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Evidence for complete ion-beam mixing in thermally immiscible Fe/Ag multilayers from conversion electron Mössbauer spectroscopy

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Ion-beam mixing in Fe/Ag multilayers is studied by Ar^+ ion irradiation at 325 and 110 K using conversion electron Mössbauer spectroscopy (CEMS) and secondary ion mass spectroscopy. The present experiments show that Fe/Ag multilayers exhibit almost complete ion-beam mixing, in contrast to the earlier results in a bilayer system where only a significantly low mixing efficiency was observed. In the present study, the ion-beam mixing is found to be more rapid at 110 K as compared to the irradiation at 325 K. The ion-beam mixing at 325 K is not homogeneous even at a fluence of 3×10^{16} ions/cm^2, as one can observe the presence of a sharp sextet with the hyperfine field of 33 T (corresponding to Fe atoms in the interior of the α-Fe) along with a doublet and a broad sextet, whereas for the 110 K irradiation we observe that the Fe is intermixed with Ag at a fluence of 1×10^{15} ions/cm^2 as evidenced by the presence of a single doublet only in the CEMS. The observation of complete mixing in Fe/Ag multilayers but not in a bilayer is attributed to the increase in interfacial free energy stored in the interfaces of the as-prepared sample and to the reduction of atomic migration length for intermixing and alloying due to nanosized Fe layer thickness.

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Ion-beam mixing in metallic layers has been studied for a variety of systems.[1] In most of the bilayer systems composed of miscible elements (having negative heat of mixing), the measured mixing rates were up to ten times larger than those predicted ballistically,[2] and found to depend on thermodynamic properties like heat of mixing and cohesive energy. Transient diffusion in local thermal spikes was able to explain these results for a overwhelming number of miscible bilayer systems.[3] However, the situation in the immiscible system like Fe/Ag is quite different, as there is no chemically driven transport. For these immiscible systems (having positive heat of mixing), there is little experimental work on ion-beam mixing.[4] Moreover, ion-beam mixing in these systems has become important now as nanoscale granular Fe/Ag and Co/Ag films that can be prepared by ion-beam mixing exhibit the technologically important phenomenon of giant magnetoresistance.[4] The present investigation is on thermally immiscible Ag/Fe multilayers. According to the fractal geometry approach of Cheng,[5] spikes are found in systems having a mean atomic number ($\overline{Z}>20$). Formation of thermal spikes is expected in the Fe/Ag system due to its high mean atomic number ($\overline{Z}=36.5$). Further, due to the large positive heat of mixing in both the solid and liquid phases ($\Delta H_{mix}^{sol}=42$ kJ/mole, $\Delta H_{mix}^{liq}=28$ kJ/mole), these systems have an extremely low solubility within the solid and liquid equilibrium states. Ion-beam irradiation of energetic inert gaseous ions on Ag/Fe bilayer at room temperature and also at low temperature has been carried out by Sosa *et al.*[6] in order to study the mixing mechanism. These authors observed a very poor mixing efficiency, which is significantly smaller than the prediction of the ballistic model. The low mixing has also been attributed to the demixing and phase separation occurring in the thermal spike phase of the collision cascade. The situation of ion-beam mixing in multilayers is different as there is a certain number of interfaces which posses interfacial free energy. The interfacial free energy could contribute significantly in raising the initial state of the films to a highly energetic nonequilibrium state. Further, in multilayers having nanosized layers, the atomic migration length required for intermixing and alloying is reduced considerably, which could alter the ion-beam mixing phenomenon in them. In this paper, we report the mixing of Fe/Ag multilayers by low energy ion irradiation at 110 K. The local magnetic properties of Fe layers have been studied by measuring the magnetic hyperfine fields at Fe sites using ^{57}Fe conversion electron Mössbauer spectroscopy (CEMS). The secondary ion mass spectroscopy (SIMS) technique has been used for the depth profiling of the multilayers. The mixing in Fe/Ag multilayers was found to be substantially different from the one that was reported earlier for bilayers.

