Surface Science 189/190 (1987) 710–723
North-Holland, Amsterdam

EXPERIMENTAL STUDIES OF SURFACE MAGNETISM WITH POLARIZED ELECTRONS

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Received 6 April 1987; accepted for publication 15 April 1987

The same electron spectroscopies that are so powerful for studying surfaces generally, can be made sensitive to magnetic properties when electron spin polarization is included as a parameter, for example by probing with a spin polarized electron beam or measuring the spin polarization of emitted electrons. Such properties as the spontaneous magnetization, Curie temperature, temperature dependence of the magnetic order, anisotropy, spin-dependent electronic structure, magnetization curves, elementary excitations, and magnetic microstructure may be different from the bulk in a thin film or at the surface of a semi-infinite ferromagnet. Recent results on chemisorption induced changes in surface magnetism studied by spin polarized inverse photoemission, on magnetic surface anisotropy investigated by polarized photoemission, and on magnetic microstructure determined by scanning electron microscopy with polarization analysis, have been selected for discussion as illustrations of polarized electron studies of surface magnetism.

1. Introduction

The magnetic properties of low-dimensional systems, such as the surface of a semi-infinite ferromagnet or a ferromagnetic thin film, may differ substantially from those of a three-dimensional bulk ferromagnet. Factors contributing to the different properties include the lower symmetry, the absence of translational periodicity normal to the surface, and the different number and possibly even different arrangement of neighboring atoms at a surface. Segregation of a species to the surface or chemisorption at the surface can induce changes in magnetic properties. The study of the special magnetic properties of surfaces and other low-dimensional systems is of both fundamental and technological interest. Advances in theory and computational facilities now allow specific predictions of surface and thin film magnetic behavior. New capabilities for growth of materials by atomic and molecular beam epitaxy make possible the atomic engineering of specimens with unique properties. The timely developments in computation, materials preparation, and magnetic measurement techniques using polarized electrons join to make the investigation of magnetic properties of low-dimensional systems especially exciting and rich in opportunity.

0039-6028/87/$03.50

The key feature of a ferromagnet that makes electron spin polarization measurements so important in probing magnetic properties in that by definition a ferromagnet (or ferrimagnet) exhibits a net spin density. That is, within a domain, more electron spins are oriented in one direction along the magnetization axis than the other. In fact, in transition metal ferromagnets where the orbital contribution to the ferromagnetism is largely quenched, the magnetization is proportional to the net spin density $n\uparrow - n\downarrow$,

$$M = -\mu_B(n\uparrow - n\downarrow), \tag{1}$$

where $n\uparrow(n\downarrow)$ is the density of spin up (down) electrons in the material. The minus sign arises because the electron spin $\boldsymbol{s}$ (units of $h/2$) and the electron spin magnetic moment (units of Bohr magneton, μ_B) are in opposite directions, $\boldsymbol{\mu} = -\mu_B \boldsymbol{s}$. Thus the magnetization can be determined if it is possible to probe the net spin density by ejecting the electrons into vacuum and measuring their spin polarization $\boldsymbol{P}$. A spin analyzer can be used to measure each component of the polarization which, along the z axis for example, is given by

$$P_z = \frac{N\uparrow - N\downarrow}{N\uparrow + N\downarrow} \tag{2}$$

where $N\uparrow(N\downarrow)$ is the number of electrons parallel (antiparallel) to the z direction. From eq. (1), the polarization $\boldsymbol{P}$ so determined is proportional to the magnetization $\boldsymbol{M}$ and in the opposite direction. Photoemission, field emission, secondary electron emission, and Auger electron emission have all been used to eject electrons from the material for polarization measurement.

Alternatively, information on magnetic properties is obtained if, instead of ejecting electrons from the specimen, a beam of polarized electrons is directed at the surface. There is a spin dependent scattering potential caused by the exchange interaction, which ultimately is a consequence of the Pauli exclusion principle. The intensity of the scattered electrons depends on the relative orientation of the spin polarization of the incident beam and the net spin density of the material. An incident polarized electron may lose energy by making a transition into an unoccupied state of the same spin at a lower energy and simultaneously emit a photon. This "inverse photoemission" process probes the net spin density of unoccupied states complementing spin polarized photoemission which probes the spin density of occupied states.

