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Structural and magnetic properties of annealed $[Fe_{19}Ni_{81}]_{12\ Å}/Ag_{11\ Å}$ multilayers studied by Mössbauer spectroscopy, x-ray diffraction and magnetization measurements

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Abstract. The influence of annealing on the properties of an ultrathin permalloy/silver multilayer is analysed using Mössbauer effect, x-ray diffraction, magnetization and magnetoresistance experiments. A thick permalloy film is studied for comparison. From Mössbauer experiments, the existence of a 'magnetically dead' layer is ruled out. An interfacial zone corresponding to about 1.2 monolayers is observed in the low hyperfine field range in the as-deposited film and disappears after annealing at $T_a = 450\,^{\circ}C$, when the layer structure is destroyed. The evolution of the hyperfine field is associated with both densification of the NiFe layers and interface improvement. From Mössbauer and magnetization measurements, the individual Ni and Fe magnetic moments have been evaluated. They exhibit different thermal dependences, in agreement with results on multilayers based on pure Fe or Ni layers.

1. Introduction

Ultrathin permalloy/silver multilayers prepared by dc sputtering exhibit magnetoresistive (MR) properties related to the existence of antiferromagnetic coupling between NiFe layers. The MR ratio increases after annealing up to 250 °C and decreases at higher temperatures. The changes in structural and magnetic properties upon annealing are followed by x-ray diffraction, magnetization measurements, electrical resistivity experiments and can also be observed with ^{57}Fe Mössbauer spectroscopy.

The purpose of the present investigations is to study the annealing dependence of the properties of NiFe/Ag multilayers at the origin of the evolution of the magnetoresistive properties. We discuss the relationship of the information collected from ^{57}Fe in Mössbauer spectra, acting as a local probe, with bulklike properties such as magnetization and magnetoresistance.

2. Experimental procedure

The 5 μm thick $[Fe_{19}Ni_{81}]$/Ag samples were prepared by triode dc sputtering onto glass substrates maintained at 100 K; the permalloy layers were 12 Å thick and the silver layers

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were 11 Å thick. After the growth process, the temperature is increased from 100 K to 300 K and the multilayers spontaneously detach from their substrates. Thermal annealings were performed successively on the same film for 10 min at the following temperatures: 240, 300, 320, 360, 400 and 450 °C under a 10^{-5} Torr vacuum; the film was analysed at each step of the annealing treatment.

As a reference, a 2.5 μm self-supported film of $Fe_{19}Ni_{81}$ (permalloy), prepared under the same conditions as the multilayered film, was studied in the virgin state and after annealing at 500 °C for one hour. A 5000 Å thick permalloy film adherent to its glass substrate was also studied in the as-deposited state.

Conventional transmission (for the self-supported films) and conversion electron Mössbauer (for the adherent film) spectroscopies were performed for temperature ranging from 4 K to room temperature. The incoming γ-ray direction was normal to the sample surface. The recording time is about 8 days for a 10 μm thick sample with 1 cm^2 area. The radiation source was ^{57}Co in a Rh matrix. The natural ^{57}Fe abundance was sufficient for our measurements. The Mössbauer spectrum of the samples under study is a broad sextet because different iron sites coexist in the multilayers. Each site has its own hyperfine field (H_{hf}), isomer shift (IS) and quadrupole splitting (QS) and was simulated with six Lorentzian lines. According to Tomala's method [1], the recorded spectrum was fitted using a histogram for the hyperfine field distributions due to the different iron sites. A linear correlation between hyperfine field, isomer shift and quadrupole splitting was used to reproduce the spectrum asymmetry. The isomer shifts are given relative to metallic iron at 300 K.

X-ray reflectivity experiments were carried out on a Siemens two-circle goniometer using the Cu Kα radiation. Some of the x-ray results presented here were extracted from a fitting of the diffraction curves with the help of the SUPREX refinement program [2]. The magnetoresistance experiments were carried out on a 3 mm × 10 mm sample in a furnace that allows *in situ* resistivity measurements from 30 to 500 °C with applied field from −0.37 to +0.37 T. The magnetoresistance ratio (MR) is defined as $MR = 100(R_0 - R_s)/R_s$ where R_0 is the zero-field resistance and R_S is the resistance at high magnetic field (i.e. when all the magnetic moments are parallel). A standard four-probe method in a transverse geometry was used: the magnetic field is parallel to the plane of the layers and perpendicular to the current. Magnetization measurements were performed on a SQUID magnetometer between 5 K and 400 K. Some of these results have already been published [3, 4].

3. Results and discussion

3.1. Bulk permalloy film

Figures 1(a) and 1(b) present the room-temperature conversion electron Mössbauer and conventional transmission Mössbauer spectra of the adherent, 5000 Å thick $Fe_{19}Ni_{81}$ film and of the self-supported, 2.5 μm thick one, respectively. The Mössbauer spectra consist of magnetic superimposed sextets giving rise to hyperfine field (H_{hf}) distributions. The H_{hf} direction is 20 degrees out of the film plane for the adherent sample and 60 degrees for the self-supported sample. The H_{hf} direction is again in the film plane when a 10 mT magnetic field is applied as shown in figure 1(c). This is in agreement with magnetoresistance measurements, where an 8 mT applied field is enough to saturate the magnetoresistance of the self-supported film. Low-temperature growth of the films might induce strains and defects that are stabilizing factors for such a magnetic moment configuration where the shape anisotropy of the film is not the prevailing parameter.