The $\mathrm{Ag}(185\ \text{Å})/[^{57}\mathrm{Fe}(70\ \text{Å})/\mathrm{Ag}(185\ \text{Å})]_{x2}$ multilayer films were deposited on Si[110] substrates, using ion-beam sputtering with 99.5% enriched ^{57}Fe and 99.99% pure Ag as targets in an argon atmosphere at room temperature. The rate of deposition was 8 Å/min and 5 Å/min for the Fe layer and Ag layer, respectively. The thicknesses of the individual layers were confirmed by x-ray reflectivity measurements and the films were polycrystalline. These samples were irradiated with 85 keV Ar^+ ions with a fluence range of 5×10^{14}–3×10^{16} ions/cm^2 at room temperature and 110 K at a vacuum of $\sim3\times10^{-7}$ torr by using a 150 kV ion accelerator. In order to avoid beam heating, the beam current was limited to 0.5 μA/cm^2. The range of penetration of Ar^+ ions in the multilayered sample was found to be 397 Å using the Monte Carlo simulation SRIM 2000.[7] The Ar^+ ion energy is chosen to maximize damage in the film. A CAMECA-IMS 4F SIMS instrument was used for depth profiling of the as-prepared and irradiated samples. The primary ions used for sputtering

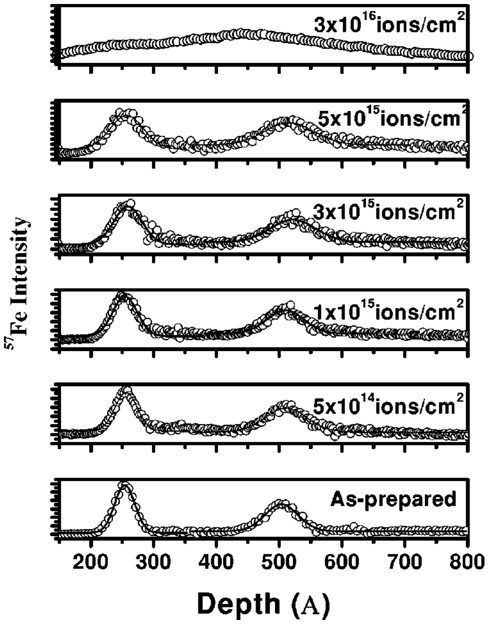

FIG. 1. ^{57}Fe concentration profile obtained from SIMS as a function of depth in Fe/Ag multilayer after irradiation with 85 keV Ar^+ ions to various fluences at room temperature. The dots correspond to the measured values and the continuous line corresponds to the fitted curve.

are Cs^+ ions of energy 5.5 keV with an ion current of about 1 nA with a raster of 200 μm×200 μm. Conversion electron Mössbauer spectroscopy measurements were performed with a ^{57}Co(Rh) radioactive source of strength $\sim$25 mCi. The conversion electrons were detected by a He/CH_4 (5% methane) gas flow proportional counter. The velocity calibration was done with a natural iron absorber. The isomer shifts are given relative to natural iron at room temperature. The magnitude of the magnetic hyperfine fields at Fe sites is assumed to be approximately proportional to the Fe magnetic moments.[8]

The depth profile of ^{57}Fe of the as-prepared sample is somewhat skewed towards higher sputtering time. The ^{57}Fe profiles were corrected for the Cs^+ irradiation broadening in order to determine the correct value of the square of the mixing length using the formalism given in Refs. 9 and 10. The thin film solution to Fick's law was applied to measure the mixing length (Dt). Figure 1 shows the Fe concentration profile as a function of irradiation fluence. A broadening of the individual layers as a function of fluence is observed, indicating atomic mixing. In order to determine the mixing rate, we have calculated the broadening of the first ^{57}Fe layer as a function of irradiation fluence. Figure 2 shows a plot of Dt versus ϕ for the samples irradiated with 85 keV Ar^+ ions. The linear dependence of Dt vs ϕ, indicates that ballistic mixing has taken place in the system.[11]

The SIMS profiles of samples irradiated at a low temperature of 110 K with various fluences are shown in Figs. 3(a)–3(d). The broadening of the individual layers as a function of fluence is more rapid than observed on irradiation at room temperature. The system is completely mixed on irradiation with a fluence of 1×10^{15} ions/cm^2 at 110 K, as seen from the SIMS profile in Fig. 3(c).

