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Magnetic irreversibility in Fe/Cu multilayers

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Abstract. The magnetic behaviour of Fe/Cu multilayers produced by electron-beam evaporation has been investigated in the range 5 to 400 K and in magnetic fields up to 1 kOe using SQUID magnetometry. The multilayers exhibit an irreversible behaviour below a characteristic temperature T_{irr}. Low-field DC magnetic moment measurements on samples cooled in zero field revealed the usual signature of a spin-glass state. Time relaxation of the remanence and frequency-shift of the peak of the imaginary part of the AC magnetic moment were also observed. The irreversibility line was fitted to a de Almeida–Thouless line with a crossover critical exponent $\phi = 3.6$. We have interpreted these results as being due to the presence of some interdiffusion between Fe and Cu at the interfaces. The surprisingly high characteristic irreversibility temperatures indicate that diffused Fe atoms aggregate in clusters at the interfaces.

1. Introduction

Research in metallic magnetic multilayers is one of the most active and exciting areas in solid state physics, since such multilayers can exhibit novel and interesting physical effects with technologically important applications like magnetic recording media, devices and sensors [1]. The structural and growth-mode correlations with magnetic properties become an ongoing challenge to materials research developments on multilayered magnetic-film structures.

In particular, Fe/Cu multilayers present antiferromagnetic exchange coupling which is accompanied by a moderate magnetoresistive effect for chemical modulation periods in the nanometre scale length [1, 2]. From a structural point of view, Fe/Cu multilayers are an interesting system. It is well known that face centred cubic (fcc) γ-Fe and body centred cubic (bcc) Cu metastable phases can be obtained in epitaxial or highly textured films [3–16]. Specifically, metastable fcc γ-Fe is a notable example of the influence of growth conditions on the properties of the resulting film. Earlier first-principles magnetic structure calculations confirmed that a rich variety of magnetic configurations can be stabilized in fcc Fe with a lattice constant range near that of fcc Cu [17–25]. It has also been shown that the magnetic anisotropy energies are rather different for the ferromagnetic and various antiferromagnetic configurations in fcc Fe [17, 18]. As a consequence, different growth conditions used by several research groups might turn out to be the source of ambiguities and experimental disagreements. Despite great interest in the magnetic properties of the Fe–Cu system, its low-field magnetic moment behaviour has not been thoroughly explored in the literature.

In separate studies [26–28], we have shown that epitaxial Fe/Cu superlattices can be grown directly on Si(111) and CaF_2(111) surfaces. Although Fe/Cu superlattices with different structural quality could be obtained (Cu and Fe buffer layers were also used), we found no meaningful way to explain the very low magnetoresistance of those samples.

In the present paper we report on structural and magnetic measurements on Fe/Cu multilayers, including a systematic characterization of the irreversible behaviour of the zero-field-cooled (ZFC) magnetization, together with field-cooled (FC) runs. Experimental results are analysed and discussed assuming that a magnetic metastable phase occurs at the interfaces due to the existence of some interdiffusion between Fe and Cu.

2. Sample preparation and techniques

Fe/Cu multilayers were grown on HF-etched Si(111) wafers, to improve the multilayer crystalline structure [26–28]. Some Fe/Cu multilayers were grown directly on Si(111) wafers, while for some others we have used Fe as a buffer layer. Both multilayers and buffer layers were grown using a Balzers UMS 500P electron-beam evaporator with a base pressure of 2×10^{-8} mbar. The Si(111) surface was HF etched to promote hydrogen passivation immediately before loading in the vacuum chamber. Multilayers were grown by successive deposition of Fe 15 Å and Cu 15 Å layers, repeated 10 times. Deposition temperature was 30 °C at a rate of 0.2 Å s^{-1} for both metals.

Multilayer structures were studied by x-ray diffraction with Cu Kα radiation, using the Bragg–Brentano θ–2θ scan, and transmission electron microscopy (TEM) in the selected area diffraction mode by using a JEOL 1200EX-II electron microscope operating at 120 keV.