Electron spin polarization can be measured as an additional parameter besides the usual measurements of intensity and momentum (energy and direction) in electron spectroscopies. With the high surface sensitivity of electrons having energies between a few eV and several hundred eV, polarized electron measurements provide superb probes of the magnetic properties of the outer few atomic layers of a material. The availability of intense beams of polarized electrons [1,2] coupled with improvements of Mott spin analyzers and the development of low energy spin analyzers [3,4] facilitates the addition of spin polarization as a parameter in most electron spectroscopies.

In the next section, a brief survey is given of magnetic properties and the spin polarized electron techniques used to investigate them. In sections 3–5, some results of work reported in the past year are used to illustrate the application of spin polarized electron techniques to investigate three surface magnetic properties: chemisorption induced changes in surface magnetism, magnetic surface anisotropy, and magnetic microstructure. The article concludes with a brief look to the future in section 6.

2. Overview of magnetic properties and polarized electron techniques

For each surface or thin film system we would like to determine a number of magnetic properties which are listed below. The salient features of polarized electron techniques used to measure these properties are highlighted. It is beyond the scope of this paper to present a comprehensive survey; the aim is rather to give a feel for the variety of magnetic questions and the polarized measurement techniques to address them. A number of excellent reviews provide further coverage and comprehensive references to work in this rapidly growing field [2–8]. The following are the magnetic properties to be investigated:

(1) Spontaneous magnetization – Is the sample magnetic or not? Is the moment enhanced by the lower dimension of the system or by an artificially expanded lattice constant (negative pressure)? Although any polarized electron technique is sensitive to the existence of magnetism, the *element specific* nature of polarized Auger spectroscopy [9] is advantageous to separate the magnetic behavior, for example of a material of interest from a magnetic substrate.

(2) Temperature dependence of magnetic order – What is the Curie temperature and temperature dependence of the magnetization? The *simplicity* and *efficiency* of polarized electron scattering, where only the asymmetry in the intensity of elastically scattered electrons needs to be measured, has been used to advantage in determining low-temperature spin deviations [10], critical exponents [11], and surface Curie temperatures different from the bulk [12].

(3) Electronic states – What is the spin dependence of the electronic structure? Polarized photoemission [6,13] and inverse photoemission [14,15] determine the energy distribution of spin dependent states contributing to the magnetism, i.e. an *energy distribution of magnetic states*.

(4) Magnetic surface anisotropy [16] – Spin polarized photoemission has been used [17] as discussed below to investigate whether the surface anisotropy is perpendicular to the surface or parallel to it.

(5) Elementary excitations – What is the spin wave dispersion at the surface? What is the spectrum of Stoner excitations? Spin polarized inelastic scattering [18,19] has been used to investigate Stoner excitations. The maximum information is obtained when a polarized incident beam is scattered and its change in energy and polarization measured [20].

(6) Structure – What is the spatial variation of the magnetization from the surface into the bulk? Is there a layer-dependent magnetic moment? Measurement of spin polarized secondary electron emission at different primary and secondary electron energies has been used for *magnetic depth profiling* [8,21]. Careful analysis of spin polarized LEED intensities gives information on the layer-by-layer magnetic moment [22].
(7) Magnetic domains – What size and shape do the magnetic domains take to minimize the total free energy? The *very high spatial resolution* of scanning electron microscopy with polarization analysis (SEMPA) [23,24] provides images of the domain microstructure as discussed below.
(8) Magnetization curves – Is the remanant magnetization and coercive field at the surface different from the bulk? Polarized electron scattering [25] and secondary electron emission measurements [9] have been used to measure surface hysteresis curves. Secondary electron emission depth profiling has obtained evidence for domain nucleation at the surface [21].
(9) Dynamics – What is the temporal variation of the magnetization when the applied field is changed? Polarized photoemission with nanosecond laser pulses provides an extremely *fast* magnetic measurement [26].
(10) Chemistry – How does the adsorption or segregation of an impurity layer at the surface affect the magnetic and electronic properties? Polarized Auger emission [27], polarized photoemission [28] and, as discussed below, inverse photoemission [14,29] have many advantages for studying chemical effects at magnetic surfaces.