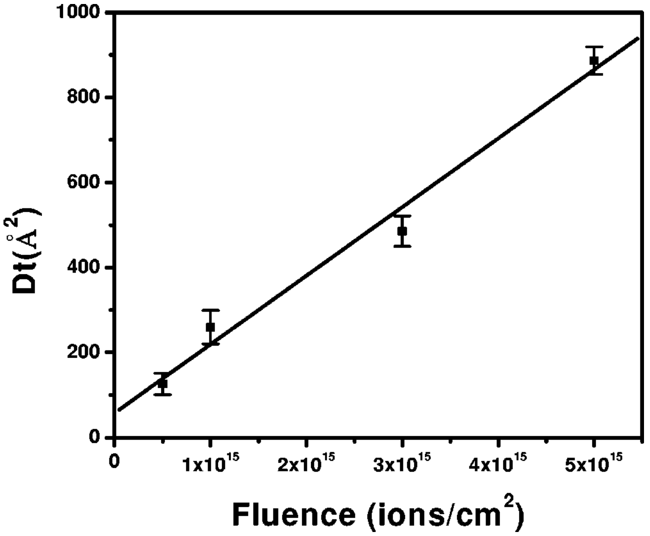

FIG. 2. Dt versus ϕ plot for an Fe/Ag multilayer irradiated with 85 keV Ar^+ ions at 325 K.

The ^{57}Fe Mössbauer spectrum of the as-prepared sample is shown in Fig. 4(a). The as-prepared sample spectrum consists of a sharp sextet with a hyperfine field of 33 T which corresponds to α-Fe in the bulk Fe layer and a broad sextet corresponding to the Fe atoms at the interface between the Fe and Ag layers with different atomic configurations, i.e., the number of nearest neighbor Fe atoms around Fe is varied

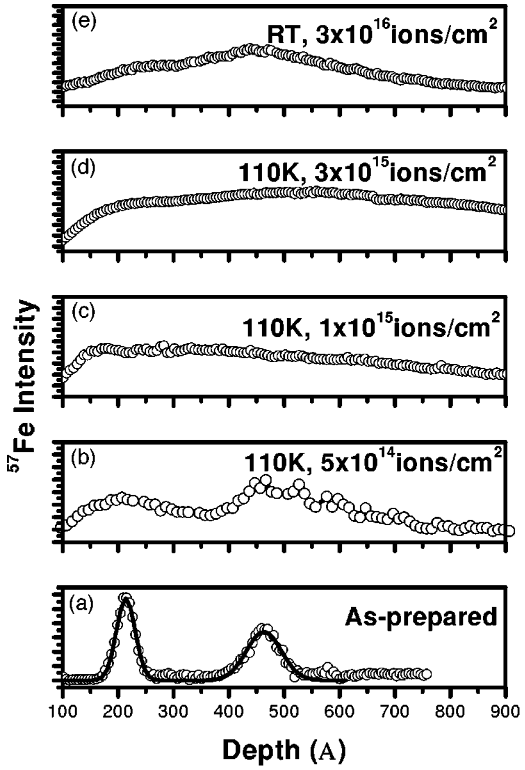

FIG. 3. The SIMS depth profile of ^{57}Fe: (a) the as-prepared sample; (b)–(d) the samples irradiated at 110 K with various fluences; and (e) the sample irradiated with the fluence of 3×10^{16} ions/cm^2 at room temperature.