Magnetic moment measurements $\mu(T)$, performed in the temperature range 5 to 400 K and fields up to 1 kOe, were taken with a SQUID magnetometer, model MPMS-5S, supplied by Quantum Design. Each measurement resulted from two sample scans over a length of 6 cm, with 40 point readings per scan. The standard deviation was generally very small, roughly of the same size as plotted symbols. Low-field magnetic moment measurements as a function of the temperature for different values of the applied field were carried out using a routine combining a zero-field-cooled (ZFC) warming run followed by a field-cooled cooling (FC) experiment. To start the procedure, the sample was first heated up to 400 K and then cooled down to 5 K in the absence of magnetic field. Magnetization loops were also measured for many temperatures in between 5 and 400 K. Remanent demagnetization cycles were done at 400 K to prevent spurious irreversible effects associated with magnetic history of the sample in the ZFC measurements. Real and imaginary parts of the low-field AC moment ($\mu(T)_{AC} = \mu'(T) + i\mu''(T)$) of the samples were also measured using the SQUID magnetometer with an excitation field $h_{AC} = 3.0$ Oe. The temperature dependence of $\mu(T)_{AC}$ at different frequencies was measured while cooling down the samples, although no specific reason exists for this particular choice, as $\mu(T)_{AC}$ proved to be reversible. All experiments were carried out in such a way that the applied fields (AC and DC) and the direction of magnetic moment detection were parallel to each other and to the plane of the film.

3. Structural properties

Typical high-angle x-ray diffraction patterns are shown in figure 1(a). A broad peak appears at $2\theta \approx 43.4^\circ$ originating from reflections of bcc Fe(110) and fcc Cu(111) planes. Due to this overlapping of the diffraction peaks, we were not able to evaluate the coherent length perpendicular to the films from the x-ray diffraction peak. High-angle satellite peaks associated with the periodic modulation are not seen probably due to the reduced number of bilayers. Figure 1(b) shows the low-angle x-ray diffraction of an Fe/Cu multilayer. The periodicity of

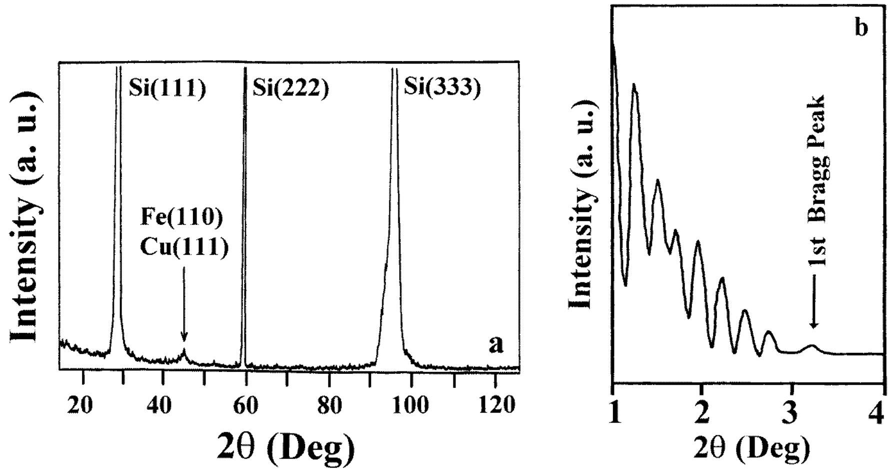

Figure 1. (a) Typical high-angle x-ray diffraction pattern of Fe/Cu multilayers directly grown on Si(111). (b) Low-angle x-ray diffraction of the Fe/Cu multilayer. Note the sharp Kiessig fringes and also a single Bragg peak which leads to a calculated modulation length of 28.5 Å.

the Kiessig fringe pattern is a good indication of flatness of the upper and lower multilayer interfaces on the substrate. The angular position of the first Bragg peak corresponds to a chemical modulation period of 28.5 Å which, as expected, is very close to the nominal modulation length.

