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Surfactant controlled interfacial alloying in thermally evaporated Cu/Co multilayers

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ABSTRACT

The effect of Ag surfactant on interfacial alloying in nm-range Cu/Co thin film multilayer structures is reported in this work. The multilayer were prepared by electron beam evaporation method with or without using Ag as a surfactant. When multilayers are annealed, the interdiffusion process get suppressed when Ag surfactant is introduced prior to deposition of the multilayer structures. In absence of a surfactant, the asymmetry in the free energy results in formation of a metastable CuCo-alloys. The Ag surfactant balances this difference in the surface free energy, resulting in reduced interdiffusion. The obtained results directly evidence the role of the Ag surfactant in balancing the asymmetry in the surface free energy of Cu and Co in Cu/Co multilayers and thereby controlling interfacial alloying.

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1. Introduction

It is well-known that Cu–Co is equilibrium immiscible system with positive heat of formation of +10 kJ/mol and therefore does not form any stable intermetallic compounds [1]. However, when thin layers of Cu and Co are staked together in the form of thin film multilayers, their interfacial alloys may be formed. This happens due to a large difference between the surface free energy (γ) of Cu and Co. It is reported in the literature that metastable structure of a mixed CuCo phase may exist at the interfacial regions in Cu/Co thin film multilayers [1–3]. In case of Cu/Co multilayers, growth of Co-on-Cu is thermodynamically unfavorable because $\gamma_{Co} > \gamma_{Cu}$ [4]. In an earlier study it was found that Co make islands over Cu while Cu makes a smooth layer when deposited on Co [5]. The experimentally observed values of γ for the average face (polycrystalline) of Co and Cu are 2.55 Jm^{-2} and 1.8 Jm^{-2} respectively [6,7]. The experimental and theoretical studies in Cu/Co multilayers suggest that the surfactant atoms affect the growth of the multilayers [8–10]. If the surfactant atoms balance the γ of Cu and Co, their intermixed metastable phases should not be formed. However this has not been evidenced experimentally. In the present work we observed that by adding the Ag surfactant, the interface roughness decreases and more importantly the formation of intermixed CuCo phases can be avoided completely. This gives a direct evidence that Ag surfactant indeed results in balancing the surface free energy in Cu/Co multilayers.

In a multilayer structure two or more different elements are forced to get deposited on top of each other. In this condition an inherent asymmetry in their surface free energy will result in an asymmetry of the interfaces in terms of its topological distribution in the interfacial regions [11,12]. An element with low γ will wet the element with high γ. On the other hand, when this situation is reversed, agglomeration of a high γ element will occur on a low γ element. This situation will lead to a rougher interface at each alternate layer and give rise to asymmetric interfaces. Such asymmetric interfaces are a primary source to many undesirable effects; an enhanced interdiffusion across the interfaces affects the performance of the multilayer structures most severely [13]. For example, interdiffusion at the interfaces affects the magnetic properties of the multilayers. In particularly, the magnetic coupling may get affected in magnetic multilayers due to an enhancement in the interfacial width [14,15].

It has been demonstrated in the literature that by using surfactants, the problem of asymmetric interfaces can be reduced to a large extent [16–22]. The prerequisites for a material to qualify as a surfactant is its optimal quantity and a low γ as compared to other elements present in the multilayer. The low γ of a surfactant helps in complete wetting of a high γ surface and its floating-off ability ensures that incoming atoms essentially interact with the γ of the surfactant. The experimental and theoretical studies have shown that surfactant atom floats over the surface by an atomic exchange process leaving behind the smooth and symmetric interfaces [17,18,23,24]. Essentially, surfactant reduces the surface