3. Chemisorption induced changes in surface magnetism

The sensitivity of the magnetic properties of a surface to adsorbed foreign atoms has been explored for many years by using bulk magnetic measurements on high surface area samples [30]. Now, using polarized electron spectroscopies it is possible to investigate the correlated effect of chemisorption on surface magnetism and electronic structure. For example, consider the chemisorption of CO on a Ni(110) surface studied by spin polarized inverse photoemission spectroscopy (SPIPES) [29]. The majority spin d-electron states in Ni are filled whereas the minority spin d states are only partially filled such that approximately half an unfilled state (minority spin d hole) per Ni atom is contained in a rather flat band just above E_F. These d holes are clearly central to ferromagnetism in Ni. As demonstrated in the first SPIPES measurements [31], there is a strong peak in the photon intensity when incoming minority spin electrons make transitions into the unfilled d holes.

The changes in the d hole spectrum as a function of CO chemisorption are shown in the spin resolved spectra of fig. 1. In these measurements the energy of the incident electron beam is varied and transitions at a fixed photon

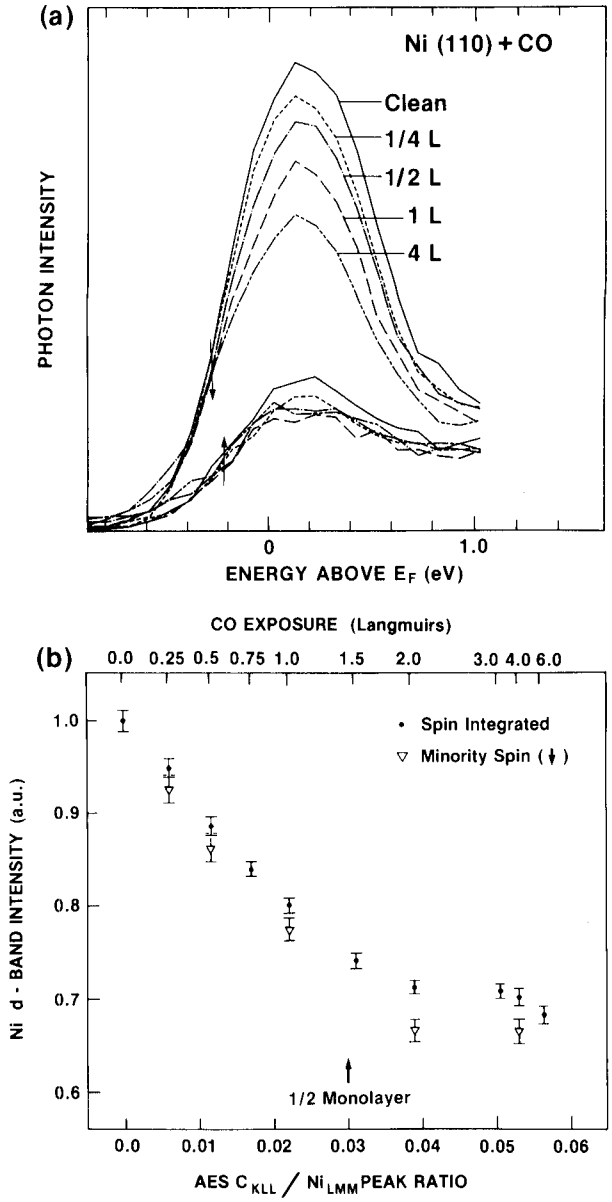

Fig. 1. (a) Spin resolved inverse photoemission spectra of clean Ni(110) and with different exposures of CO. Representative photon intensity spectra are shown for an incident electron beam with majority (↑) and minority (↓) spin polarization as a function of the energy of the final state. (b) The d-band peak intensity is plotted as a function of coverage given by the AES C(272 eV)/Ni(848 eV) peak ratio (from ref. [29]).