Figure 2 shows the electron diffraction pattern of the Fe/Cu multilayer in selected area diffraction mode. The electron diffraction pattern from the multilayer confirmed the bcc-Fe and fcc-Cu structures with low texture. The presence of a few monolayers of bcc-Cu or fcc-Fe at the interfaces cannot be discarded due to their isomorphism with the bcc-Fe and fcc-Cu structures.

4. Magnetic properties—DC measurements

Figure 3 shows typical hysteresis loops obtained for an Fe/Cu multilayer at $T = 5$ K and 300 K. Apart from the small coercivity of the sample, which clearly appears in the figure, we observe ferromagnetic cycles with relatively low saturation fields. Hysteretic loops with low saturation fields were observed at all temperatures, confirming that the samples are ferromagnetic and providing evidence for the absence of very small Fe clusters and isolated Fe atoms within the Cu layers, thus ruling out superparamagnetism.

Figure 4 shows the DC magnetic moment as a function of the temperature for six different magnetic fields: (a) $H = 10$ Oe; (b) $H = 20$ Oe; (c) $H = 50$ Oe; (d) $H = 100$ Oe; (e) $H = 200$ Oe and (f) $H = 1000$ Oe. For each field, two curves are shown: the lower is for the multilayer film initially cooled to 5 K in zero magnetic field (ZFC) while the upper is for the film cooled in the measuring field (FC). Clearly, the multilayer presents an irreversibility commonly observed in spin-glass-like systems. In addition to a ferromagnetic component in the FC curves, there is another contribution whose behaviour below a given irreversibility temperature, T_{irr}, can be attributed either to a spin-glass or to mictomagnetic clusters (i.e. when magnetic behaviour is dominated by the presence of large magnetic clusters) which would freeze below T_{irr}. This magnetic behaviour could be explained by taking into account certain roughness effects at the Fe/Cu interfaces. In particular, one might admit some interdiffusion

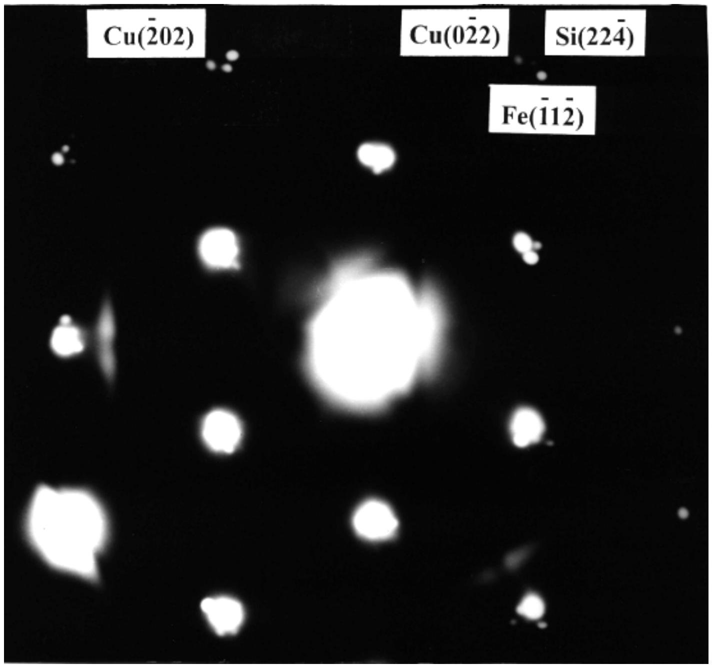

Figure 2. Electron diffraction patterns of the highly textured Fe 15 Å/Cu 15 Å multilayer. The hexagonal pattern of the diffraction spots of Si is visible. The Fe and Cu diffraction spots are identified with the aid of the x-ray diffraction but their signalization relative to the Si spots is done arbitrarily.

between Fe and Cu atoms at the interfaces since, according to Brodde *et al* [29], for room temperature deposition, Fe initially condenses on the Cu(111) surface mostly in oblong islands with typical lateral size of 10 Å × 30 Å and heights of about 2 Å.

