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Static and dynamic magnetic behaviour of iron nanoclusters on magnetic substrates

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Abstract

The static and dynamic magnetic behaviour of Fe nanoclusters with controlled sizes in the range 140–270 atoms (1.5–1.8 nm) deposited *in situ* from a gas aggregation source on magnetic vitrovac amorphous ribbons has been studied using synchrotron radiation. The static magnetization of the cluster films in the exchange field of the substrates was measured as a function of coverage using magnetic linear dichroism in the angular distribution (MLDAD) of the Fe 3p core level photoemission. The switching dynamics were studied on the nanosecond timescale by time-resolved spin-polarized photoemission. For a given particle size, the magnetization of the Fe cluster film increases with coverage and saturates at a coverage of about 0.4 cluster monolayers. Modelling the growth of the magnetization gives an effective exchange field at the interface of ~ 20 T. Dense cluster films with several cluster layers have an MLDAD signal at saturation that is $\sim 5\%$ higher than a molecular beam epitaxy (MBE)-grown film, indicating an enhanced spin moment even when clusters are in contact. Coating an exposed sub-monolayer cluster layer with Co increases the Fe MLDAD signal by 35%, indicating a substantially increased magnetic moment within the Fe clusters. At low coverages, below the percolation threshold, the switching dynamics of the sample remains the same as in the clean substrate. At around the percolation threshold, however, a significant acceleration of the magnetic reversal is observed with a fast component due to a reversal propagating through the cluster film. We show that, on average, each cluster switches in about 10 ns.

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1. Introduction

Recently a great deal of attention has been focused on the study of magnetic nanoparticles which have been produced in the gas phase and contain a few to a few thousand atoms [1]. This is partly due to a fundamental interest in mesoscopic magnetism as gas-phase clusters afford the opportunity to study how magnetism develops in matter as it is built atom-by-atom from the monomer. In addition, there is a growing realization of the enormous potential of cluster-assembled films in the production of high-performance magnetic materials. It has been known for a decade that free clusters have novel magnetic properties such as a significant increase in the magnetic moment per atom relative to the bulk [2, 3], the appearance of magnetism in paramagnetic metals [4], and ferrimagnetism in anti-ferromagnetic materials [5]. It has since been discovered that substantially increased magnetic moments are retained in 3d transition metal clusters when they are deposited on a surface [6–9] or embedded in a matrix [10].

Increasingly attention has been focused on the effect of interactions in cluster deposits that can occur between the clusters [11], the clusters and substrate, and clusters and coating materials [9]. In this context the exchange interaction is of great importance, since the exchange length is significant compared to the cluster diameter and in interacting cluster films produced by depositing the clusters from the gas phase, it is the dominant mechanism [11, 12]. This produces a number of interesting and technically useful effects. For example, in very dense Fe and Co cluster films, the frustration resulting from the inter-particle exchange interaction and the randomly oriented intra-cluster anisotropy produces a correlated super-spin glass ground state that is magnetically soft [13]. Exchange-coupled nanostructures have also generated a great deal of interest in the context of ‘exchange-spring’ magnets produced by magnetically hard and soft phases interacting via exchange [14].

The study of the behaviour of magnetic nanoclusters deposited on magnetic substrates in ultra-high vacuum (UHV), which is the most elementary exchange-coupled nanoparticle system, is therefore important. The clusters have a direct metal-to-metal contact with the substrate at the atomic level and this evokes the exchange interaction between the cluster and the substrate. The dynamics in this type of system are also important because of the likely use of cluster-assembled materials in magnetic recording applications [15].

Here we report static and dynamic measurements of the magnetic behaviour of Fe nanoclusters with diameters in the range 1.47–1.80 nm (140–270 atoms) deposited *in situ*, using a gas aggregation source, onto amorphous vitrovac (an alloy containing Co, Si, Mn and O) substrates. We have studied the behaviour as a function of cluster size and cluster density and observed the effect of coating the exposed cluster deposits with Co *in situ*. The experimental methods allowed us to determine the magnetic behaviour specifically within the Fe clusters and independently of the substrate or coating materials. The magnetization dynamics of Fe clusters embedded in Ag matrices have been reported previously [16], but here we investigate the magnetization dynamics in clusters on a surface exposed in UHV.

