4224 (2000).
- [38] U. Nowak, A. Misra, and K. D. Usadel, J. Appl. Phys. 89, 7269 (2001).
- [39] J. Wang, W. N. Wang, X. Chen, H. W. Zhao, J. G. Zhao, and W. Sh. Zhan, Appl. Phys. Lett. 77, 2731 (2000).
- [40] J. Wang, W. N. Wang, X. Chen, H. W. Zhao, J. G. Zhao, and W. Sh. Zhan, J. Appl. Phys. 89, 3897 (2001).
- [41] H. Umebayashi and Y. Ishikawa, J. Phys. Soc. Jpn. 21, 1281 (1966).
- [42] Y. Endoh and Y. Ishikawa, J. Phys. Soc. Jpn. 30, 1614 (1971).

- [43] J. S. Kouvel and J. S. Kasper, J. Phys. Chem. Solids 24, 529 (1963).
- [44] S. J. Kennedy and T. J. Hicks, J. Phys. F: Met. Phys. 17, 1599 (1987).
- [45] T. C. Schulthess, W. H. Butler, G. M. Stocks, S. Maat, and G. J. Mankey, J. Appl. Phys. 85, 4842 (1999).
- [46] A. Sakuma, J. Phys. Soc. Jpn. 69, 3072 (2000).
- [47] S. Akbar, Y. Kakehashi, and N. Kimura, J. Phys.: Condens. Matter 10, 2081 (1998).
- [48] D. Spišák and J. Hafner, Phys. Rev. B 61, 11569 (2000).
- [49] R. D. Hemstead, S. Krongelb, and D. A. Thompson, IEEE Trans. Magn. MAG-14, 521 (1978).
- [50] C. Tsang and K. Lee, J. Appl. Phys. 53, 2605 (1982).
- [51] R. Jungblut, R. Coehoorn, M. T. Johnson, Ch. Sauer, P. J. van der Zaag, A. R. Ball, Th. G. S. M. Rijks, J. aan de Stegge, and A. Reinders, J. Magn. Magn. Mater. 148, 300 (1995).
- [52] K.-M. H. Lenssen, A. E. M. De Veirman, and J. J. T. M. Donkers, J. Appl. Phys. 81, 4915 (1997).
- [53] L. Tang, D. E. Laughlin, and S. Gangopadhyay, J. Appl. Phys. 81, 4906 (1997).
- [54] C. M. Schneider, PhD thesis FU Berlin, 1990 (unpublished).
- [55] Handbook of Chemistry and Physics, edited by D. R. Lide, CRC Press, Boca Raton, 2011.
- [56] see for example Practical Surface Analysis, edited by D. Briggs and M. P. Seah, Wiley, Chichester, 1990.
- [57] J. B. Pendry, Low Energy Electron Diffraction, Academic Press, New York, 1974.
- [58] see for example S. D. Bader and J. L. Erskine, in Ultrathin Magnetic Structures II, edited by B. Heinrich and J. A. C. Bland, Springer-Verlag, Berlin, 1994.
- [59] G. Schönhense, J. Phys.: Condens. Matter 11, 9517 (1999).
- [60] Focus IS-PEEM.
- [61] W. Swiech, G. H. Fecher, Ch. Ziethen, O. Schmidt, G. Schönhense, K. Grzelakowski, C. M. Schneider, R. Frömter, H. P. Oepen, and J. Kirschner, J. Electron Spectrosc. Relat. Phenom. 84, 171 (1997).
- [62] C. M. Schneider, J. Magn. Magn. Mater. 175, 160 (1997).
- [63] W. Kuch, R. Frömter, J. Gilles, D. Hartmann, Ch. Ziethen, C. M. Schneider, G. Schönhense, W. Swiech, and J. Kirschner, Surf. Rev. Lett. 5, 1241 (1998).
- [64] S. Anders, H. P. Padmore, A. Scholl, M. R. Scheinfein, J. Stöhr, and J. Lüning, Synchrotron Radiation News 12, 17 (1999).
- [65] J. Stöhr, J. Electron Spectrosc. Relat. Phenom. 75, 253 (1995).
- [66] J. Stöhr, H. A. Padmore, S. Anders, T. Stammler, and M. R. Scheinfein, Surf. Rev. Lett. 5, 1297 (1998).
- [67] R. Nakajima, J. Stöhr, and Y. U. Idzerda, Phys. Rev. B 59, 6421 (1999).