Figure 5 shows a probable scheme for the interdiffusion between Fe and Cu at the interfaces assuming terrace-like shapes. The interfacial profile cannot be estimated from our measurements.

According to figures 4(a) to (f), T_{irr} associated with the bifurcation in the ZFC/FC curves is strongly dependent on the applied magnetic field. We can also observe a glass temperature T_G associated with the position of the maximum in the ZFC curve for some magnetic field values.

If diffused Fe atoms were isolated in a Cu layer, they could yield a spin-glass phase. It is known that up to 20% of Fe impurities in Cu yield spin-glass systems with freezing temperatures up to 50 K [4, 30]. However, figure 4 shows T_G values as high as 270 K, strongly suggesting that diffused Fe atoms aggregate in rather large clusters. On the other hand, the number of Fe atoms contributing to the spin-glass phase is limited to about 25% of Fe in Cu [4, 30]. Above this value, the alloy becomes ferromagnetic below 100 K. Thus, we can conclude that some diffusion of Fe atoms in Cu layers can occur, promoting the appearance of a mictomagnetic phase together with a ferromagnetic one. This ferromagnetic phase might be due to Fe clusters either at the central portion of the layers or at the interfaces.

A similar scenario is reported in different systems e.g. $La_{1.85}Sr_{0.15}CuO_4$ [31, 32], CdMnTe/CdTe [33] and Cu/Co [34]. According to those reports, the coexistence of paramagnetic and spin-glass behaviour in the system might be attributed to the existence

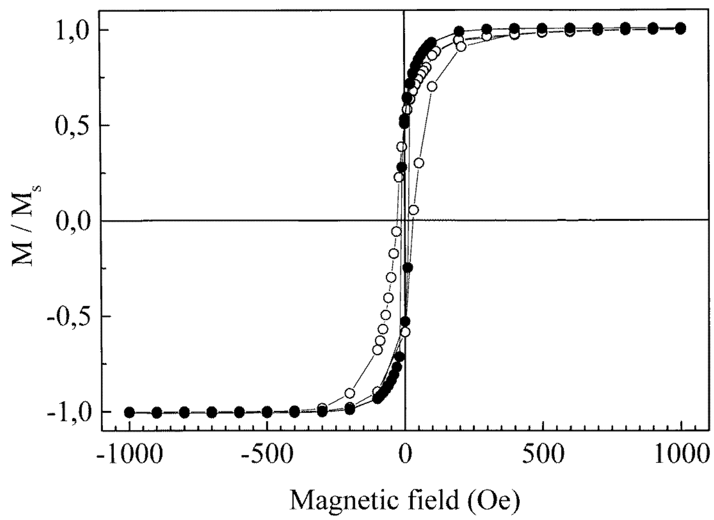

Figure 3. Typical hysteresis loops for a Fe/Cu multilayer measured at $T = 5$ K (○) and $T = 300$ K (●).