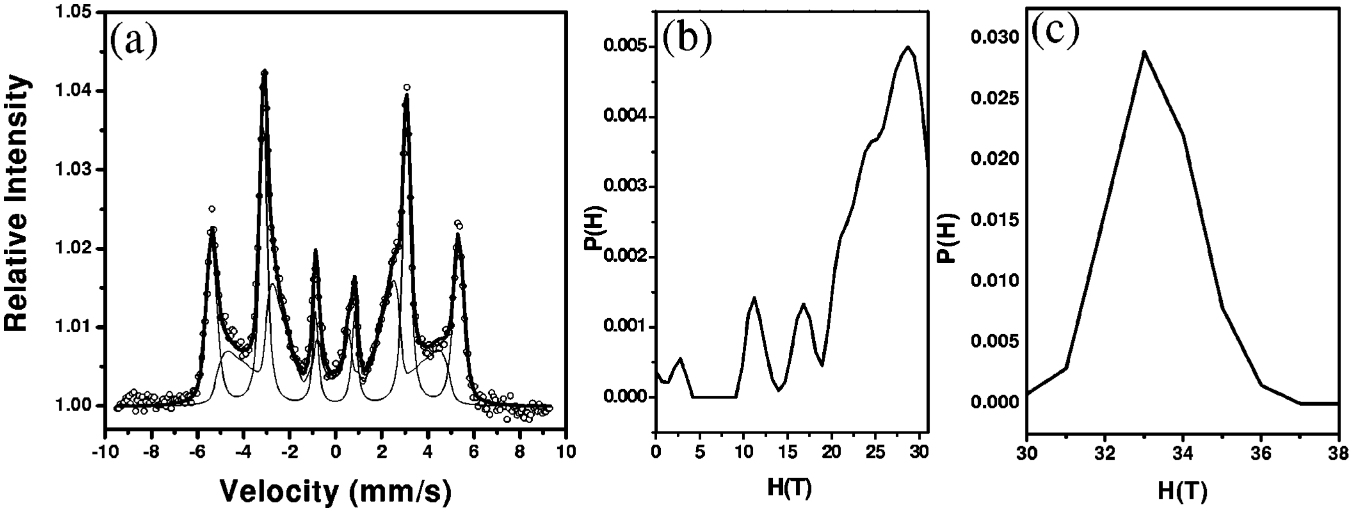

FIG. 4. (a) The CEMS spectra of the as-prepared sample: the continuous curve (thick line) represents the best fit to the experimental data, obtained by taking two independent hyperfine field distributions corresponding to the sharp sextet representing bulk α-Fe and the remaining broad sextet representing the interfacial region. (b) and (c) The hyperfine field distribution of the broad sextet and the sharp sextet, respectively.

(i.e., one, two, and three Fe atoms are replaced by Ag atoms in a total of eight nearest neighbor atoms).[12] The relative intensities of the two components are 52% and 48%, respectively. The area under the broad hyperfine component has contributions from Fe atoms from the intermixed region at the interface. A qualitative estimate of the intermixed layer is obtained from the Mössbauer spectrum. If one can neglect the difference in the recoilless fraction of the Fe atoms in the bulk α-Fe layer and interfacial region, one can take 47% of the Fe atoms as residing in the intermixed region. The absence of a doublet component in the Mössbauer spectrum indicates the interface is Fe rich. Taking the composition of interdiffused layer as $Fe_{0.5}Ag_{0.5}$,[13] about 45% of Fe atoms in the interdiffused region give approximately 1.3 nm thickness at each interface. The Mössbauer spectrum of the irradiated sample with a fluence of 3×10^{16} ions/cm^2 at room temperature (given in Fig. 5) has three components: (i) a sharp sextet with a hyperfine field of 33 T, (ii) a broad sextet from the Fe atoms at the interface, and (iii) a doublet (with a peak value of the quatrupole splitting of 0.75 mm/s and width of the quadrupole distribution of 0.98 mm/s) due to the Ag-rich intermixed region. The relative areas of the three components are 15%, 54%, and 31%, respectively. The 15% of the sharp sextet with a hyperfine field of 33 T can be from either α-Fe in the Fe layer or ferromagnetic Fe atoms in the interior of the α-Fe particles. The SIMS measurement on the sample irradiated with a fluence of 3×10^{16} ions/cm^2 at room temperature [given in Fig. 3(e)] does not show the presence of layer structure. Hence 15% of the sharp sextet is expected to arise from Fe atoms in the interior of the α-Fe particles.[14]