- [68] K. J. S. Sawhney, F. Senf, M. Scheer, F. Schäfers, J. Bahrdt, A. Gaupp, and W. Gudat, Nucl. Instr. and Meth. A 390, 395 (1997).
- [69] M. R. Weiss, R. Follath, K. J. S. Sawhney, F. Senf, J. Bahrdt, W. Frentrup, A. Gaupp, S. Sasaki, M. Scheer, H.-C. Mertins, D. Abramsohn, F. Schäfers, W. Kuch, and W. Mahler, Nucl. Instr. and Meth. A 467-468, 449 (2001).
- [70] W. Kuch, J. Gilles, F. Offi, S. S. Kang, S. Imada, S. Suga, and J. Kirschner, J. Electron Spectrosc. Relat. Phenom. 109, 249 (2000).
- [71] J. H. Neave, B. A. Joyce, P. J. Dobson, and N. Norton, App. Phys. A 31, 1 (1983).
- [72] S. T. Purcell, B. Heinrich, and A. S. Arrott, Phys. Rev. B 35, 6458 (1987).
- [73] C. M. Schneider, A. K. Schmid, P. Schuster, H. P. Oepen, and J. Kirschner, in Magnetism and Struture in Systems of Reduced Dimension, edited by R. F. C. Farrow et al., Plenum Press, New York, 1993.
- [74] C. M. Schneider, P. Bressler, P. Schuster, J. Kirschner, J. J. de Miguel, R. Miranda, and S. Ferrer, Vacuum 41, 503 (1990).
- [75] P. Krams, F. Lauks, R. L. Stamps, B. Hillebrands, and G. Güntherodt, Phys. Rev. Lett. 69, 3674 (1992).
- [76] R. Mégy, A. Bounouh, Y. Suzuki, P. Beauvillain, P. Bruno, C. Chappert, B. Lecuyer, and P. Veillet, Phys. Rev. B 51, 5586 (1995).
- [77] W. Weber, A. Bischof, R. Allenspach, Ch. Würsch, C. H. Back, and D. Pescia, Phys. Rev. Lett. 76, 3424 (1996).
- [78] R. Jungblut, R. Coehoorn, M. T. Johnson, J. aan de Stegge, and A. Reinders, J. Appl. Phys. 75, 6659 (1994).
- [79] see for example P. Poulopoulos and K. Baberschke, J. Phys.: Condens. Matter 11, 9495 (1999).
- [80] C. M. Schneider, P. Bressler, P. Schuster, J. Kirschner, J. J. de Miguel, and R. Miranda, Phys. Rev. Lett. 64, 1059 (1990).
- [81] see for example P. Bayer, S. Müller, P. Schmailzl, and K. Heinz, Phys. Rev. B 48, 17611 (1993).
- [82] Binary Alloy Phase Diagrams, edited by T. B. Massalski and H. Okamoto, ASM International, Ohio, 1990.
- [83] C. Hwang, R. H. Geiss, and J. K. Howard, J. Appl. Phys. 64, 6115 (1988).
- [84] G. Choe and S. Gupta, Appl. Phys. Lett. 70, 1766 (1997).
- [85] S. S. P. Parkin and V. S. Speriosu, in *Magnetic Properties of Low-Dimensional Systems II*, edited by L. M. Falicov, F. Mejía-Lira, and J. L. Morán-López, Springer-Verlag, Berlin, 1990.
- [86] T. Ambrose and C. L. Chien, Phys. Rev. Lett. 76, 1743 (1996).
- [87] D. Alders, L. H. Tjeng, F. C. Voogt, T. Hibma, G. A. Sawatzky, C. T. Chen, J. Vogel, M. Sacchi, and S. Iacobucci, Phys. Rev. B 57, 11623 (1998).
- [88] M. D. Stiles and R. D. McMichael, Phys. Rev. B 63, 064405 (2001).