2. Experimental methods

2.1. Preparation of cluster films

All cluster films were deposited *in situ* using a UHV gas aggregation source, described elsewhere [17], coupled directly to the UHV end-station of beam line SU7 on the super-ACO storage ring, LURE, Orsay, France. The cluster source produces a log-normal distribution of particle sizes with a typical standard deviation, σ , of 1.5–2 [12]. It incorporates a quadrupole mass filter operating up to masses 350 000 amu, corresponding to clusters containing 6300 Fe

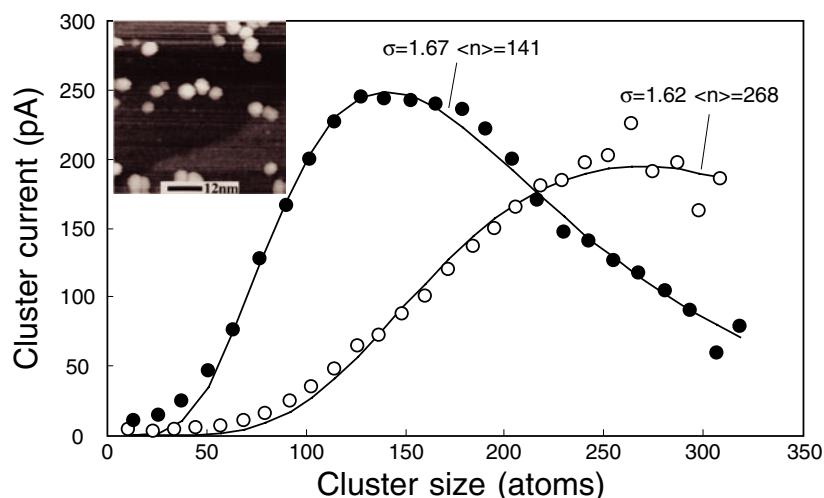


Figure 1. Mass spectra of clusters produced with bath gas pressures of 5 mbar (filled circles) and 10 mbar (open circles). The lines show best-fit log-normal curves and the corresponding $\langle n \rangle$ and σ values. The inset shows a previously obtained *in situ* STM image of clusters produced by the source operating under similar conditions.

atoms (5.2 nm diameter) with an ultimate mass resolution of 0.5%. During the experiment the very high flux of unfiltered neutral clusters was used in order to keep deposition times to a few minutes and minimize contamination of the exposed clusters. The most probable size within the distribution was controlled by varying the He pressure within the Fe vapour in the gas aggregation region and using the quadrupole filter to measure the corresponding mass spectrum so that a known size distribution was investigated. An example of mass spectra obtained at two different pressures is shown in figure 1, which also displays an *in situ* STM image obtained in a previous study of unfiltered Fe clusters deposited on Si(111) under similar preparation conditions. It has been verified that, in the case of Fe clusters, the mass distribution measured in the free cluster beam is unchanged after depositing on a substrate [18] or embedding in a matrix [11]. The mass spectra show the expected log-normal shape and the curves in figure 1 are optimized fits as a function of the mean size ($\langle n \rangle$) and the standard deviation (σ). The substrate was an amorphous vitrovac ribbon which could be kept magnetized by a small magnetic coil on the sample holder. This was cleaned by cycles of Ar sputtering and annealing prior to each cluster deposition.

2.2. Static magnetic measurements

The time-averaged magnetization in the clusters was determined by magnetic linear dichroism in the angular distribution (MLDAD), which measures the linear dichroism in the angle-resolved Fe 3p core level photoemission spectrum [19]. The technique is element specific and surface sensitive so that the behaviour of the Fe clusters could be determined independently of the substrate. During the measurements the vitrovac substrates were kept magnetized so that the clusters were under the influence of the exchange field at the interface and the fringing field above the substrate. The geometry of the experiment is shown in figure 2(a) and the dichroism was determined by reading two values of the photoemission intensity at each kinetic energy with the direction of the magnetization reversed in between. A typical pair of spectra obtained from approximately 0.1 monolayer of clusters with a diameter of 1.8 nm ($\langle n \rangle = 268$ atoms) is