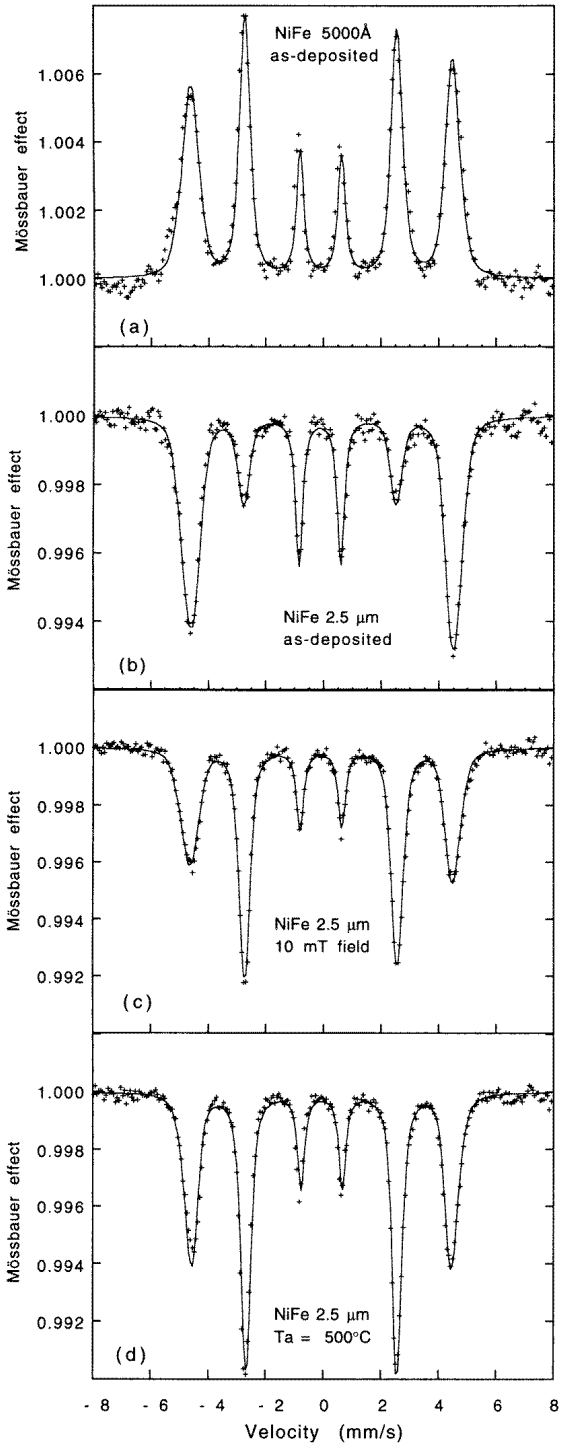

Figure 1. Mössbauer spectra recorded at 300 K for (a) as-deposited 5000 Å thick $Fe_{19}Ni_{81}$ film; (b) as-deposited 2.5 μm self-supported permalloy film; (c) 2.5 μm film with a 10 mT applied magnetic field; (d) 2.5 μm permalloy film annealed at $T_a = 500\,^{\circ}$C for one hour.

The spectra present some asymmetry: the area of the sixth line is equal to the area of the first one, but the full width at half maximum is different for these two lines. The same observation can be made for the second and the fifth lines.

A linear correlation between hyperfine field (H_{hf}) and isomer shift (IS) was first introduced to account for the asymmetric spectrum as usual in homogeneous disordered alloys; permalloy is supposed to form a homogeneous solid solution and each iron atom can have up to twelve iron atoms as nearest neighbours; variations in distances between adjacent atoms cannot be ruled out. Large values of H_{hf} are associated with small values of IS (large value of s electron density); this case corresponds to iron atoms surrounded by a maximum number of iron atoms as first neighbours while small values of H_{hf} (large IS) correspond to iron atoms surrounded by a maximum number of Ni atoms as first neighbours. But with such a fit the spectrum asymmetry is not fully reproduced. So we also introduced a quadrupole splitting (QS) distribution correlated with the hyperfine field one.

The same asymmetry was reported by Dumpich [5] for a 2000 Å thick $Fe_{19}Ni_{81}$ film deposited by electron beam evaporation onto quartz substrates maintained at room temperature. It was not observed by Zinn and Kalvius [6] in a 2.5 μm film evaporated on a glass substrate at 250 °C. Lippmaa *et al* [7] did not mention this asymmetry for a 2.5 μm thick FeNi foil. It can be assumed that asymmetry probably originates from stress due to the substrate and is mainly dependent on substrate temperature, argon pressure and applied voltage [8].

The calculated QS values are small and their mean value is about 0.008 mm s^{-1}. Generally, the electric field gradient is predominantly generated by the electronic orbitals in the environment of the resonant nucleus, and to a lesser extent by the distribution of lattice charges. In the present system, the origin of QS is not clear; the presence of distortions in the fcc lattice was not observed by x-ray diffraction, but the presence of some heterogeneities cannot be ruled out.