energy, $\hbar\omega = 9.7 \pm 0.35$ eV, are recorded and plotted with respect to the energy above the Fermi level of the final state of the transition. There is some photon intensity due to transitions to unfilled sp states which gives the small peak on the background in the majority ($\uparrow$) spin spectrum. The spectrum is dominated by transitions to the d holes in the minority ($\downarrow$) spin spectrum which we will focus on here. The complete set of data is summarized in fig. 1b where the d band intensity is plotted as a function of CO coverage measured by Auger electron spectroscopy (AES) peak ratios. The d band intensity of spin integrated spectra is also shown since it was ascertained that the changes that take place primarily reflect changes in transitions to minority states.

From consideration of fig. 1 we see that the reduction of surface magnetism on CO chemisorption is not due simply to a disruption of the long range order of the magnetic moments. Disordered moments would have no net spin density and the majority spin spectrum and minority spin spectrum would be the same. There is clearly no tendency in this direction as CO coverage is increased. Rather the data point to a reduction of the Ni moment by a filling of the minority spin d holes, i.e. the minority spin band is pulled below the Fermi level, by CO chemisorption. Another noteworthy feature can be seen in fig. 1b: the Ni d-band intensity decreases to about 0.7 of its initial value at a coverage of 0.5 ML and then does not decrease further with increasing CO coverage. That the CO coverage continues to increase is further evidenced by a spin independent peak in the spectra at 3.7 eV above E_F due to transitions to a CO π^* antibonding state; the intensity of this peak, not shown in fig. 1, was found to increase monotonically with coverage over the range measured. The saturation of the decrease in the d-hole peak at 0.5 ML coverage is significant. It suggests one CO molecule effectively eliminates the equivalent of the magnetic moment of two Ni atoms.

4. Magnetic surface anisotropy

The magnetocrystalline anisotropy determines the easy axes of magnetization of a material and strongly influences its magnetic reversal properties. The magnetocrystalline anisotropy arises from an interplay between the spin–orbit coupling and the local crystalline electric field. The lower symmetry at the surface, as opposed to the bulk, produces an additional lower symmetry surface anisotropy term, which in many cases can be larger than the bulk. For a high symmetry surface, i.e. (001), (111), this energy is given by

$$\sigma = K_s \cos^2\theta \tag{3}$$

where K_s is the surface anisotropy constant and θ is the angle between the spontaneous magnetization M_s and the surface normal [16]. When $K_s < 0$, the energy is minimized for $\theta = 0$ corresponding to perpendicular magnetization,

and when $K_s > 0$ the energy is minimized for $\theta = \pi/2$ or in-plane magnetization.

Both measurements [17,32] and calculations [33] of the past year suggest the possibility of large surface anisotropies. Calculations for a single layer of Fe(100) indicate a surface anisotropy K_s that is negative and approximately 100 times that of the bulk. The negative surface anisotropy favors out-of-plane magnetization for a single layer Fe film and competes with the shape anisotropy corresponding to an energy $2\pi M_s^2 V$ that increases with volume (V) such that the magnetization lies in-plane for thicker films. Jonker et al. [32] observed no in-plane component of spin polarization of photoemitted electrons for a 2.5 ML Fe film as shown in fig. 2a. They suggested that this could be explained by the presence of a strong perpendicular anisotropy leading to an out-of-plane magnetization to which the measurements were not sensitive. The Fe films were grown epitaxially on a Ag(100) substrate. For thicker (5