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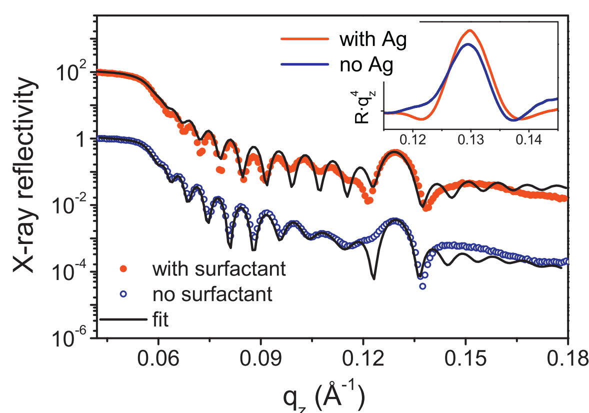

Fig. 1. X-ray reflectivity of Cu/Co multilayers deposited without surfactant and with Ag surfactant. Inset shows the Bragg peak intensity at Bragg peak. The pattern on y-axis have been shifted for clarity.

tension thereby enhances the wetting capability of one element over the other. Surfactants have been commonly used in chemical solutions and crystal growth process. Introduction of small impurity as surfactant in thin films induces layer-by-layer type growth and has been observed in a variety of systems [25–32].

2. Experimental

In this work we used electron (e)-beam evaporation technique to deposit Cu/Co multilayers with and without the Ag surfactant at room temperature. A Cu buffer layer of thickness 10 nm was deposited on a Si (1 0 0) substrate before the deposition of multilayer structure to minimize the substrate effects. The thickness of the growing film was measured *insitu* using a quartz crystal thickness monitor installed in the vicinity of substrates. The deposition rates achieved were about 1.2 nm/min, for Cu, Co and Ag. The substrates were placed at a distance of about 600 mm from the e-beam source to ensure uniformity of the samples. The base pressure in the chamber prior to deposition was about 5×10^{-10} mbar and during the deposition it was typically 5×10^{-8} mbar. The nominal structure of the multilayer is Cu (10 nm)/x/[Cu (3 nm)/Co (2 nm)]$_{10}$, with x = 0 or 0.2 nm Ag. The deposition of the samples with or without surfactant was carried out together.

The X-ray reflectivity (XRR) measurements were carried out using X-rays of wavelength 1.54 Ågenerated using Cu-Kα source (Bruker D8 Discover) equipped with a Göbel mirror. The X-ray diffraction (XRD) measurements were carried out using 1.54 ÅX-rays in the θ-2θ geometry using a standard diffractometer (Bruker D8 Advance) equipped with a fast 1D detector based on silicon drift technology (Bruker LynxEye). The hysteresis loops were recorded using a magneto-optic Kerr effect (MOKE) system operating in the longitudinal mode. The magnetic field was always applied in the plane of the sample and the measurements were carried out at room temperature.

3. Results and discussion

3.1. X-ray reflectivity

The X-ray reflectivity measurements were carried out in the as-deposited samples and after annealing them at different temperatures. The XRR measures the layer thickness and rms interface roughness of a thin film sample in a non destructive way. In case of a multilayer, a Bragg peak appears in the XRR pattern due to constructive interference of X-rays reflected from the repeated interfaces. The position of Bragg peak is defined in terms of momentum transfer vector, $q_z = 4\pi\sin\theta/\lambda$, with λ being the wavelength of the X-rays and θ, the angle of X-rays with respect to the surface of the sample. The reflectivity from rough interfaces result in a loss of intensity at a Bragg peak.