After thermal annealing at 500 °C (figure 1(d)), the H_{hf} direction is in the film plane as expected from the overall shape anisotropy of the film. This result is in agreement with magnetoresistance studies: the saturation field decreases from 8 mT for the as-deposited permalloy to 0.5 mT for the annealed film. $\langle H_{hf} \rangle$ measured at 4 K is 29.9 T for the as-deposited film and 29.4 T after thermal annealing, indicating a decrease of the iron magnetic moment upon appearance of atomic order. The $\langle \mathrm{IS} \rangle$ measured at 4 K is 0.130 mm s^{-1} for the virgin permalloy and 0.154 mm s^{-1} for the annealed sample. These phenomena can be correlated with the lattice expansion upon annealing, seen by x-ray diffraction measurements [3]: the lattice parameter is slightly smaller (-0.2%) in the as-deposited sample than in the annealed sample.

The spectra of the annealed permalloy recorded at 300 K (figure 1(d)) and 4 K are symmetrical; the best fit is obtained with QS equal to zero for every iron site indicating a more homogeneous, less disordered lattice. Moreover, one can observe in figure 2, relative to the H_{hf} distribution of bulk permalloy, a smaller width of the distribution after annealing; the IS distribution width has the same behaviour as the H_{hf} distribution. It can be assumed that the asymmetry of the as-deposited permalloy probably originates from stress, defects and inhomogeneities that are created during deposition and are relaxed by thermal treatment. Moreover the change in $\langle H_{hf} \rangle$, $\langle \mathrm{IS} \rangle$ and $\langle \mathrm{QS} \rangle$ upon annealing can also be interpreted as a homogenization of permalloy: some small parts of the as-deposited permalloy film could have variations in iron concentration (iron rich clusters have large H_{hf} and small IS) and concentration homogenization could occur during annealing. These remarks are in agreement with x-ray experiments carried out in the reflection geometry: one observes a decrease of the width of the (111) peak after thermal annealing indicating better structural quality.

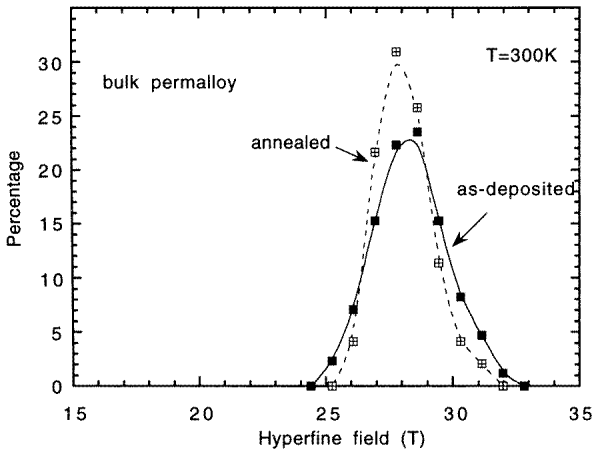

Figure 2. Hyperfine field distributions of the as-deposited and annealed bulk permalloy.

3.2. $[Fe_{19}Ni_{81}]_{12\ Å}/Ag_{11\ Å}$ *multilayered film*

After each step of thermal annealing, x-ray experiments, magnetization and magnetoresistance measurements were carried out. Some of the results have been already published [3, 4, 9] and can be summarized as follows.

3.2.1. Annealing studied by x-ray and magnetoresistance experiments. In the virgin state, x-ray experiments show that both Ag and NiFe are (111) textured. Silver expands in the (111) direction (about 1%) and is contracted perpendicularly, that is in the (220) direction (about 1.3%). The behaviour of NiFe is not so classical: NiFe expands in all directions; the (111) distances are 1.8% larger (see figure 3(a)) than in bulk NiFe and the (220) distances are also about 2% larger. This can be interpreted as the result of some alloying at the interfaces. Upon annealing, the (220) and (111) NiFe distances start decreasing significantly above 150 °C up to 320 °C, whereas Ag distances begin to relax only from 250 °C up to 380 °C (see figure 3(a) for the (111) Ag and NiFe distances and [4] for the (220) ones). The multilayered structure improves up to 320 °C, as shown in figure 3(b): the structural coherence length (the maximum thickness preserving a perfect atomic stacking order) is obtained from the width of the large-angle reflection peak by the Scherrer formula [10] and exhibits a maximum (about 140 Å, that is nearly seven bilayers) at this temperature. This improvement results from the increase of the distance between Ag and NiFe atoms at the interfaces with a simultaneous decrease of the width of its distribution (see figure 3(c)), that is interface smoothing. Above 320 °C, the multilayer starts degrading and above 380 °C, the destratification is completed: no more periodicity is found in large-angle reflection diffraction and the slope of the saturation resistance R_s/R_{smax} increases (figure 4(a)). The densification of the NiFe layers is accompanied by an increase in the Curie temperature, without any effect on the magnetic moment. As a result of this increase of Curie temperature and of the decrease in the saturation resistance, the magnetoresistance ratio $\mathrm{MR} = 100(R_0 - R_s)/R_s$ presents a maximum at 250 °C in figure 4(a) (12% in the as-deposited state, 16% after annealing at 250 °C). Above this temperature, the magnetoresistance decreases rapidly to zero at about 320 °C; in parallel, a remanent magnetization appears (see figure 4(b)), which is attributed to