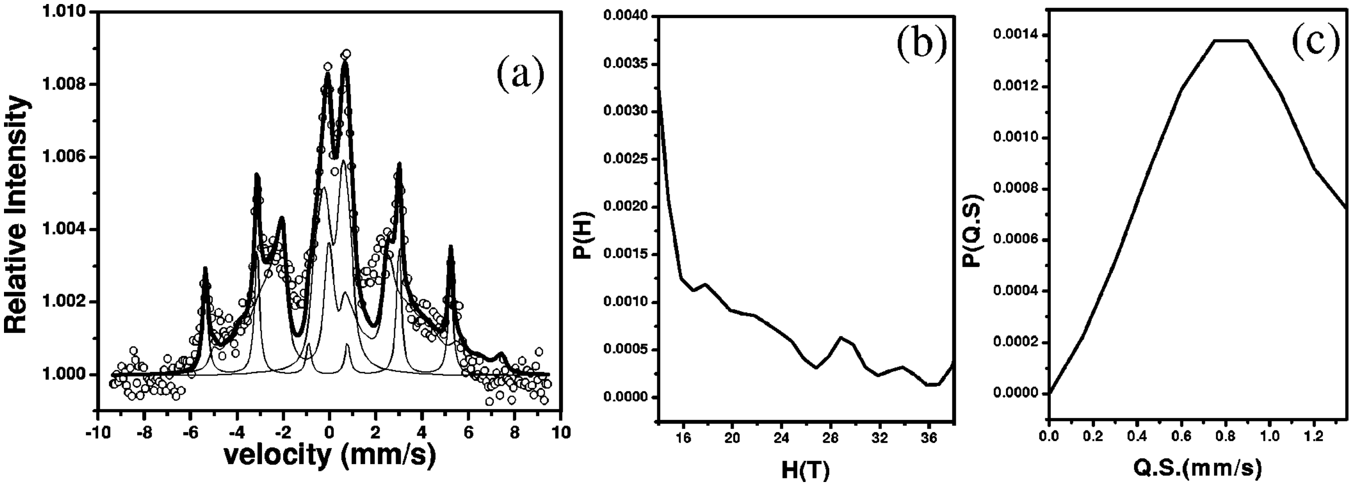

FIG. 5. (a) The CEMS spectra of the sample irradiated at room temperature up to a fluence of 3×10^{16} ions/cm^2. The continuous curve (thick line) represents the best fit to the experimental data, obtained from the hyperfine field distributions representing the interfacial region and the discrete Fe hyperfine field representing the bulk α-Fe and the quadrupole splitting distributions. (b) The hyperfine field distributions arising from the interfacial region. (c) The quadrupole splitting distributions.

54% of the broad sextet corresponds to the Fe atoms at the interface between the α-Fe particle and Ag matrix with different atomic configurations. In comparison with the as-prepared sample, the number of Fe atoms at the interface has now increased. Moreover, there is a significant number of Fe atoms completely surrounded by Ag atoms, which give rise to the doublet.

In Fig. 6(a), we present the Mössbauer spectrum of the sample irradiated at 110 K to a fluence of 5×10^{14} ions/cm^2. The spectrum is fitted with three components as was done in the case of the sample irradiated at room temperature. The relative areas of the three components (i.e., a sharp sextet, a broad sextet, and a doublet) are 14%, 51%, and 35%, respectively. In this case, 14% of the sharp sextet with a hyperfine field of 33 T can arise from both α-Fe in the bulk Fe layer and ferromagnetic Fe atoms in the interior of the α-Fe particles. The SIMS result [Fig. 3(b)] shows that the layer structure is still present; hence the mixing is not complete at this fluence. However, on further irradiation, to a fluence of 1×10^{15} ions/cm^2 at 110 K [Fig. 6(b)], the Mössbauer spectrum shows only a single broad doublet with a peak value of quadrupole splitting of 0.69 mm/s and the width of quadrupole distribution being 0.83 mm/s. This suggests that the Fe is completely intermixed with Ag as the Fe environment is nonmagnetic. Lonworth and Jain[15] and Marest *et al.*[16] have implanted ^{57}Fe in Ag up to a fluence of 5×10^{16} ions/cm^2 and characterized the samples by Mössbauer spectroscopy. They have reported that Fe atoms completely surrounded by Ag atoms give rise to a singlet with an isomer shift around 0.5 mm/s. But the absence of such a singlet in our sample is not unexpected as the average iron percentage is around 30% (assuming the sample is completely mixed) and hence iron atoms may not have isolated Ag atoms at all the 12 nearest neighbor sites, a necessary condition for cubic symmetry. Some of these sites are likely to be occupied by iron atoms, thereby destroying cubic symmetry. The observed isomer shift (0.35 mm/s) in our sample is close to the isomer shift observed in an Fe-Ag alloy,[17] which indicates that the intermixed state of our sample could be a random alloy. The Mössbauer spectrum of the sample irradiated with a fluence of 3×10^{15} ions/cm^2 at 110 K [Fig. 6(c)] shows a single broad doublet only. However, the quadrupole splitting and the width of its distribution have been reduced considerably (0.60 mm/s and 0.62 mm/s). The reduction in the width of the distribution in quadrupole splitting suggests that the intermixed region has become more homogeneous. The reduced average value of the quadrupole splitting suggests that the local environment around Fe atoms has become more symmetric.