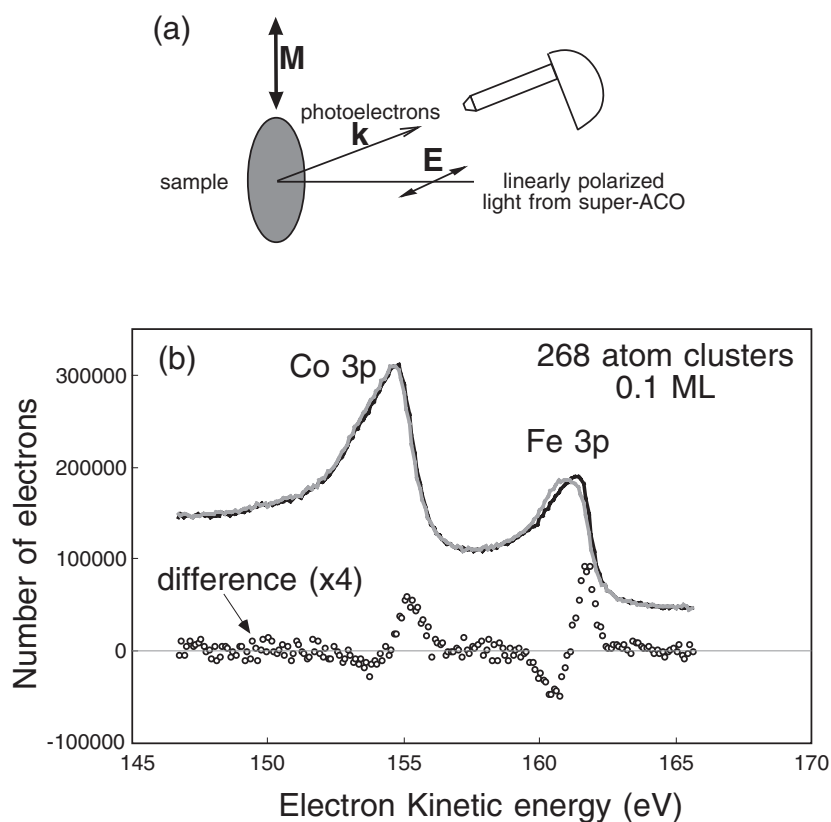


Figure 2. (a) Geometry of the MLDAD measurements. (b) Photoelectron spectra in the Fe and Co 3p region from 1.8 nm Fe clusters ($n = 268$ atoms) on vitrovac at a coverage of ~ 0.1 ML. The black and grey curves show the spectra for opposite magnetizations and the circles show the difference (dichroism).

shown in figure 2(b). Also shown is the difference highlighting the strong dichroism in the Fe 3p feature and the weaker dichroism in the substrate Co 3p spectrum. Note the same sign of the dichroism at the Fe and Co 3p features, showing the ferromagnetic coupling between the clusters and the substrate.

2.3. Dynamic magnetic measurements

The time structure of the synchrotron light from super-ACO was exploited to measure the magnetization dynamics by time-resolved electron polarimetry, as described in a previous report [20]. Briefly, ‘white’ light pulses with a duration of 500 ps and a frequency of 8.33 MHz (120 ns spacing) from the storage ring illuminate the sample and the spin polarization of each corresponding secondary electron pulse is measured using a Mott polarimeter. The external magnetic field is applied to the sample by means of a current pulse in a low inductance coil. A steady magnetization state (saturation) in the substrate is maintained by a constant bias current in the coil and the super-ACO clock signal is used to trigger a reverse current, with a rise time of about 70 ns, that can be varied between a few milliamperes and 40 A. The reverse current is maintained for a chosen integer of clock pulses, after which the current returns to its bias

value. The sequence is re-triggered a fixed integer of clock cycles later and the secondary electron polarization is monitored throughout the reversals at 120 ns intervals. A variable time delay with 1 ns steps between the clock pulse and the coil trigger enables the sample magnetization throughout the reversal to be determined every nanosecond. The experiment is surface sensitive and, as shown in previous studies [21], an Fe overlayer produces a much higher spin polarization than the vitrovac substrate and the polarization is dominated by an Fe film only 3 Å thick.

3. Results and discussion

3.1. Static magnetization

Figure 3 shows a series of photoemission spectra taken with a photon energy of 220 eV over the Fe and Co 3p core level regions for anti-parallel magnetizations of the vitrovac substrate (filled and open circles) as a function of coverage for clusters with a mean size of 141 atoms. Each cluster has a diameter greater than the scattering length of the photoelectrons at these kinetic energies so, ignoring cluster edge effects, the ratio of the Fe:Co 3p peak intensities, after correcting for the (small) cross-section difference, is the same as the ratio of the Fe cluster:exposed Co areas. The coverage in the figure and throughout the rest of the paper is expressed in cluster monolayers (ML), where a dense monolayer is assumed to be a close-packed hexagonal array. The higher coverages were obtained by measuring the time required to produce a thinner film and then scaling the coverage in proportion to the deposition time.

The dichroism (curves) plotted in figure 3 grows with cluster coverage and the inset shows the total area in the dichroism spectra normalized to the areas of the Fe 3p spectra summed over the two anti-parallel magnetizations (filled circles). The magnetic saturation of the cluster films is observed and there is a significant magnetization of the isolated clusters at very low coverage. This is the magnetization in the super-paramagnetic isolated clusters exposed to the magnetic field, B , at the vitrovac surface. The effect of the ferromagnetic substrate is demonstrated by comparing the data to previously reported MLDAD measurements [22] from Fe clusters produced by the same source and deposited *in situ* onto highly oriented pyrolytic graphite (HOPG) surfaces at 40 K. Anti-parallel saturating fields have been applied to these clusters prior to taking spectra, but at low coverages—despite the lower temperature—the super-paramagnetism in the supported clusters returns the magnetization to zero until the coverage is a complete monolayer and the film becomes ferromagnetic.

In an applied field, B , the relative magnetization in an assembly of non-interacting clusters is given by the Langevin function:

$$L(x) = \coth(x) - \frac{1}{x}, \quad (1)$$

where $x = \mu B/kT$ and μ is the magnetic moment of the cluster. From the relative magnetization of the 141 atom clusters at the lowest coverage, the field in which they are immersed can be estimated to be just over 3 T. A more reliable value can be obtained through a Monte Carlo simulation of the average island size as a function of coverage and by assuming that the larger islands remain superparamagnetic. The curve in the inset shows the best fit through the data optimized by adjusting B as the single parameter, which gives a field of 4 T. It is observed that the film saturates magnetically at coverages lower than the 2D percolation threshold (infinite island), which is at a coverage of between 0.6 and 0.7 ML, depending on the morphology of the clusters [23].