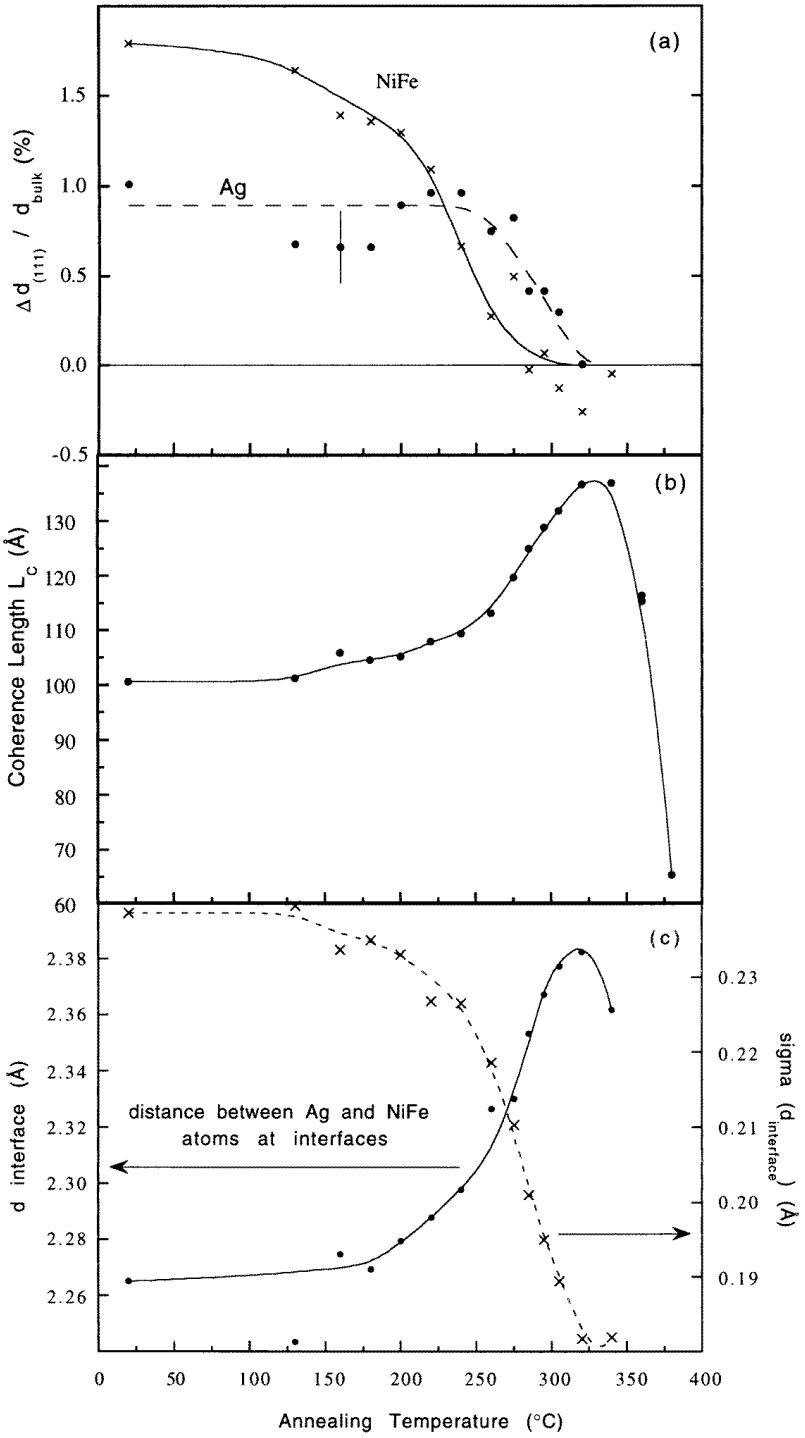

Figure 3. Annealing temperature dependence of (a) (111) distances in Ag and NiFe (relative to bulk ones); (b) coherence length; (c) distance between Ag(111) planes and NiFe(111) planes at interfaces and its distribution. Lines are guides for eyes.