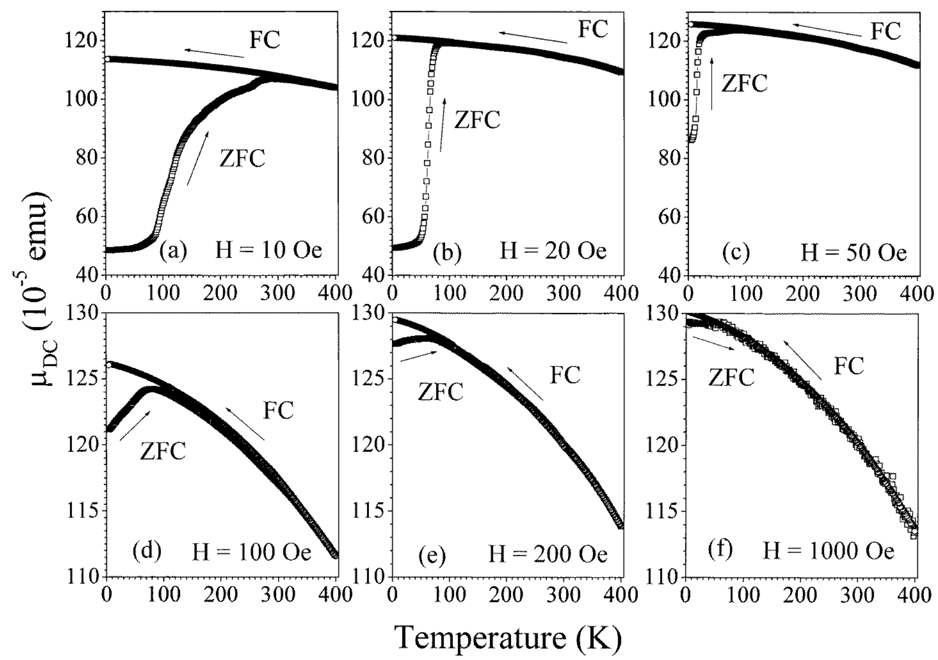

Figure 4. Temperature dependence of the DC magnetic moment for six different magnetic fields. Two curves are shown for each measuring field. The lower curve (□) is for the Fe/Cu multilayer initially cooled to 5 K in zero magnetic field (ZFC), while the upper curve (○) is for the film cooled in the measuring field (FC).

of magnetic atoms occupying nonequivalent sites at the surface or bulk of a grain and inner interfaces of the multilayered structures.

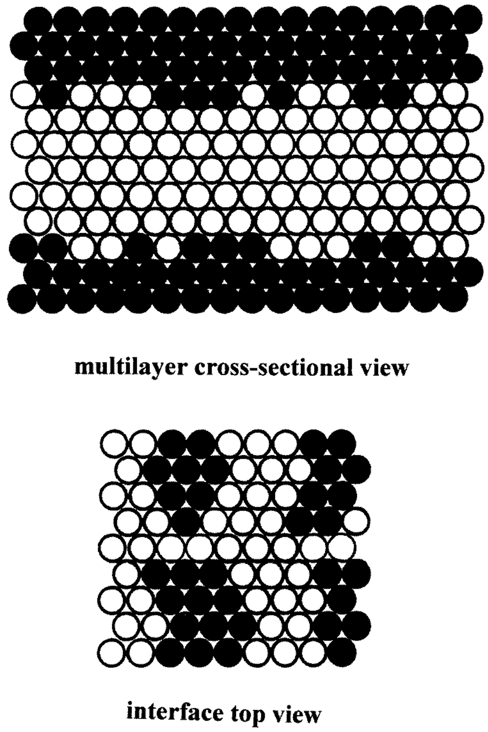

Figure 5. Schematic representation of the structure of the multilayers which may be used to illustrate some interdiffusion between (●) Fe and (○) Cu at interfaces.

It is important to emphasize that the alternative picture, based on a randomly distributed ferromagnetic/antiferromagnetic coupling across the Cu spacer layers along the multilayer structure, could not explain the observed experimental results. In fact, if a random interlayer ferromagnetic coupling is enhanced (for example, through pinhole effects) and largely overcomes a random interlayer antiferromagnetic coupling, the clusters would become large and high values of T_{irr} should be expected. However, the global response of the sample should become ferromagnetic, since the irreversibility temperature is determined by the maximum size of magnetic clusters [34–36]. On the other hand, if a random antiferromagnetic interlayer coupling is moderate or predominant and large clusters are formed to give high T_{irr}, the sample would then exhibit an antiferromagnetic global response. In both cases, the experimental predictions are clearly in disagreement with observed results. Besides, a moderate magnetoresistance (MR) would be usually observed for this Cu layer thickness [2]. A small negative magnetoresistance is, however, observed over the wide range of thicknesses investigated [27, 28]. A possible explanation for the deterioration in the MR ratio could be the existence of magnetically clustered regions in these samples. According to the two-current model, at finite temperature sufficiently lower than the Curie temperature, the magnitude of the MR ratio is strongly dependent on the momentum transfer between the two spin-channels, the so-called spin-mixing effect [37]. At room temperature the spin-flip is predominantly mediated by electron–magnon scattering. Thus, the existence of magnetically clustered regions at interfaces would reduce the spin-flip diffusion length, which tends to diminish the magnetoresistance [37].