This is the average field inside the clusters and is clearly much larger than the dipolar fringing field above the vitrovac, whose internal field at saturation is about 1 T. Its source is the

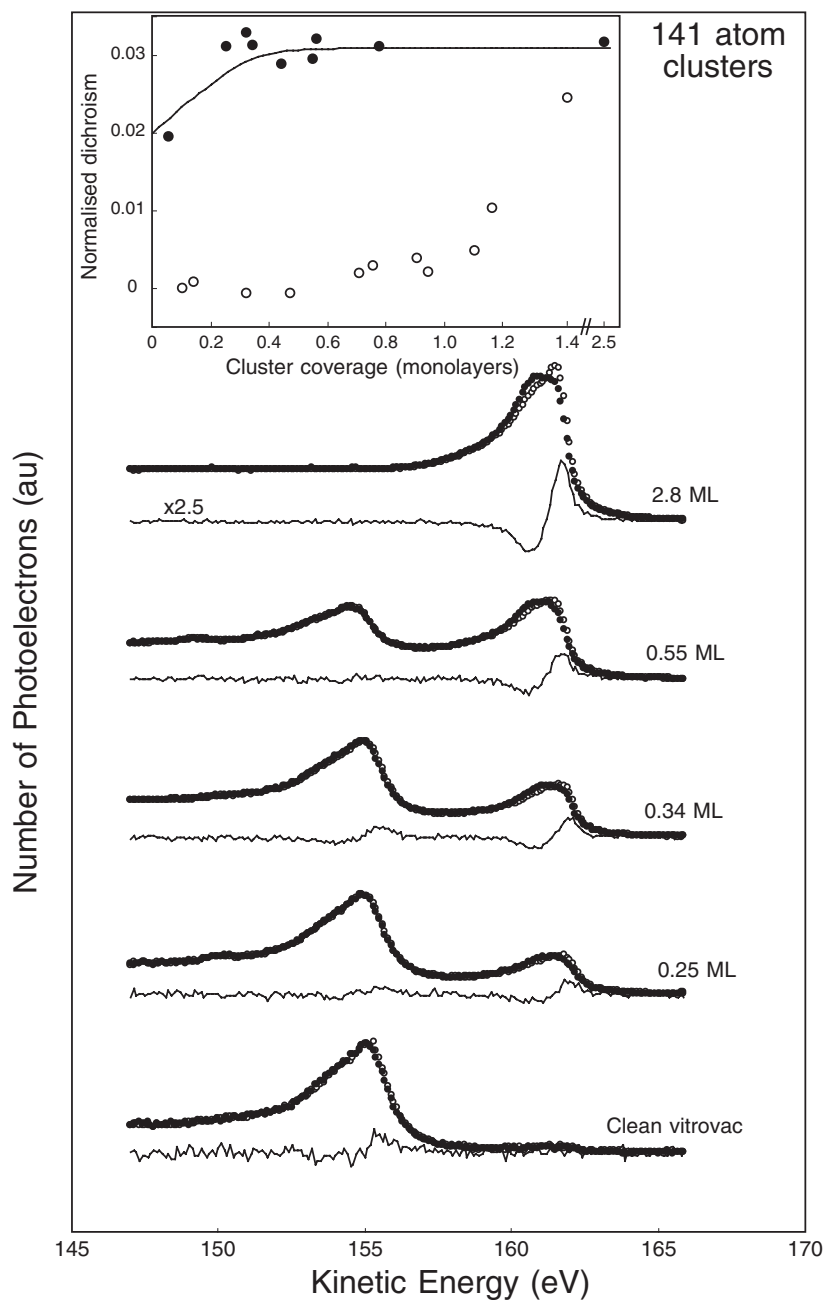


Figure 3. Photoemission spectra over the Fe and Co 3p regions as a function of coverage of Fe clusters with $\langle n \rangle = 141$ atoms. The spectra taken with anti-parallel magnetization of the vitrovac substrate are denoted by open and filled circles. The curves show the difference (dichroism). The inset shows the strength of dichroism, normalized to the Fe 3p peak intensity, as a function of coverage for growth on magnetic vitrovac substrates (filled circles) and HOPG (open circles—reproduced from [22]). The curve is a Monte Carlo simulation of the magnetization in growing superparamagnetic cluster islands in the interface exchange field as a function of coverage (see text).