The present experiments clearly demonstrated that Fe/Ag multilayers irradiated by Ar ions exhibit ion-beam mixing. The ion-beam mixing as a function of irradiation fluence is more rapid when irradiated at 110 K as compared to 325 K. The ion-beam mixing at 325 K is not completely homogeneous even at a fluence of 3×10^{16} ions/cm^2, whereas at 110 K we observe that the Fe is completely intermixed at a fluence of 1×10^{15} ions/cm^2, as evidenced from the Mössbauer measurements. These observations are in contrast to the observation of Sosa *et al.*[6] in an Ag/Fe bilayer, where they have shown that the ballistic ion mixing of the Ag/Fe interface is significantly counterbalanced by a chemically driven relaxation mechanism, which results in the demixing of the two metals. The thermal spike diffusion process was considered to be the cause of demixing even at low temperatures. Neubauer *et al.*[18,19] proved the importance of thermal spikes in mixing and estimated the average size of thermal spikes to be about 5 nm. These authors also observed the transport of a few Ag atoms into Fe, limited to the first 15 nm depth from the surface. The above results for the bilayer systems are consistent with the observation of Averback *et al.*,[20] who found that systems which are miscible in the liquid state, but not in the solid state, are intermixed on ion irradiation at 6 K but not when irradiated at 300 K. The system Ag/Fe is immiscible even in the liquid state and hence does not mix at any temperature. As stated above, we have observed a complete mixing in Ag-Fe multilayers at 110 K. We attribute this observation to the nature of our sample. The Fe layer thickness of 7 nm used in our case is substantially smaller than the thickness (67 nm) used in the earlier work[6] and hence the atomic migration length[21] required for intermixing and alloying between Ag and Fe layers is substantially reduced. Additionally, our sample consists of four interfaces in comparison to two interfaces of earlier work,[6] which will increase the interfacial free energy. The interfacial free energy is positive in proportion to the fraction of the interfacial atoms versus the total atoms in the film. We have calculated the interfacial free energy of our sample by using the formalism of Liu *et al.*,[21] which is as follows.

According to Gerkema and Miedema,[22] the interfacial free energy is

$$\Delta G^f = \alpha \Delta G_f^0, \tag{1}$$

where α is the fraction of interfacial atoms versus the total atoms in the films and ΔG_f^0 is the interfacial free energy per mole of interfacial atoms and is calculated from $\Delta G_f^0 = S^{AB}\gamma^{AB}$, where S^{AB} is the surface area occupied by one mole of interfacial atoms, γ^{AB} is a constant for a specific system. γ^{AB} can be calculated by[22]

$$\gamma^{AB} = 0.15(\gamma_A^{S,0} + \gamma_B^{S,0}) + \gamma_{SS}^{chem}, \tag{2}$$

where $\gamma_A^{S,0}$ and $\gamma_B^{S,0}$ are the surface energies of metals A and B, contributing a positive term of elastic energy caused by size difference, while γ_{SS}^{chem} is the chemical contribution, which has the same sign as the heat of mixing (ΔH_{mix}^{sol}).

The schematic configuration of the Fe/Ag multilayer films is shown in Fig. 7. Suppose N_A and N_B are the numbers of layers of metals A and B, $d_{A,i}$ and $d_{B,i}$ are the thicknesses of the ith layers of metals A and B, respectively, n_A and n_B are the numbers of the interfaces (A on B) and (B on A), and $\Delta d_{A,i}$ and $\Delta d_{B,i}$ are the thicknesses of the ith interfaces (A on B) and (B on A), respectively. It is noted that the relationship $n_A + n_B + 1 = N_A + N_B$ always holds for these four parameters. α_A and α_B are the fractions of interfacial atoms A and B versus the total number of atoms in the multilayered films, respectively, and they are given by