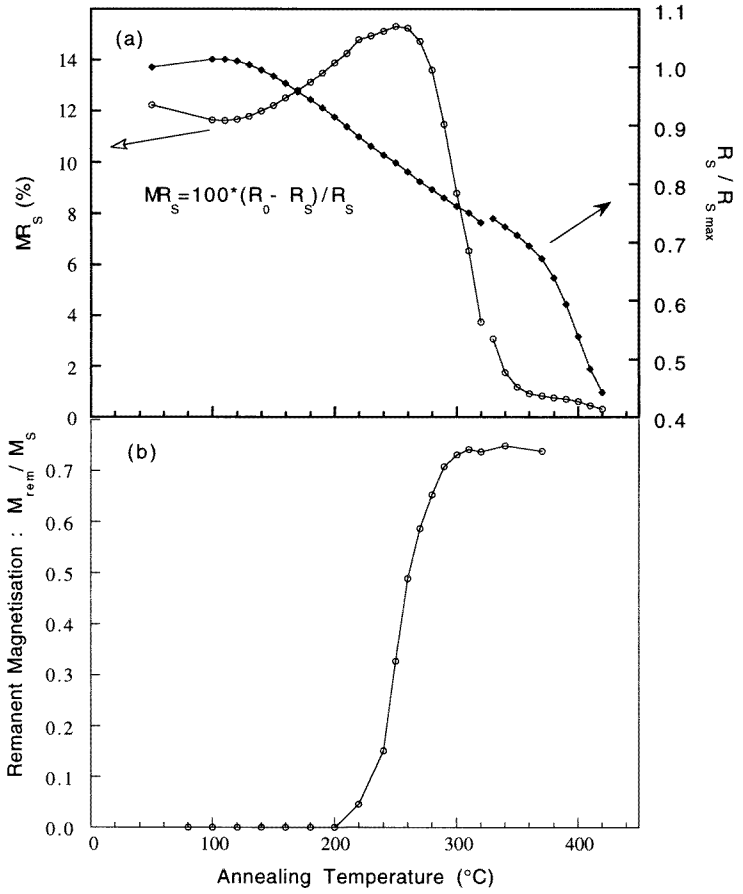

Figure 4. Annealing temperature dependence of (a) magnetoresistance (MR_S) and saturation resistance (the curve discontinuities are due to the use of two different samples); (b) remanent magnetization.

the formation of ferromagnetic bridges. Their number is large enough to lead to a decrease of the magnetoresistance from 250 °C, but they can only be detected by x-ray diffraction above 320 °C.

3.2.2. Annealing studied by Mössbauer spectroscopy carried out at 300 K and 4 K. Mössbauer spectra were recorded at 300 K (figure 5) and at 4 K on the multilayer film after each step of the annealing procedure. From figure 5, one can see that no non-magnetic layer is present at interfaces since there is no paramagnetic contribution in the centre of the spectra. Moreover, the relative intensities of the lines indicate that the magnetization lies in the film plane.

The $\langle H_{hf} \rangle$ values of the as-deposited multilayer film, measured at 4 K, is about 3.5% (and 2%) smaller than the as-deposited (and annealed) bulk permalloy one: the lowering of $\langle H_{hf} \rangle$ can be associated with the lattice expansion (2%) and the presence of the interfaces; this last contribution is all the more important as the permalloy layers are thin. The $\langle$IS$\rangle$ value

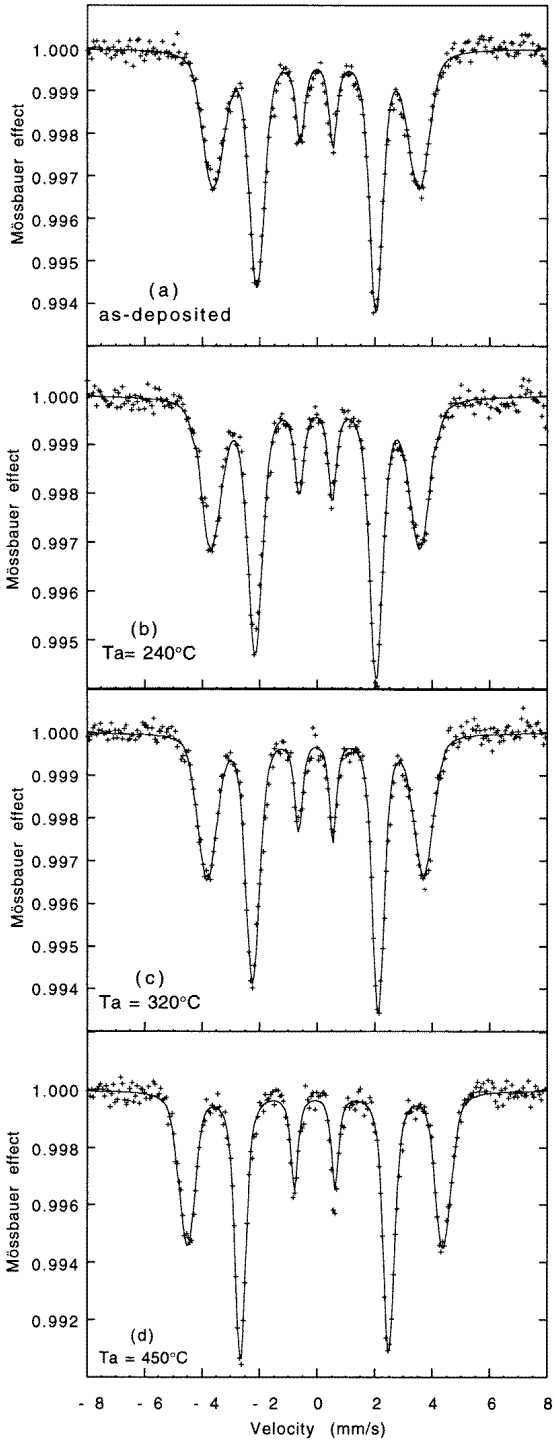

Figure 5. Mössbauer spectra recorded at 300 K for $[Fe_{19}Ni_{81}]_{12\ Å}/Ag_{11\ Å}$ multilayer: (a) as-deposited; (b) annealed at $T_a = 240\,^{\circ}C$; (c) annealed at $T_a = 320\,^{\circ}C$; (d) annealed at $T_a = 450\,^{\circ}C$.

estimated at 4 K is 0.184 mm s^{-1}; the evolution occurring between bulk and multilayered film is in agreement with the two above hypotheses: IS increases because of lattice expansion as observed in bulk permalloy, as it does for increasing Ag concentration in FeAg alloys. This last observation was reported by Larica *et al* [11] on Fe_xAg_{1-x} alloys studied at 5.5 K; for example IS changes from +0.20 mm s^{-1} for $Fe_{0.5}Ag_{0.5}$ to +0.38 mm s^{-1} for $Fe_{0.2}Ag_{0.8}$. This agrees with x-ray experiments, where alloying at interfaces was proposed as an explanation for the expansion of NiFe in all directions.