The XRR pattern of the Cu/Co multilayer samples prepared with and without Ag surfactant are shown in Fig. 1. As can be seen there, a Bragg peak corresponding to the periodicity of the multilayer appears around $q_z = 0.13$ Å^{-1}. The obtained value of the bilayer thickness is 5.3 nm which is close to the nominal value of 5 nm. A comparison of the reflected intensity at the Bragg peak shows an enhanced reflectivity for the sample prepared using the Ag surfactant. The inset of Fig. 1 compares the intensity at the Bragg peak (here the decay due to Fresnel reflectivity is removed by multiplying the reflectivity data by q_z^4 for a better comparison). This indicates that by adding Ag surfactant the interface roughness decreases. The fitting of XRR pattern was done using Parratt's formulism [33,34], and the fitted parameters are given in Table 1. As can be seen from the table the interface roughness of Co-on-Cu interface is about 1.6(±0.1) nm while that of Cu-on-Co interface is 1(±0.1) nm in the sample where no surfactant was used. However, the roughnesses of these interfaces becomes almost equal at about 1 nm when the Ag surfactant is added. As expected Co-on-Cu interface is rougher due to agglomeration of Co atoms on the Cu layer as $\gamma_{Co} > \gamma_{Cu}$. By adding Ag surfactant the surface free energies are balanced making the roughness of both interfaces almost equal. It may be noted that the values of overall roughness in the present case are significantly higher as compared to samples prepared using ion beam sputtering technique [10]. The basic difference between these deposition techniques can be envisaged by the energy of atoms (adatoms) reaching to substrates which is orders of magnitude higher in sputtering as compared to e-beam evaporation. With lower adatom energy it is expected that growth will be more island type as adatoms do not have sufficient energy to relocate after condensation at a substrate.

Table 1
Parameters obtained from the fitting of X-ray reflectivity data, bilayer period (ℓ) and roughness (σ) of the as-deposited and annealed samples prepared with and without Ag surfactant.

Sample State	Parameter	No surfactant (nm)	Ag surfactant (nm)
As-deposited	ℓ	5.34±0.05	5.32±0.05
	$\sigma_{[Co-on-Cu]}$	1.6±0.1	1.1±0.1
	$\sigma_{[Cu-on-Co]}$	1.0±0.1	1.0±0.1
473 K	ℓ	5.33±0.05	5.32±0.05
	$\sigma_{[Co-on-Cu]}$	1.7±0.1	1.2±0.1
	$\sigma_{[Cu-on-Co]}$	0.8±0.1	1.2±0.1
573 K	ℓ	5.63±0.05	5.34±0.05
	$\sigma_{[Co-on-Cu]}$	2.1±0.1	1.2±0.1
	$\sigma_{[Cu-on-Co]}$	1.4±0.1	1.2±0.1
673 K	ℓ	–	5.38±0.05
	$\sigma_{[Co-on-Cu]}$	–	1.3±0.1
	$\sigma_{[Cu-on-Co]}$	–	1.3±0.1

In order to study the thermal stability of the samples, vacuum annealing of the samples was done. The base pressure in the annealing chamber was typically 1×10^{-6} mbar or better. In order to ensure a similar environment, samples prepared with and without surfactant were always annealed together. Fig. 2 shows the XRR pattern of samples prepared with and without Ag surfactant in the as-deposited state and after annealing at different temperatures. The fitted parameters bilayer period (ℓ) and interface roughness (σ) are given in Table 1. The density of the layers correspond to their bulk values. As can be seen for the sample prepared without Ag, the intensity at the Bragg peak increases when annealed at 473 K, and decreases when annealed at higher temperatures. Whereas, no change in the peak intensity can be observed in case of the sample prepared using the Ag surfactant. First, an increase in the peak intensity for the sample prepared without surfactant indicates that some sort of relaxation may be responsible for such behavior. As mentioned above the roughness of the Co-on-Cu interface is significantly larger than Cu-on-Co interface, in this condition the intrinsic stress may built up as the growth of the multilayer takes place. After annealing, this stress may get relieved giving rise to a higher intensity at the Bragg peak position. Such an increase in the intensity at moderate annealing temperatures has also been observed in other multilayer structures [35–37]. The fitting of XRR pattern also indicate a reduction in the interface roughness of the Cu-on-Co interface. However, this stress may not get completely relieved.