The ferromagnetic phase contribution can be evaluated by fitting Bloch's $T^{3/2}$ law ($\Delta M/M = \beta T^{3/2}$) to the FC curves at low temperatures. According to the fitting of the FC curve measured at 1000 Oe, we can estimate a β value for the ferromagnetic contribution of about 9.63×10^{-6} $K^{-3/2}$. This is around three times the value obtained for bulk Fe ($\beta = 3.4 \times 10^{-6}$ $K^{-3/2}$) [38]. As the values of the exchange coupling constant for Fe layers thicker than 5 Å become practically identical to bulk values [39], we can estimate that the magnetic moment per atom is reduced by about 50%. This means that the magnetic moment of some Fe atoms is reduced by the effect of its Cu neighbourhood. In fact, Durand *et al* [4] reported a strong reduction of the magnetic moment in Fe/Cu multilayers. Although Fe layers growth in a fcc phase, their observed values for the magnetic moment of Fe atoms are still lower than those expected for fcc-Fe bulk. However, the FC curves show that our sample is ferromagnetic up to 400 K, which means that large ferromagnetic regions of our sample contribute to the spontaneous magnetization.

In order to analyse the behaviour of the characteristic temperature $T_{irr}(H)$ associated with the bifurcation point of the ZFC and FC magnetization curves for each value of H, we introduce a reduced temperature, $t = T_{irr}/T_c$. The ferromagnetic–paramagnetic transition temperature T_C was chosen to be the critical temperature of bulk Fe, in view of the above mentioned fact that exchange coupling converges rapidly to bulk values even for samples having only a few monolayers of Fe [39].

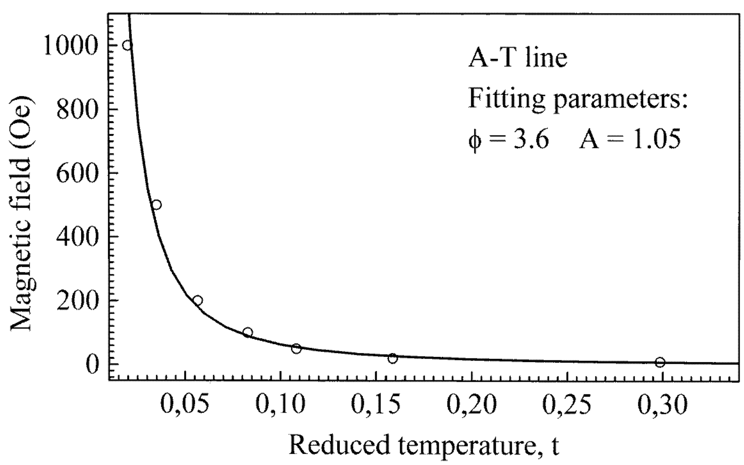

Figure 6. The irreversibility line plotted in its usual reverse format. Notice that a reduced temperature $t = T_{irr}/T_c$ appears on the horizontal axis. The solid line is a best fit to a de Almeida–Thouless (A–T) line; $\phi = 3.6$ is the crossover critical exponent and $A = 1.05$ Oe $[K]^{-1.8}$ is a fitting parameter.

Figure 6 shows the irreversibility line in its usual reverse format, i.e. H versus t. The solid line is a best fitting plot of the experimental data to the function $H = At^{\phi/2}$, where A is a fitting parameter, and ϕ is the crossover critical exponent. In the present case, we found $\phi = 3.6$, to be compared to the theoretical prediction [40] of a de Almeida–Thouless line with $\phi = 3.0$. This is a fairly reasonable agreement which, associated with the strong irreversibility actually exhibited by the system, encourages the adoption of Mydosh's picture of a multi-valley space-configuration to describe the free energy in Fe/Cu interfaces [40].