From the analysis of the hyperfine field distributions measured at 4 K and presented in figure 6(a), the H_{hf} distribution of the as-deposited multilayer film can be divided into two components. One component was taken identical to the as-deposited bulk permalloy distribution, with $\langle H_{hf}\rangle = 29.9$ T and accounts for 60% of the iron sites. As a 12 Å thick permalloy layer corresponds to about six monolayers (6 ML), the bulklike part of the permalloy layer involves 3.6 ML. Then we assumed that the remaining contribution (40%) is from permalloy at the interfaces and involves 1.2 ML at each interface with $\langle H_{hf}\rangle = 26.8$ T.

Upon annealing up to 450 °C, the $\langle H_{hf}\rangle$ value of the multilayer, measured at 300 K and at 4 K, is approaching the bulk NiFe value (figures 6(b) and 7(a)). Mössbauer spectra of the multilayered film annealed at 450 °C and of bulk permalloy annealed at 500 °C are identical if recorded at 4 K as seen from the deduced hyperfine field distributions plotted in figure 6(c). The $\langle H_{hf}\rangle$ values at 4 K are independent of annealing up to $T_a = 400$ °C and then slightly increase from 28.8 to 29.3 T indicating that the magnetic moment is only slightly perturbed.

A wide range of $\langle H_{hf}\rangle$ values (22.1 to 27.6 T) is observed when spectra are recorded at 300 K in agreement with large variations in the Curie temperature (T_C). At 300 K, one can observe that $\langle H_{hf}\rangle$ is increasing very slowly, up to the annealing temperature of 320 °C; this corresponds to an increase of T_C from about 500 to 600 K, estimated from magnetization measurements, which is due to both the improvement of the interface quality and to the densification of the NiFe lattice. For comparison the Curie temperature of bulk permalloy is 850 K. Then $\langle H_{hf}\rangle$ increases rapidly and finally nearly reaches the bulk value at 450 °C. At that temperature $\langle H_{hf}\rangle$ is only 0.5 T smaller than for annealed bulk permalloy.

The large increase of $\langle H_{hf}\rangle$ starts when the MR ratio is already zero. This means that the Curie temperature of the material approaches that of bulk permalloy only when destratification takes place, i.e. when the size of the NiFe grains is such that the percentage of magnetic atoms at the surface is small compared to that of magnetic atoms in the bulk.

The H_{hf} distributions relative to experiments carried out at 300 K, presented in figure 6(b), show that the distribution width is nearly constant for annealing temperature up to 320 °C; it is smaller at 450 °C, but still larger than for annealed bulk permalloy (figure 6(b) is compared with figure 2).

The annealing temperature dependence of $\langle$IS$\rangle$ is shown in figure 7(b); the isomer shifts of the as-deposited multilayer, measured at 300 K and at 4 K, are larger than those corresponding to bulk permalloy until the annealing temperature reaches about 300 °C. At this temperature, the permalloy lattice is being relaxed, Ag atoms diffuse and destratification starts. So the change in $\langle$IS$\rangle$ indicates again that this parameter is very sensitive to the modification of interfaces. The dependence of the IS distribution width upon annealing is similar to the above-mentioned $\langle H_{hf}\rangle$ distribution width.

The mean value of the quadrupole splitting is small whatever the annealing temperature. This parameter is changing at 450 °C from negative (with a large distribution) to zero (with a very narrow distribution if any). This evolution can be directly observed looking at the asymmetry of the spectra in figure 5. The symmetrical spectrum of the multilayered film annealed at 450 °C indicates that the iron environment is homogeneous.