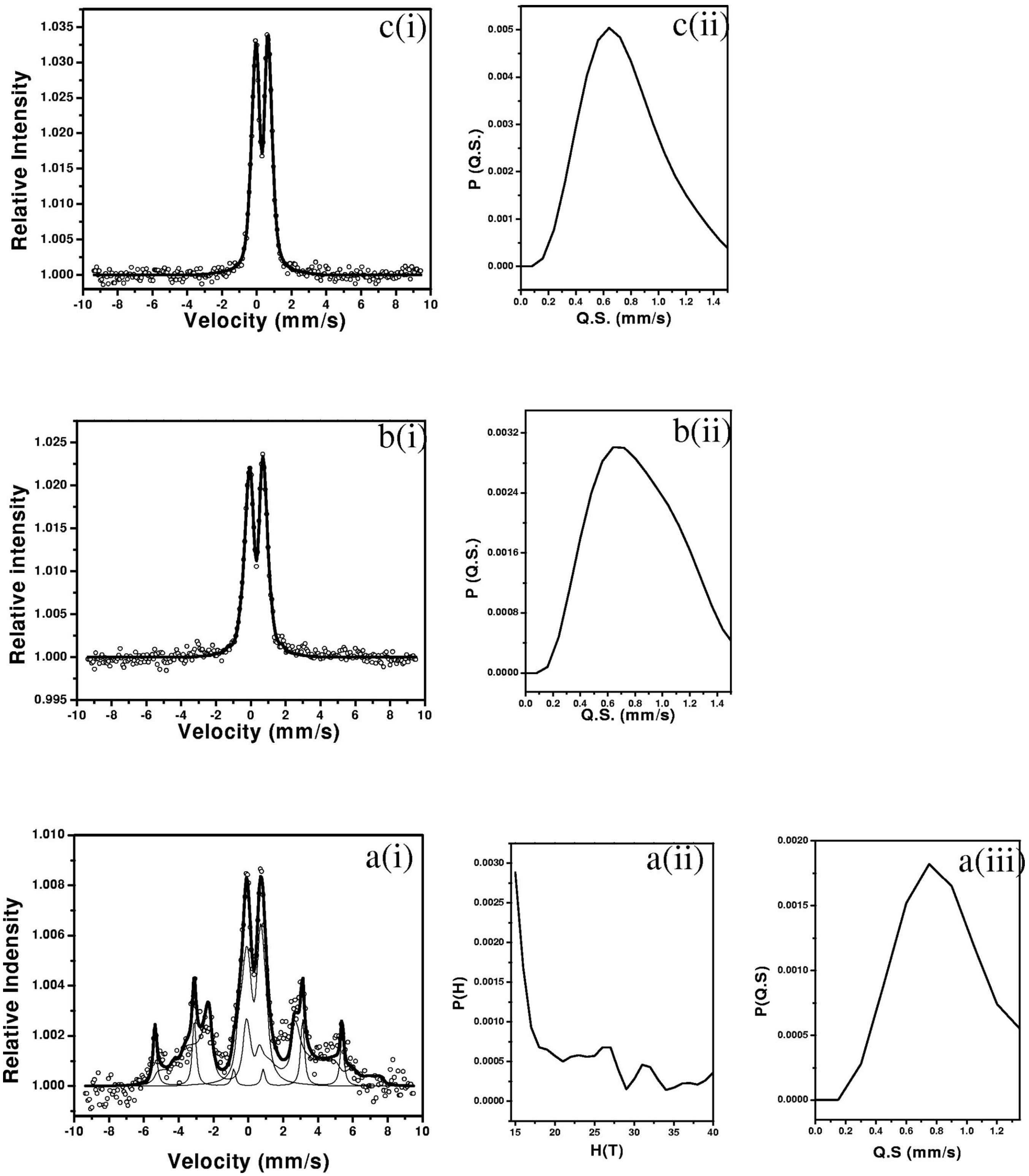

FIG. 6. The CEMS spectra of the sample irradiated at 110 K up to a fluence of (a) 5×10^{14} ions/cm^2, (b) 1×10^{15} ions/cm^2, and (c) 3×10^{15} ions/cm^2. The curves [a(ii)] and [a(iii)] represent the best fit to the experimental data obtained from the hyperfine field distributions corresponding to the interfacial region and the quadrupole splitting distributions showing evidence for mixing. Additionally, a sharp sextet corresponding to bulk α-Fe is also fitted. The continuous curves [b(ii)] and [c(ii)] are the distributions of quadrupole splittings, confirming the complete mixing of Fe/Ag multilayers.