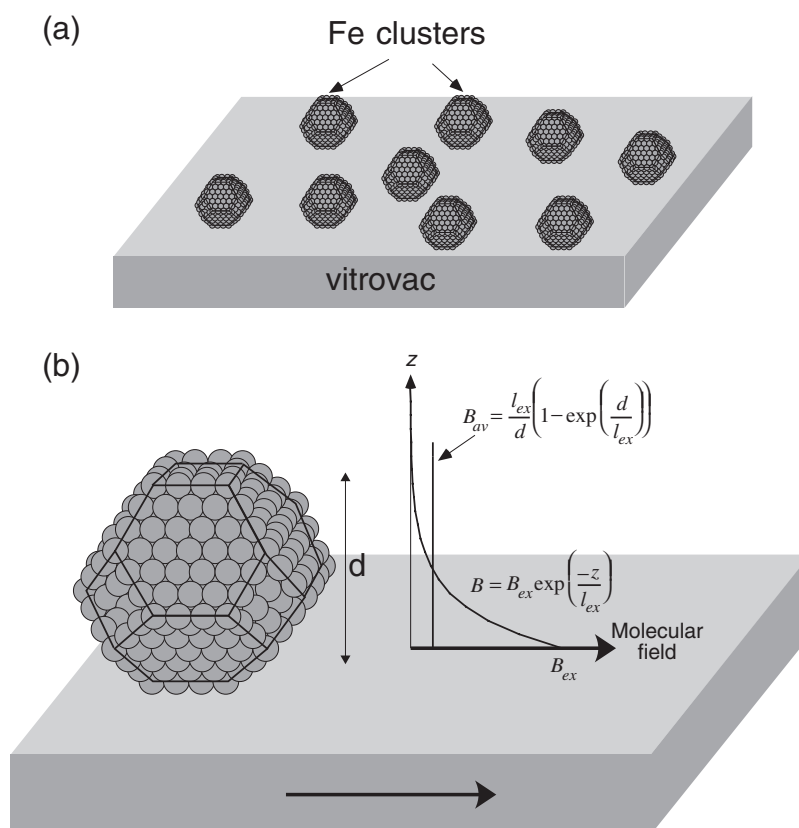


Figure 4. Schematic diagram of the molecular field within Fe clusters adsorbed on vitrovac due to the exchange interaction at the contact point between the clusters and the substrate.

molecular field due to the exchange interaction between the particles in direct atomic contact with the vitrovac surface. The molecular field can be assumed to decay exponentially with height above the surface with a characteristic exchange length, l_{ex} , of about 3 \AA , as shown schematically in figure 4. The average field within a cluster with a diameter d is given by:

$$B_{av} = B_{ex} \frac{l_{ex}}{d} \left(1 - \exp\left(-\frac{d}{l_{ex}}\right) \right), \quad (2)$$

where B_{ex} is the effective field due to exchange at the contact point of the clusters on the vitrovac. Thus to obtain a mean field of 4 T requires an interface exchange field of $\sim 20 \text{ T}$. This is about 50 times smaller than the molecular field in bulk Fe and is of the same order of magnitude as the exchange coupling between neighbouring Fe clusters [11].

Figure 5(a) shows the MLDAD spectra from a thick (50 \AA or 2.8 ML) layer of Fe clusters with a mean size of $\langle n \rangle = 141$ atoms on vitrovac. This is compared (after normalizing the Fe 3p line intensities) to the spectrum from a similar-thickness conventional Fe film grown on vitrovac using an MBE source. Both films are saturated in either direction by the exchange field at the interface and it is observed that there is a measurable increase in the MLDAD signal from the cluster film, indicating an enhanced spin moment per atom. It is well known that free clusters in this size range have enhanced magnetic moments [2] and previous x-ray magnetic circular dichroism (XMCD) measurements on exposed and isolated clusters