Another general feature observed in this study is the time relaxation of the remanent magnetization. The remanent state was obtained by applying a magnetic field of 1000 Oe for

30 min immediately after cooling the sample in a zero field. Remanent magnetization was measured as a function of the time elapsed after switching off the applied field. This procedure is called ZFC relaxation. Figure 7 shows a $\log t$-plot of the remanent magnetization at 2 K. The first 100 seconds are omitted from the figure, since the exponential decay of the magnetization varies excessively fast to be picked up by the measuring technique, whose acquisition time was set to about 10 seconds per experimental point. The origin of the $\log t$-behaviour is related to the energy barrier distribution which determines the fraction of magnetic clusters that become unblocked due to thermal fluctuations [36].

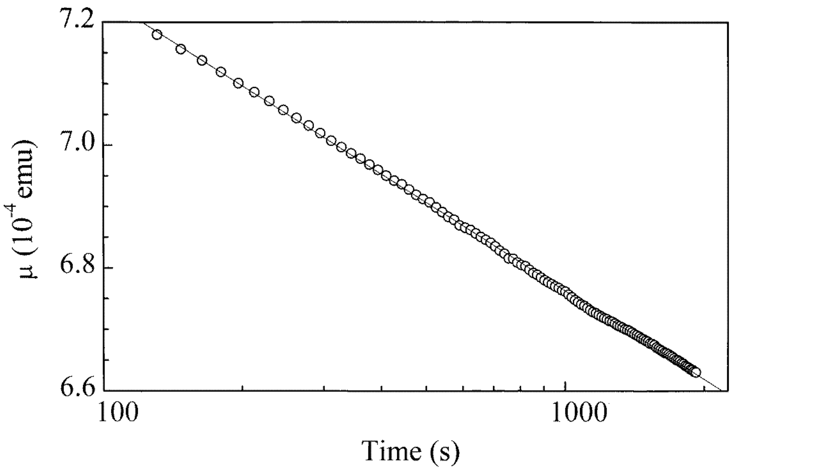

Figure 7. Typical $\log t$-plot of the remanent magnetization of the Fe/Cu multilayer. Experimental data (○) and $\log t$-fit (solid line) are given.

5. Magnetic properties—AC measurements

Figures 8(a) and (b) show, respectively, the real $\mu'(T)$ and imaginary $\mu''(T)$ parts of the low-field AC magnetic moment of the Fe/Cu multilayer. The temperature dependence of $\mu'(T)$ is not significantly changed by the frequency. However, a small hysteretic effect dependent on the frequency was observed. Such a hysteretic effect is another signature of metastable magnetic systems. Finally, figure 8(b) shows a typical frequency-shifted peak observed in mictomagnetic systems. The temperature where the $\mu''(T)$ curve shows a maximum, as well as its shape, are basically determined by the competition between the increase in the fraction of clusters that become unblocked with increasing temperature and the magnetic moment decrease due to thermal fluctuations. If the coupling mechanisms between particles is strong enough to increase the typical cluster size in the system, particle interactions will also influence the shape of the $\mu''(T)$ curve. By increasing the frequency used to measure the AC magnetic moment a shift towards high temperatures is predicted [40] for the maximum of the $\mu''(T)$ curve. As expected, this shift is observed in figure 8(b).