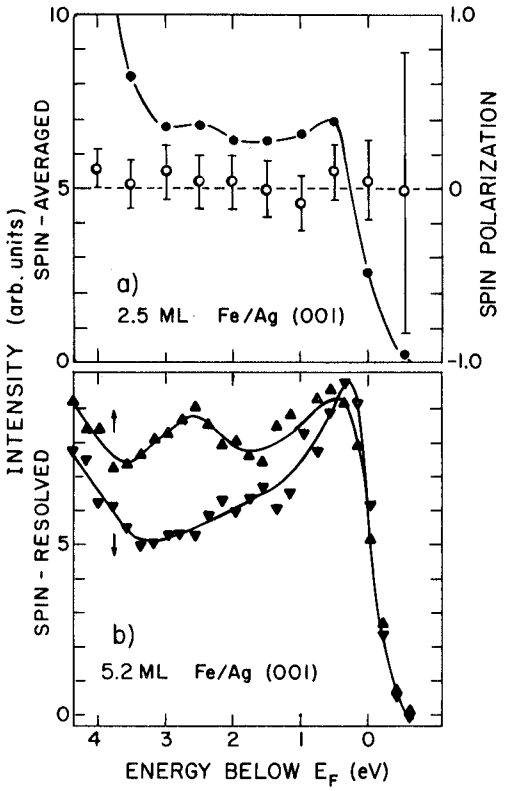

Fig. 2. (a) Spin integrated energy distribution curve (EDC) (closed circles) and spin polarization (open circles) versus binding energy for a 2.5 ML Fe film on Ag(001). (b) Spin resolved EDC's (majority spin, minority spin) for a 5.2 ML Fe film (from ref. [32]).

ML) films the magnetization lies in the plane and the characteristic exchange-split electronic structure is apparent in fig. 2b. It is spin, energy, and angle resolved measurements such as these that allow the determination of the energy position of the electronic states which contribute to the magnetism. An alternative explanation [32] of the data is that the Curie temperature of the thin film is reduced below the temperature of the measurements (room temperature). The cause of this absence of in-plane remanent magnetization at room temperature for the 2.5 ML film remains to be clarified by further experiments.

Spin polarized photoemission measurements of a different type have been carried out very recently for thin films of Fe on Cu(100) [17]. The spin polarization of threshold photoelectrons originating from near E_F was measured as a function of the applied magnetic field normal to the surface. Considering first fig. 3b, it is clear that at 30 K a 1 ML film is ferromagnetic as indicated by the saturation above 5 kOe. The behavior is very different from that expected for a film with only shape anisotropy. In that case, the polarization would increase linearly up to the demagnetizing field of 22 kOe

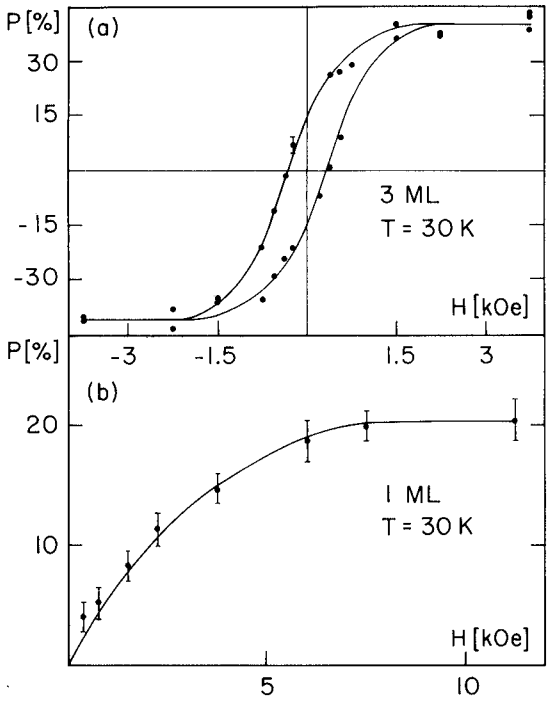

Fig. 3. Polarization $P(H)$ at 30 K versus the applied field H perpendicular to the plane of the 3 ML Fe film on Cu(001) (a) and for 1 ML (b) (from ref. [17]).

and then saturate. The curvature of the polarization curve and saturation at much lower applied field implies a strong contribution from a perpendicular surface anisotropy.