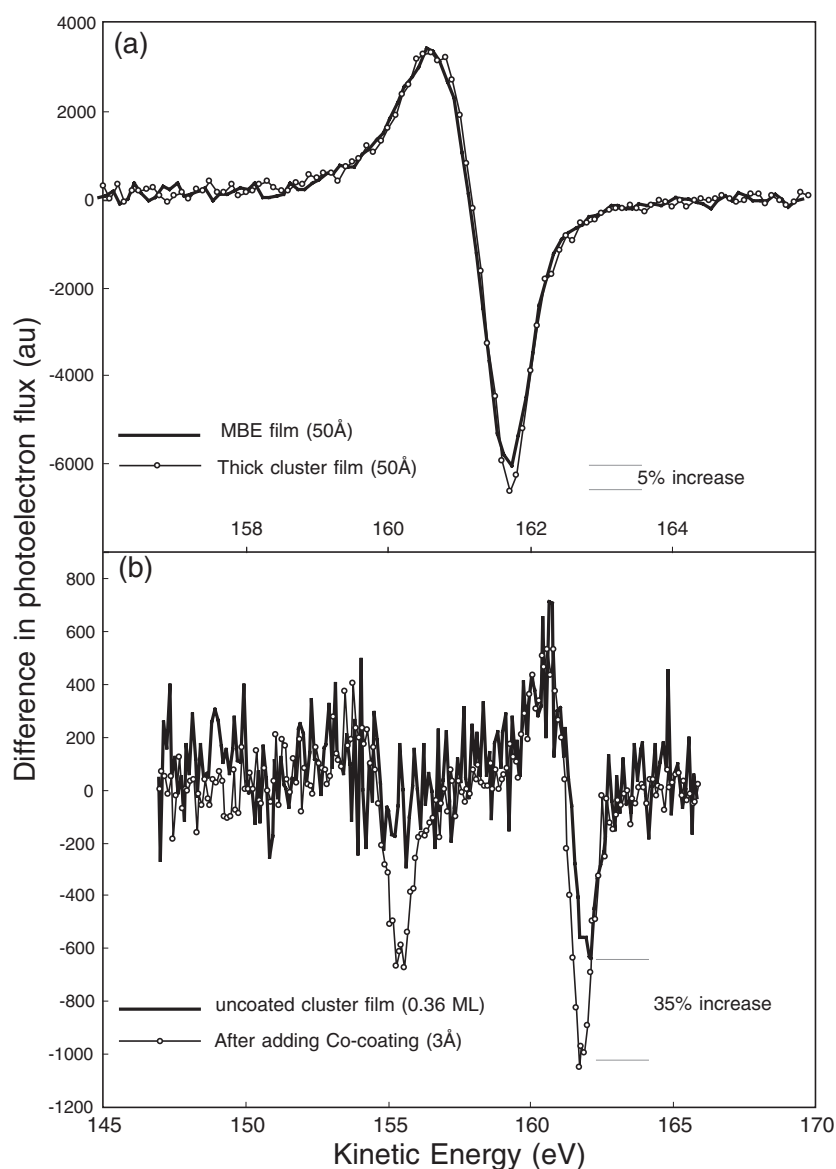


Figure 5. (a) A comparison of the MLDAD signal from a thick layer (50 Å) of Fe clusters with $\langle n \rangle \approx 260$ atoms on vitrovac (thin curve with open circles) with a similar-thickness MBE-grown Fe film on vitrovac (thick curve). Both films are magnetically saturated in both directions by the exchange field at the interface and it is observed that the MLDAD signal is 5% higher in the cluster film. (b) A comparison of a sub-monolayer coverage of Fe clusters with $\langle n \rangle \approx 260$ atoms on vitrovac (thick curve) and the same film after depositing a 3 Å thick layer of Co *in situ* (thin curve with open circles). The Co coating layer increases the MLDAD signal by 35%.

deposited on HOPG showed enhanced orbital and spin magnetic moments [9]. The question of what happens to these enhanced moments at increased densities when the clusters are in contact has never been fully answered. An earlier study by XMCD of the orbital and spin moments in exposed Fe clusters with a mean size of 600 atoms as a function of coverage

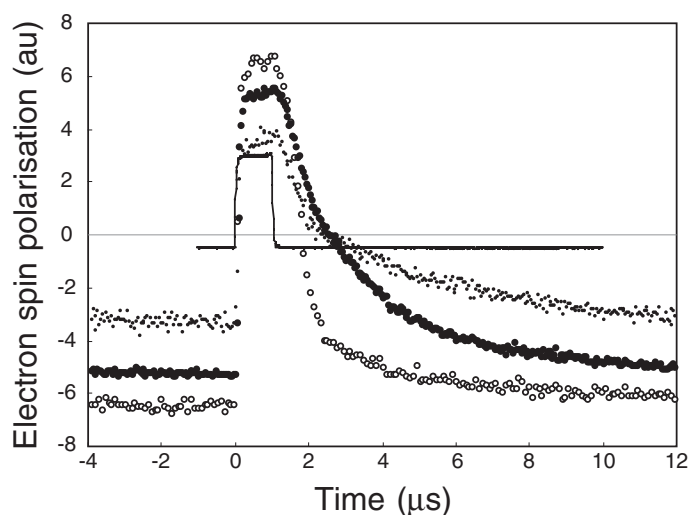


Figure 6. Spin polarization (proportional to surface magnetization) versus time during a reverse magnetization pulse (curve) for the clean substrate (dots), a coverage of 0.32 ML of clusters with $\langle n \rangle = 140$ (filled circles) and a coverage of 0.57 ML of clusters with $\langle n \rangle = 141$ (open circles). The substrate is maintained at saturation by a small negative bias current and the reverse magnetization pulse is about $6 \times$ the bias, thus the rise response in all samples is faster than the decay. Note the increase in spin polarization amplitude produced by the clusters.

on HOPG [6] showed that the orbital moment, which has a value 2.5 times the bulk value at very low coverages, rapidly converges to the bulk value as the clusters come into contact. However, the behaviour of the spin moment could not be untangled from the variation in the dipole contribution as a function of the cluster density. It appears from the present study that enhanced spin moments are maintained in the clusters even in thick multi-layered films. In the free clusters the increased spin moments are attributed to a narrowing of the transition metal 3d bands, producing an increased spin polarization per atom.