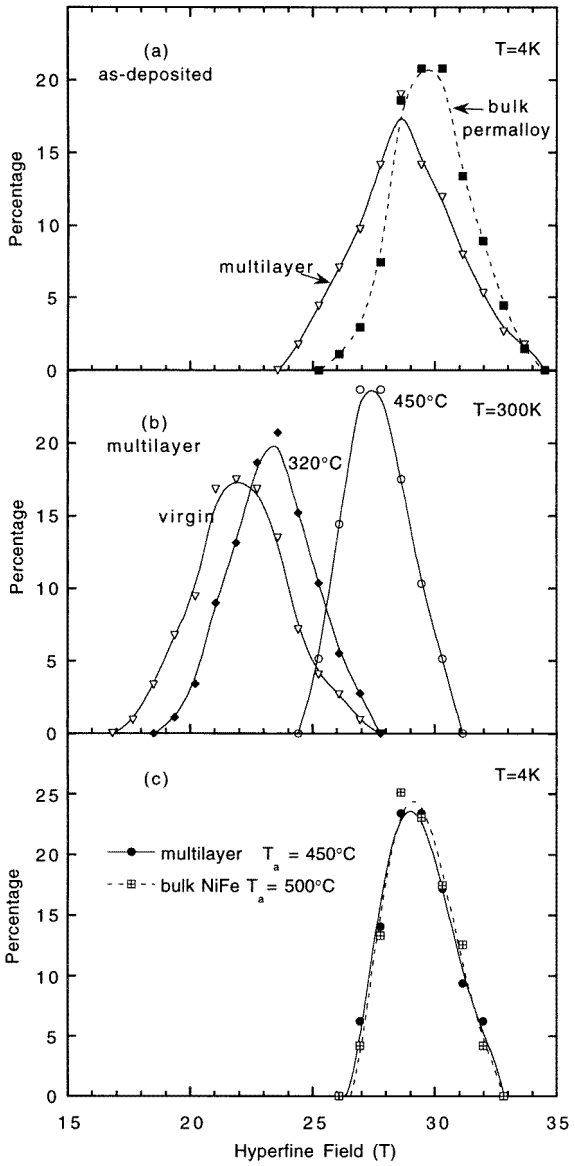

Figure 6. Hyperfine field distributions: (a) of the as-deposited bulk permalloy and of the as-deposited multilayer; (b) of the multilayer at different steps of the annealing process; (c) of the bulk permalloy annealed at $T_a = 500\,^{\circ}$C and of the multilayer annealed at $T_a = 450\,^{\circ}$C.

3.3. Temperature dependence of hyperfine field and magnetization: Debye temperature and coupling interactions

Mössbauer spectra were recorded from 4 K to 295 K on the as-deposited sample, on the sample annealed at $T_a = 240$, 320, 450 °C and on the thick permalloy film. From the temperature dependence of the isomer shift, we can determine the Debye temperature Θ_D

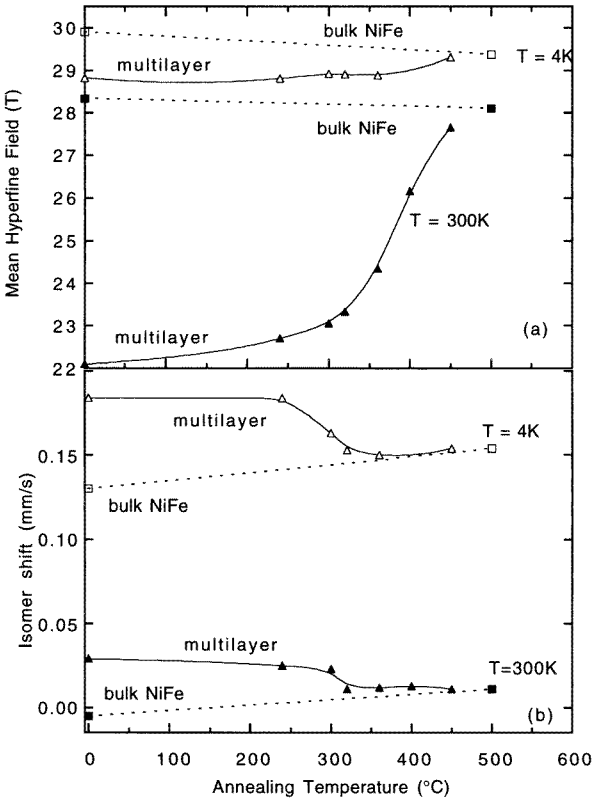

Figure 7. Annealing temperature dependence (a) of the mean hyperfine field $\langle H_{hf}\rangle$; (b) of the mean isomer shift ⟨IS⟩ of for $[Fe_{19}Ni_{81}]_{12\ Å}/Ag_{11\ Å}$ multilayer calculated from Mössbauer spectra recorded at 300 K and at 4 K (solid triangle is for multilayer at 300 K, open triangle for multilayer at 4 K, solid square for bulk permalloy at 300 K and open square for bulk permalloy at 4 K).

of the films. Θ_D was estimated at 310 K and 500 K $\pm$ 20 K for the as-deposited sample and for the sample annealed at 320 °C respectively.

The variation of the ratio $\langle H_{hf}\rangle/\langle H_{hf}\rangle_{4\ K}$ as a function of temperature is shown in figure 8(a) for the as-deposited multilayered sample and for the films annealed at 240, 320 and 450 °C. For comparison, the variation of the saturation magnetization normalized to its value at 0 K ($\mu_{alloy,T}/\mu_{alloy,0\ K} = M_s/M_{s,max}$) is presented in figure 8(b). The saturation magnetization is the mean value of the magnetic moments of iron and nickel atoms in FeNi alloy, whereas H_{hf} is related to iron atoms only. One can observe that H_{hf} has a nearly linear temperature dependence for $T > 70$ K with different slopes for each annealing treatment. This behaviour is different from the temperature dependence of the mean magnetic moment.

The iron and the nickel magnetizations were evaluated separately (figure 8(c)) assuming $\mu_{alloy} = 0.81\mu_{Ni} + 0.19\mu_{Fe}$ and the empirical formula $H_{hf} = a\mu_{Fe} + b\mu_{alloy}$ [12, 13] with $a = 8.7$ T μ_B^{-1} and $b = 6.05$ T μ_B^{-1} [14], where the coefficients a and b were estimated from the variations of H_{hf} and μ_{alloy} as a function of concentration in fcc FeNi alloys. At 4 K, the measured value of μ_{alloy} in the multilayers [9] is about 0.98 μ_B (3% lower