There is hysteresis in the magnetization curve of the 3 ML film shown in fig. 3a and remanent magnetization is observed. For a 5 ML film, a remanent magnetization equal to the saturation magnetization was observed at 30 K, showing that the surface anisotropy is larger than the shape anisotropy; no remanence was observed at room temperature. The anisotropy depends on the temperature and film thickness. Owing to the global nature of the magnetization (hysteresis) curve, it is difficult to infer details about the anisotropy. What is needed is additional information on the nature of the magnetic domains in such thin films and the mechanism of magnetization reversal. A measurement of the spin polarization (in-plane and out-of-plane components) with high spatial resolution as a function of temperature would be ideal; a possible way to accomplish this is described in the next section.

5. Magnetic microstructure

In general, macroscopic magnets break up into domains of uniform magnetization in order to minimize the free energy. The free energy includes contributions from the exchange energy, the magnetostrictive energy, the anisotropy energy, the demagnetization energy, and (in an applied field) the magnetostatic energy. Because there are so many terms in the free energy which depend in detail on material properties and geometry, it is impossible to calculate the domain configuration, except in the most idealized cases, and one must rely instead on observation. Drawbacks of domain imaging techniques include limited spatial resolution of optical methods, restriction to thinned ($\sim$ 100 nm) samples in transmission electron microscopy techniques, and until recently rather poor contrast in scanning electron microscopy (SEM) methods. A few years ago, the first measurements of the energy distribution of the spin polarization of secondary electrons from a ferromagnet showed the high polarization of the lowest energy electrons [34,35]. The advantages for domain imaging of measuring the polarization of secondary electrons in a SEM – high spatial resolution, high contrast, accessibility of the magnetic domain image simultaneously with but independently of the topographic image – were pointed out [34]. The first measurements were made using a Mott spin analyzer by Koike and Hayakawa [36] and subsequently using a compact low energy spin analyzer by Unguris et al. [37].

The principle of scanning electron microscopy with polarization analysis (SEMPA) is shown schematically in fig. 4. The polarization of the secondary electrons generated in the region of the finely focused electron beam of the SEM is proportional to the magnetization at that point from eq. (1). An

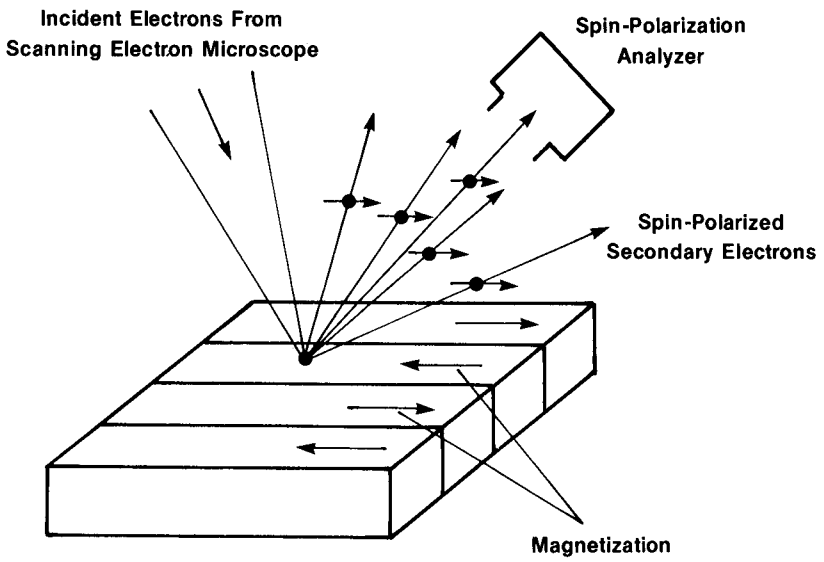

Fig. 4. Principle of Scanning Electron Microscopy with Polarization Analysis (SEMPA).

estimate of the polarization is found by considering that the low energy electrons are the result of exciting many electron–hole pairs in the valence band. The polarization of a representative sample of valence band electrons is $P = n_B/n$ where n_B is the spin density or Bohr magneton number/atom and n is the number of valence electrons/atom; for Fe we have $P = 2.2/8 = 0.28$.