Figure 5(b) shows the MLDAD spectrum from a sub-monolayer coverage of Fe clusters with a mean size of $\langle n \rangle = 260$ atoms on vitrovac and how this is modified by coating with a Co layer of thickness 3 \AA *in situ*. A 35% increase in the amplitude of the MLDAD signal is observed, indicating a substantial increase in the Fe spin moment. A previous XMCD study of mass-selected Fe clusters on HOPG in the size range 180–690 atoms showed that coating with Co *in situ* increases the spin moment in the Fe clusters by 10% without significantly changing the orbital moment. The underlying mechanism and a detailed theory of MLDAD have been described by van der Laan [24] who showed that, in the limit of large spin field relative to the spin–orbit interaction, the magnitude of MLDAD is proportional to the spin–orbit interaction. Since the orbital moment is not significantly affected by the Co coating, the relative change in the MLDAD signal is proportional to the relative change in the Fe moment, indicating in this case an Fe magnetic moment in the Co-coated clusters of $\sim 3 \mu_B/\text{atom}$. This is similar to the atomic values observed in Fe–Co alloys [25].

3.2. Magnetization dynamics

Figure 6 shows the measured spin polarization, proportional to the surface magnetization as a function of time during an entire reverse pulse ON/OFF cycle for the clean substrate and for

two different coverages by Fe clusters with a mean size $\langle n \rangle = 141$. Prior to the application of the pulse, the sample is maintained in a magnetized state by a bias current. The strength of the applied reverse current pulse is about six times the bias current, so the dynamic response at the leading edge is faster than on the trailing edge. The most apparent changes in the cluster-covered sample are the increase in the spin polarization and a modification of the dynamics on the fall side. There appear to be two components to the dynamic response of the thickest cluster layer, i.e. a greatly accelerated reversal followed by a slower decay and it is tempting to associate the rapid change with the clusters and the slower response with the substrate. This separation is confirmed by the analysis described below.

The magnetization reversal process in continuous thin films is known to occur through the nucleation of reverse domains and subsequent propagation of the domain walls [26]. The switching rate is determined by both the rate of formation of the reverse magnetization nuclei and the speed of the propagation of the domains. Which one is dominant is sensitive to the microstructure of the film. A model to describe the shape of the switching pulse in terms of these parameters was reported by Fatuzzo [27] and, although originally developed for ferroelectrics, it is applicable to any type of magnetically or electrically ordered system [28]. The model assumes that reverse domains nucleate at a constant rate R with an initial radius r_c and that the walls of the reverse domains propagate outwards with a velocity v . The shape of the reverse magnetization pulse is then given by:

$$M(t) = M_s(2e^{g(\tau, \kappa)} - 1), \quad (3)$$

where $\tau = Rt$, $\kappa = v/Rr_c$ and

$$g(\tau, \kappa) = -2\kappa^2 \left[1 - \left(\tau + \frac{1}{\kappa} \right) + \frac{\left(\tau + \frac{1}{\kappa} \right)^2}{2} - e^{-\tau} \left(1 - \frac{1}{\kappa} \right) - \frac{1 - \tau}{2\kappa^2} \right]. \quad (4)$$

The shape of the reversal depends on the ratio, κ , of the wall propagation speed to reverse domain nucleation rate.

Figure 7(a) shows the magnetization reversals of the vitrovac substrate, normalized to ± 1 on the rise (reversed for comparison) and fall sides of the magnetization pulse. The fits using the above model are also shown and the corresponding values of R and κ are indicated. The parameters were obtained by fixing R and optimizing the fit as a function of κ then repeating for a different value of R to find the global minimum. For the clean substrate it is observed that the switching regime changes from one dominated by the reverse magnetization domain wall propagation speed on the rise side to one dominated by the reverse magnetization nucleus formation rate on the fall side.

Figure 7(b) shows the corresponding data and fit for a deposition of 0.32 ML of clusters with a mean size $\langle n \rangle = 141$ and, within the uncertainty of the fitting procedure, parameters on the rise and fall side the same as for the clean substrate. At a coverage of 0.32 ML the cluster film consists of small separate islands and it is evident that the switching mechanism is unchanged, with the overlayer acting as a passive amplifier of the electron spin polarization.

The character of the reversal on the fall side changes markedly when the coverage by the clusters is increased to 0.57 ML, as shown in figure 7(c). On the logarithmic timescale the superposition of a fast and a slow process, alluded to above, is even clearer and it becomes impossible to obtain a reasonable fit with a single Fatuzzo curve. The fit shown is to the fast process only and the residual data emerging from the fit in the slow process can be fitted using the same parameters as for the clean substrate. The implication is clear—that is, at the higher coverage (which corresponds to the percolation threshold of the cluster layer) a direct reversal path becomes available through the cluster film itself with the reversal transmitted by the exchange interaction between the clusters. The high value of κ shows that the reversal