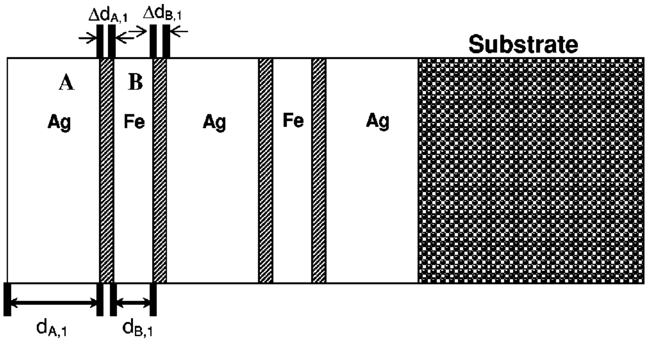

FIG. 7. Schematic configuration of the A-B multilayered films. $d_{A,1}$ and $d_{B,2}$ are the thicknesses of the first layers of A and B, respectively, and $\Delta d_{A,1}$ and $\Delta d_{B,1}$ are the thicknesses of the first interfaces (A on B) and (B on A), respectively.

$$\alpha_A = X_A \frac{\Sigma_i^{n_A} \Delta d_{A,i}}{\Sigma_i^{N_A} d_{A,i}}, \quad \alpha_B = X_B \frac{\Sigma_i^{n_B} \Delta d_{B,i}}{\Sigma_i^{N_B} d_{B,i}},$$

where $\Sigma_i^{n_A} \Delta d_{A,i} / \Sigma_i^{N_A} d_{A,i}$ and $\Sigma_i^{n_B} \Delta d_{B,i} / \Sigma_i^{N_B} d_{B,i}$ are the fractions of interfacial A atoms versus the total number of A atoms and of interfacial B atoms versus the total number of B atoms respectively. These atoms, multiplied by X_A and X_B, respectively, give the fractions of A and B interfacial atoms versus the total number of $(A+B)$ atoms in the multilayers. X_A and X_B are the concentrations of A and B of the whole film. In our case, A is Ag and B is Fe and for simplicity of calculation the interfacial layers of Ag on Fe and Fe on Ag are assumed to be the same. We have taken the interfacial layer thickness as the interdiffused layer thickness, which was estimated to be 1.3 nm from the Mössbauer spectrum of the as-prepared sample. The calculated interfacial free energy of the Fe/Ag system is 35 kJ/mole, which is higher than the heat of mixing for a random mixture of $Fe_{30}Ag_{70}$, which is 25 kJ/mole.[23] Hence, a metastable $Fe_{30}Ag_{70}$ random alloy was formed at 110 K by ion-beam mixing assisted with the interfacial free energy stored in the initial multilayers and aided by the reduction of atomic migration length required for intermixing and alloying due to the nanosized Fe layer thickness.

However, on irradiation at 325 K, thermally activated long-range defect migration becomes possible,[6] which provides a picture of competing dynamics, where mixing by irradiation is in competition with thermal decomposition by radiation enhanced diffusion. According to the empirical formula proposed by Rossi *et al.*[24] a linear dependence exists between the critical temperature (T_c) for the onset of radiation enhanced diffusion and the average cohesive energy (E_{coh}). Using this empirical relation $T_c = 96.5 E_{\mathrm{coh}}$ (eV/atom), we obtain a value of $T_c = 349$ K for the Fe/Ag system. Since T_c is close to the temperature of irradiation of 325 K, we expect mobility due to radiation enhanced diffusion, which leads to the clustering of Fe particles, and the mixing is inhomogeneous at this temperature compared to that at 110 K.

It is concluded that the complete mixing of Fe/Ag multilayers as observed in the present study is due to the presence of an increase in interfacial free energy stored in the interfaces of the multilayer and to the reduction of atomic migration length required for intermixing and alloying. This is very different from the results observed in Fe/Ag bilayers, which do not mix at any temperature.

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