To illustrate the SEMPA technique, a domain image from a Fe–3%Si single crystal is shown in fig. 5. This relatively low magnification image (in contrast to higher magnification images [3,23,24]) was selected because of the interesting domain pattern. This is nominally a (001) surface cut approximately 4° off

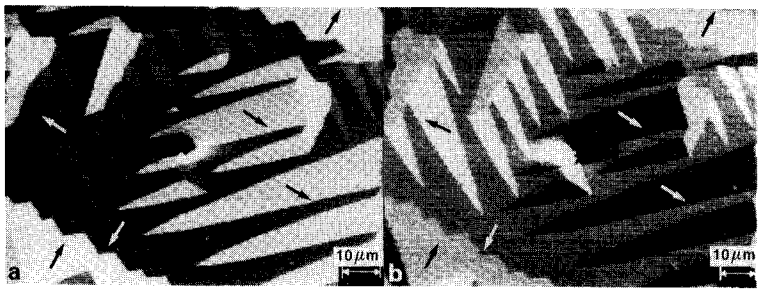

Fig. 5. Images (a) and (b) from measuring two in-plane components of the polarization in an Fe–3% Si single crystal. The gray levels give the four different magnetization directions in the domains as marked by the arrows (from ref. [24]).

the [100] easy axis of magnetization. This leads to the very striking, irregular domain patterns, including the so called "fir tree" pattern. The domains are typical closure domains which form at the surface boundary to minimize the energy.

The four gray levels in fig. 5 correspond to the four easy directions of magnetization in the plane of the surface which leads to four possible orientations of the domains as indicated by the arrows. Four gray levels are observed because the axis of spin analysis is not along an easy direction of magnetization; the magnitude of the secondary electron polarization P in each domain is reduced by the cosine of the angle between the magnetization direction and the spin analysis axis. Fig. 5b shows the complementary image obtained by measuring the orthogonal component of polarization. By deflecting the beam of secondary electrons to an orthogonal spin analyzer, the component of polarization normal to the specimen surface can also be measured.

It should be noted that because the magnetic information is contained in the polarization of the secondary electrons which come from the outer atomic layers, the specimen must be cleaned and maintained in ultrahigh vacuum (UHV). Thus a UHV SEM or scanning Auger system is most suitable for SEMPA measurements. Standard surface science techniques are used to prepare specimens; the Fe surface imaged in fig. 5 was cleaned by cycles of Ar ion bombardment followed by annealing to 600 °C.

Under some circumstances, Hubert [38] has predicted that a domain wall may be Bloch-like in the bulk but Neel-like at the surface in order to minimize stray fields at the surface. The observation of Neel walls is usually restricted to thin film systems ($\lesssim 100$ nm) where it is energetically favorable for the magnetic moments to remain in the plane of the film as the moments alter direction from one domain to the next. In contrast, in a Bloch wall, the magnetic moments rotate in the plane of the wall. In a study of an amorphous ferromagnet, an Fe-based metglass ribbon 25 μm thick, we observed the structure predicted by Hubert as shown in the two orthogonal polarization images displayed in figs. 6a and 6b. The image on the left shows two domains. The magnetization direction and the spin analysis axis are parallel to the boundary between the light and dark areas. In the image on the right, the spin analysis axis is orthogonal to that in fig. 6a and we see the black and white domain areas of fig. 6a are now a constant gray level. However, along the domain boundary a light area indicating a wall region about 0.5 μm wide is observed. In this region the magnetic moments have rotated in the surface plane perpendicular to the domain wall boundary. Thus the wall, which is expected to be Bloch-like in the middle of the ribbon, is Neel-like at the surface as predicted by Hubert. Similar observations have been made by Koike et al. [39] for a 1 μm thick Co based amorphous film and a 1.4 μm thick Permalloy film.