- [89] O. A. Khomenko, I. F. Khil'kevich, G. Ye. Zvigintseva, L. A. Vaganova, and M. M. Belenkova, Phys. Met. Metall. 47, 180 (1980).
- [90] F. Nolting, A. Scholl, J. Stöhr, J. W. Seo, J. Fompeyrine, H. Slegwart, J.-P. Locquet, S. Anders, J. Lüning, E. E. Fullerton, M. F. Toney, M. R. Scheinfein, and H. A. Padmore, Nature 405, 767 (2000).
- [91] G. van der Laan, B. T. Thole, G. A. Sawatzky, J. B. Goedkoop, J. C. Fuggle, J.-M. Esteva, R. Karnatak, J. P. Remeika, H. A. Dabkowska, Phys. Rev. B 34, 6529 (1986).
- [92] P. Kuiper, B. G. Searle, P. Rudolf, L. H. Tjeng, and C. T. Chen, Phys. Rev. Lett. 70, 1549 (1993).
- [93] Y. Imry and S.-k. Ma, Phys. Rev. Lett. 35, 1399 (1975).
- [94] F. Bensmina, A. Dinia, C. Mény, P. Panissod, and D. Muller, J. Magn. Magn. Mater. 226-230, 473 (2001).
- [95] L. Xing and Z. Yansheng, J. Phys.: Condens. Matter 6, L739 (1994).
- [96] W. Weber, R. Allenspach, and A. Bishof, Appl. Phys. Lett. 70, 520 (1997).
- [97] J. C. Slonczewski, Phys. Rev. Lett. 67, 3172 (1991).
- [98] L. I. Chelaru, private communication.
- [99] C. Mathieu, M. Bauer, B. Hillebrands, J. Fassbender, G. Güntherodt, R. Jungblut, J. Kohlhepp, and A. Reinders, J. Appl. Phys. 83, 2863 (1998).
- [100] S. Riedling, M. Bauer, C. Mathieu, B. Hillebrands, R. Jungblut, J. Kohlhepp, and A. Reinders, J. Appl. Phys. 85, 6648 (1999).
- [101] The Fe/W(001) sample was imaged with a similar PEEM-beamline setting as the $Co/Fe_{50}Mn_{50}$ bilayers.
- [102] U. Gradmann and R. Bergholz, Phys. Rev. Lett. 52, 771 (1984).
- [103] M. G. Samant, J. Stöhr, S. S. P. Parkin, G. A. Held, B. D. Hermsmeier, F. Herma, M. van Schilfgaarde, L.-C. Duda, D. C. Mancini, N. Wassdahl, and R. Nakajima, Phys. Rev. Lett. 72, 1112 (1994).
- [104] see for example Y. Yonamoto, T. Yokoyama, K. Amemiya, D. Matsumura, and T. Ohta, Phys. Rev. B 63, 214406 (2001), and reference therein.
- [105] see for example S. Andrieu, M. Finazzi, Ph. Bauer, H. Fisher, P. Lefevre, A. Traverse, K. Hricovini, G. Krill, and M. Piecuch, Phys. Rev. B 57, 1985 (1998), and references therein.
- [106] see for example T. Igel, R. Pfandzelter, and H. Winter, Phys. Rev. B 58, 2430 (1998), and references therein.
- [107] O. Rader, C. Pampuch, W. Gudat, A. Dallmeyer, C. Carbone, and W. Eberhardt, Europhys. Lett. 46, 231 (1999).
- [108] F. Offi, X. Gao, W. Kuch and J. Kirschner, unpublished.

Appendix A

Curriculum vitae

Personal data:

Name:	Francesco Offi
Date of birth:	October 09, 1971
Place of birth:	Canale (CN), Italy
Marital status:	Married

Education:

Sept. 1977 – June 1982	Primary school of Canale (CN), Italy.
Sept. $1982 - June 1985$	Secondary school of Alba (CN), Italy.
Sept. 1985 – July 1990	High school at "Ginnasio-Liceo San Paolo", Alba (CN), Italy.
Sept. 1991 – March 1997	Faculty of Physics at the "Università degli Studi", Torino, Italy. Degree in Physics with the thesis: Study and opti- mization of the magnetic and structural properties of rapidly solidified alloys
Oct. 1998 – Oct. 2001	Ph.D. candidate at Max-Planck-Institut für Mikrostruktur- physik, Halle, Germany. Supervisor: Prof. Dr. Jürgen Kirschner

Halle, October 30, 2001

Francesco Offi
Appendix B

Erklärung

Ich versichere hiermit, die vorliegende Dissertation

Magnetic interaction between antiferromagnetic and ferromagnetic films: $Co/Fe_{50}Mn_{50}$ bilayers on Cu(001)

selbständig verfasst und keine anderen als die von mir angegebenen Quellen und Hilfsmittel verwendet zu haben. Die den benutzten Werken wörtlich oder inhaltlich entnommennen Stellen sind als solche kenntlich gemacht.

Halle, 30. Oktober 2001

Francesco Offi

Appendix C

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I really do not know where to start to thank Dr. Wolfgang Kuch. He introduced and carried me in the field of thin films magnetism. In these three years he taught me and corrected me. He followed each step of my work with high scientific professionalism and infinite patience. I am deeply in debt with him. Grazie Wolfgang.

I must thank Ms. Birgitt Zada. She repaired many broken things, explained and helped me in understanding a lot of technical problems. Many times she also warned me in a way to prevent the problems.

I am grate to Mr. Liviu Chelaru, not only because he has carried some "East-Latin" temperament in the group, but over all because if we obtained some good PEEM images is also thanks to him. For the same reason I am in debt with Dr. Masato Kotsugi and Mr. Keiki Fukumoto, my tennis teacher. Nights and days together during beamtimes have led to some results.

I express my gratitude to Dr. T. Igel, Dr. R. Pfandzelter, and Prof. H. Winter of the Humboldt-Universität. They suggested me the first way I used to evaporate Mn, and they gave me the first Mn pieces.

I have to thank many colleagues at the MPI, in particular the ones of the AG Bessy in Berlin, with which I worked in strict contact. Dr. Martina Seider, Mr. Jörg Gilles, and Dr. Shisou Kang were always available to help me to solve many doubts, especially at the beginning of my PhD position. From Dr. Xingyu Gao I learned the bases of working with an UHV chamber, and I had a lot of fun with him too. I want to thank Dr. Robert Frömter, for his clarity in explanations of scientific problems and his wide availability (from badminton to Zusammenfassung). I am in debt with Dr. Rüdiger Vollmer, Dr. Wulf Wulfhekel, and Dr. Haifeng Ding for the explanations

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