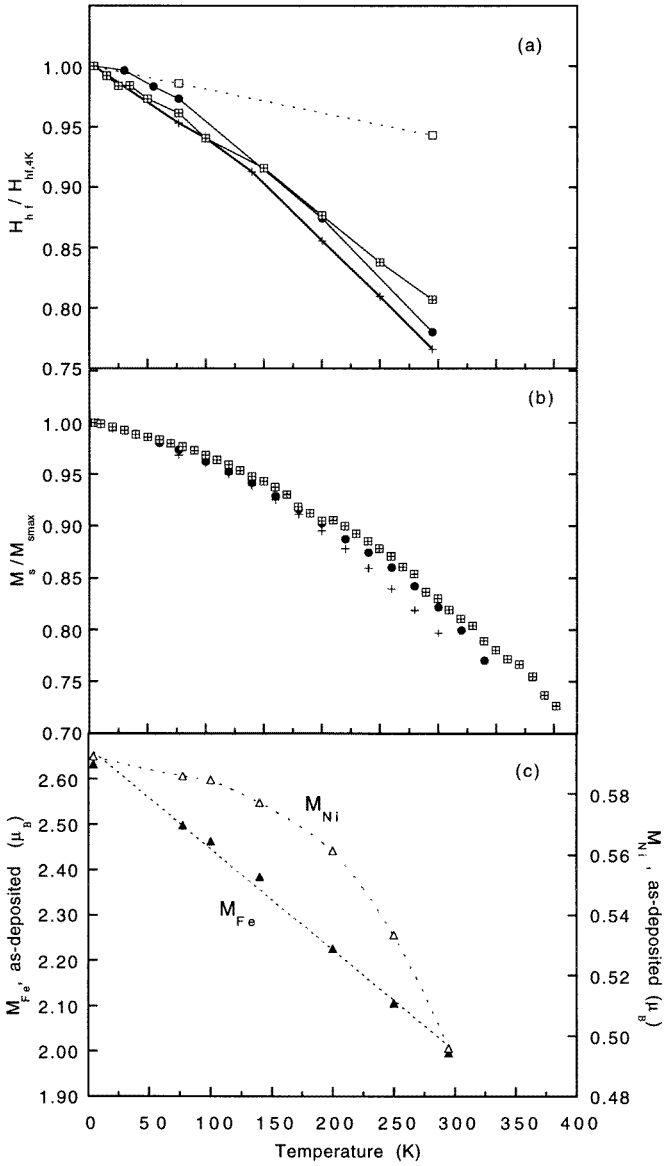

Figure 8. Temperature dependence of: (a) normalized mean hyperfine field; (b) normalized total magnetization of as-deposited multilayer (cross), multilayer annealed at $T_a = 240\,^{\circ}$C (closed circle), $T_a = 320\,^{\circ}$C (cross in square) and $T_a = 450\,^{\circ}$C (square); (c) Fe magnetization contribution (full triangle) and Ni magnetization contribution (open triangle) of the as-deposited multilayer. Lines are guides for eyes.

than the bulk value) corresponding to $\mu_{Fe} = 2.64\ \mu_B$ and $\mu_{Ni} = 0.59\ \mu_B$. These values are in agreement with $\mu_{Fe} = 2.8\ \mu_B$ and $\mu_{Ni} = 0.6\ \mu_B$ given for bulk permalloy [15]. μ_{Fe} exhibits a nearly linear temperature dependence for $T > 70$ K with different slopes after each annealing treatment, that is with different magnetic coupling intensities. This linear evolution indicates a two-dimensional behaviour as observed in Fe/Cr [16] and Fe/Ag

multilayers [17]. Ni atoms have a more tri-dimensional behaviour as reported in the Ni/Ag case [18]. These observations could suggest that Ni and Fe in the present multilayers have the same magnetic behaviour as if they were pure metals, and that Fe is more sensitive than Ni to the thinness of the magnetic layers.

4. Conclusions

Mössbauer spectra of bulk permalloy are typical of a disordered alloy. Some heterogeneities in the concentration of the as-deposited alloy and a strained lattice could explain the observed asymmetrical spectra with broad lines. Upon annealing, concentration homogenization and lattice expansion are the main factors that can account for the change in the Mössbauer parameters.

In the multilayers, the iron magnetic moment is in the film plane and the presence of a dead layer is not observed. The spectra of the as-deposited sample can be interpreted assuming two contributions: one from the central part of the magnetic layers (60%) and one from an interfacial zone (40%). This last one could be described as roughness or as a magnetic NiFeAg alloy. Upon annealing, the change in isomer shift, sensitive to the vicinity of silver, can be correlated with destratification above $T_a = 320\,^{\circ}\text{C}$.

The increase of $\langle H_{hf} \rangle$ when measured at 300 K, compared to the almost constant value measured at 4 K, is interpreted as an increase of the Curie temperature: the small change for annealing temperatures below 320 °C is associated with both NiFe lattice densification and interface improvement. For higher annealing temperatures, the layered structure disappears and $\langle H_{hf} \rangle$ increases rapidly up to the value corresponding to bulk permalloy.

From the combination of magnetization data (relative to Fe and Ni atoms) and hyperfine field (relative to iron atoms only) as a function of temperature one can determine separately the iron and nickel contributions. For the as-deposited multilayer, as well as for the annealed samples, the iron moment has a linear temperature dependence indicating a two-dimensional behaviour, whereas the nickel magnetic moment has a more tridimensionial behaviour.

Acknowledgments

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