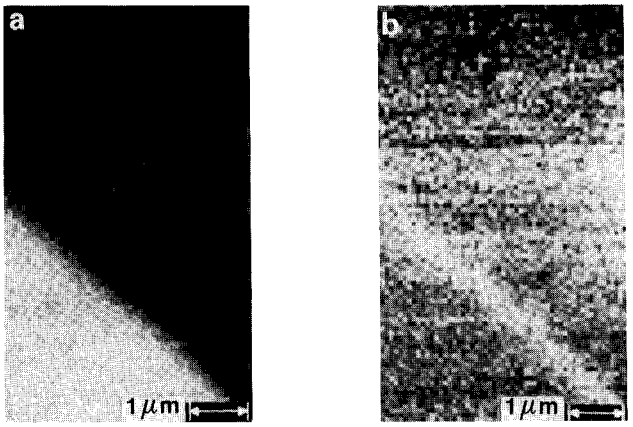

Fig. 6. Image of the polarization along the line of the domain wall (a) and perpendicular to it (b). The approximately 0.5 μm wide wall observed in (b) indicates a Neel-like wall in the surface region (from ref. [24]).

The potential of SEMPA has been clearly demonstrated for addressing many scientifically interesting and technologically important challenges. Of obvious interest is the nature of domains in films of a few monolayers such as the Fe/Cu(100) and Fe/Ag(100) systems discussed above. Can the consequences of perpendicular anisotropy be seen directly in domain images and what is the nature of magnetization reversal in such films? Much remains to be learned about the structure and movement of domain walls and the influence of reduced dimensions of magnetic structures on domain configuration. Many of the technological questions are closely related. In the quest for ever increased density of magnetic information storage, a technique such as SEMPA is ideal for observing the domains and domain walls. For example, a zig-zag domain wall may be the ultimate limitation on the abruptness of the information carrying transition from one magnetic state to another and hence a source of noise. SEMPA combined with scanning Auger to give magnetization, topographic, and chemical composition maps of an area of interest will be very important not only in improving magnetic recording media and devices but also in investigating the structure and origin of coercivity in permanent magnets like $Nd_2Fe_{14}B$.

6. Future prospects

The utility of spin polarized electron techniques to study magnetic properties of ferromagnetic surfaces and thin films has been demonstrated in the discussion of the three examples above. The study of low-dimensional mag-

netism is particularly exciting right now. We are in a new area where materials can be atomically engineered to tailor arrangements and environments of atoms to achieve novel properties. Expitaxially grown films may, in addition to being controlled in thickness, have a different lattice constant or even a different crystal structure. Electron beam and *X*-ray lithography techniques allow control of the lateral dimensions of structures on a nanometer scale. Thus systems can be fabricated to study the fundamental role of reduced size and dimensionality on magnetism. It is possible to test the specific predictions about the magnetic properties of such systems which can now be made owing to theoretical and computational advances. There have been significant advances in electron spin polarization analyzers and in sources of spin polarized electrons. The imminent commercial availability of such devices is expected to stimulate further the widespread use of spin polarized electrons in studies of surface magnetism. In addition to the many fundamental scientific questions, there are challenging technological questions to be studied in order to achieve increased density of information storage, smaller magnetically active elements, and improved device characteristics. Coupled with the economic importance of magnetic technology and the intrinsic fundamental interest in low dimensional magnetism, the study of surface and thin film magnetism with polarized electrons is expected to continue to be an exciting and fruitful area of research in the coming years.

Acknowledgements

The stimulus of my colleagues in the Electron Physics Group at the National Bureau of Standards and their contribution to the work reviewed in this article are gratefully acknowledged. Special thanks are due R.J. Celotta, J. Unguris and G.A. Prinz for helpful discussions and F. Meier for providing his manuscript [17] prior to publication. This work was supported in part by the Office of Naval Research.

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