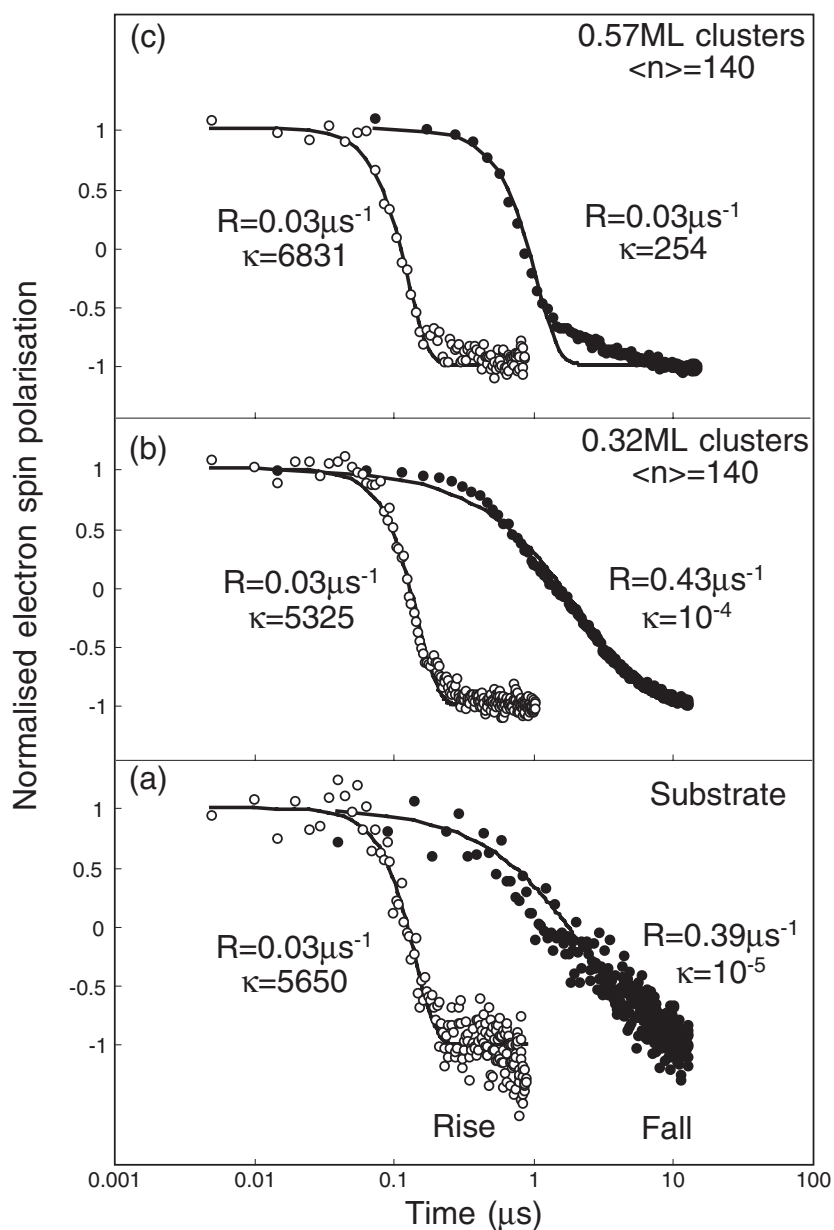


Figure 7. Magnetization reversals, normalized to ± 1 on the rise (open circles) and fall (closed circles) sides of the magnetization pulse. The solid curves are fits using the Fatuzzo model [27] with the parameters indicated by each curve. The data on the rise side has been reversed for comparison.

through the cluster film is dominated by the velocity of propagation rather than the rate of reverse magnetization nuclei, in contrast to the mechanism for reversal on the fall side for the clean substrate. On the rise side the mechanism remains the same, but the velocity of the reversal increases further.

With this reversal mechanism for the thickest cluster layer it is quite reasonable to assume that the initial radius of the reverse domains (r_c) is the cluster radius, thus we can extract the

velocity, v , of the reverse magnetization propagation. This is related directly to the average switching time, τ_s , of individual clusters by $\tau_s = d_{cluster}/v$, where $d_{cluster}$ is the cluster diameter (1.5 nm). Thus we find that $v = 0.154 \text{ m s}^{-1}$ on the rise side, corresponding to a switching time of an individual nanocluster, τ_s , of 9.7 ns, and $v = 0.0057 \text{ m s}^{-1}$ on the fall side, corresponding to $\tau_s = 263 \text{ ns}$.

4. Conclusions

An investigation, using MLDAD, of exposed Fe nanoclusters deposited *in situ* onto magnetic amorphous vitrovac substrates has revealed that the clusters are exchange coupled to the substrate with an interface molecular field of about 20 T. Thick cluster layers have a spin moment that is enhanced by about 5% relative to the bulk. Coating thin cluster layers with Co *in situ* increases the MLDAD signal by 35%, indicating an Fe spin moment in the coated clusters of about $3 \mu_B/\text{atom}$. At low cluster coverages the magnetic reversal process in the sample is similar to that observed in the clean substrate and the cluster film acts as a passive amplifier of the electron spin polarization. At the percolation threshold a new, faster reversal process is observed that propagates through the cluster film. In this process the average switching time for a cluster is about 10 ns.

Acknowledgments

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