A comparison with figure 4 shows that both magnitude and temperature dependence of low field DC magnetic moment in FC measurement differ markedly from low field and low frequency AC magnetic moment results. The difference between these measurements is due to the difference in the probing procedure of the magnetic moment. The DC magnetic moment is defined as the ratio between magnetic moment and a fixed applied field. The AC magnetic moment is the ratio between AC magnetic moment and AC applied field [41]. In

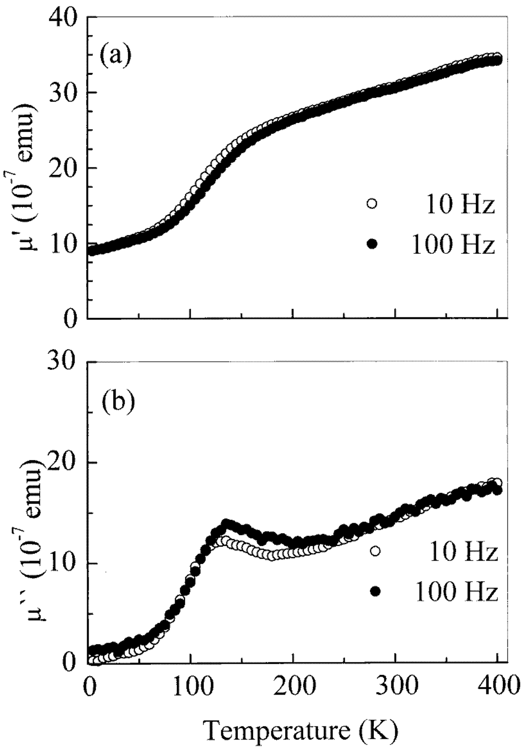

Figure 8. (a) Real ($\mu'(T)$) and (b) imaginary ($\mu''(T)$) parts of the low-field AC magnetic moment of the Fe/Cu multilayer as a function of the temperature. The temperature dependence of the AC magnetic moment was measured while cooling down the sample. A small memory effect dependent on the frequency for the $\mu'(T)$ curve and a typical frequency-shift peak of the $\mu''(T)$ curve are observed.

fact, μ_{AC} ($\omega \to 0$) tends to μ_{DC} only in perfectly ergodic systems. The ergodicity conditions are not expect to be attained in our sample, where a viscous magnetic state occurs and where the surface magnetic energy (predominant over volume magnetic energy) corresponds to a significant fraction of the total energy of the sample.

6. Final remarks

To summarize, we have shown that a magnetic metastable phase is present in our Fe/Cu multilayers. This interpretation is supported by different experimental results. Furthermore, we interpret this magnetic metastable phase as being due only to some interdiffusion between Fe and Cu at the interfaces. Besides, the magnetic cluster size in Cu layers must involve a relatively high number of Fe atoms due to substantially high values of the irreversibility temperatures measured.

In fact, our results suggest that the width of the interface mixed region should be small involving few monolayers; otherwise we could not explain the ferromagnetic behaviour of FC curves. Thus, to illustrate this mixed region, we can assume terrace-like magnetic clusters spaced by Cu layers.

Although several experimental results have revealed a mictomagnetic behaviour in our samples, the high irreversibility temperatures measured are less straightforward to understand.

For example, it is well known that the glass temperatures of thin layers of spin-glasses remain nonzero down to thicknesses of one or two monolayers. However, it is also shown that the glass temperatures decrease vary rapidly for decreasing thickness [42]. This would then exclude the possibility of regarding the multilayer as a simple addition of single layers of a spin-glass system. On the other hand, since the central portion of each layer is pure (Cu or Fe), the irreversible part of the system should originate from the Cu/Fe interfaces. If substantially large local magnetic fields are produced by the inner Fe atoms of the ferromagnetic layers, high temperatures would be necessary to change the magnetic configuration of interfacial clusters. The surprisingly high measured irreversibility temperatures could therefore arise from a certain rigidity of Fe clusters, constrained by such strong fields at the cluster sites. Further structural and magnetic characterization are in progress on these multilayers, through which we expect to achieve a deeper understanding of these processes. For example, Mössbauer measurements and scanning tunnelling microscopy should be used to clarify exchange coupling and microstructure in the Fe/Cu multilayer [43].

Finally, we would like to stress that SQUID magnetometry plays a crucial role in this study, specially in view of the extremely well resolved measurements that one can achieve by using this technique. Research on multilayers is still a rapidly growing field which benefits from studies including low field static as well as dynamic susceptibilities.

Acknowledgments

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