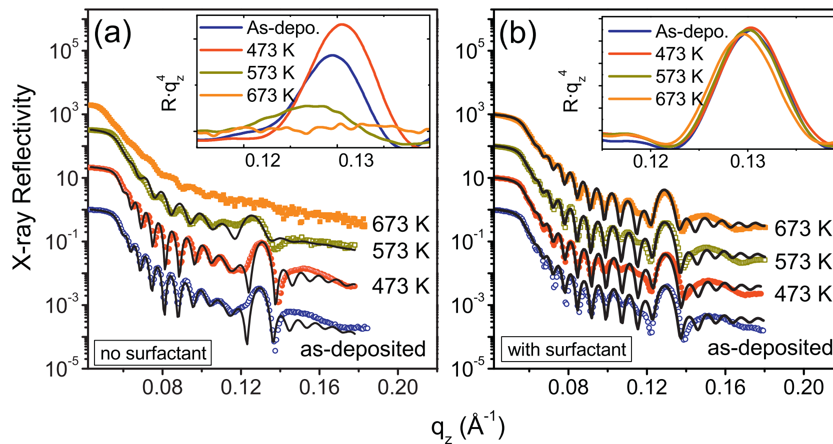

Fig. 2. XRR pattern of samples prepared without surfactant (a) and with Ag surfactant (b). Each scan is offset on *y*-scale for clarity. Scattered points are measured data and solid lines are fit to them. The inset compares the normalized reflectivities after multiplying with q_z^4.

Because if the stress gets completely relieved no further change is expected in the XRR pattern when the samples are annealed at higher temperatures. As can be seen from Fig. 2(a), after annealing at 573 K, the intensity at the Bragg peak decays considerably and at 673 K the Bragg peak vanishes completely.

On the other hand, the sample prepared using Ag surfactant, the intensity at the Bragg peak does not decrease at all up to annealing temperature of 573 K. Though at 673 K, there is marginal decrease in the Bragg peak intensity. Taking the 473 K data as a reference for the sample where no surfactant was used, the interdiffusion length may be quantified from the decay of the Bragg peak intensity using the expression [38–40]:

$$I(t) = I(0)\exp\left(-\frac{8\pi^2 D}{\ell^2}t\right), \quad (1)$$

where $I(0)$ is the intensity before annealing and $I(t)$ is the intensity after annealing time t at temperature T, ℓ is the bilayer periodicity. The diffusion coefficient (D) obtained using Eq. (1) and the inter diffusion length are given by $L_d^2 = 2Dt$ [41]. The values of D and L_d for the sample prepared without Ag surfactant at 573 K are $D = 9.61(\pm 0.11) \times 10^{-23}\ \mathrm{m^2 s^{-1}}$ and $L_d = 0.83(\pm 0.04)$ nm. Whereas in case of sample prepared with Ag surfactant there is no interdiffusion up to 573 K. However, at 673 K the intensity of the Bragg peak decays slightly and here the value of $D = 7.79(\pm 0.1) \times 10^{-24}\ \mathrm{m^2 s^{-1}}$ and $L_d = 0.24(\pm 0.01)$ nm. From the fitting of X-ray reflectivity data of annealed samples, the obtained values of interdiffusion length were similar.

3.2. X-ray diffraction

Figure 3 shows the XRD pattern of the samples in the as-deposited state and after annealing at temperatures of 473 K, 573 K and 673 K. For both samples (prepared with and without Ag surfactant) the XRD pattern up to a temperature of 473 K remain similar to that observed for the as-deposited samples. However, after annealing at 573 K the sample prepared without Ag surfactant shows new peaks around $2\theta = 35.5°$ and $45.3°$. In order to further confirm these peaks, we repeated our measurements and also annealed samples at a lower temperature of 523 K. Here also a peak around $2\theta = 45.3°$ can be seen. As observed from the XRR measurements, at 573 K, significant interdiffusion takes place in the sample prepared without Ag surfactant. It may be noted that Cu and Co is an immiscible system and therefore no CuCo alloys are formed in bulk. However, in case of Cu/Co thin films intermixed metastable CuCo phases are reported in the literature [3]. Basically these phases are face centered cubic (fcc) structures with lattice parameter $a = 0.40$ nm (for fcc-1 phase) and $a = 0.34$ nm (for fcc-2 phase) for (1 1 1) plane [3]. In our case the values of lattice constant obtained for fcc (1 1 1) plane are 0.435(4) nm and 0.344(4) nm, for fcc-1 and fcc-2 phases, respectively. The obtained values match very well with the reported values. This indicates that two fcc phases indicated as CuCo-1 and

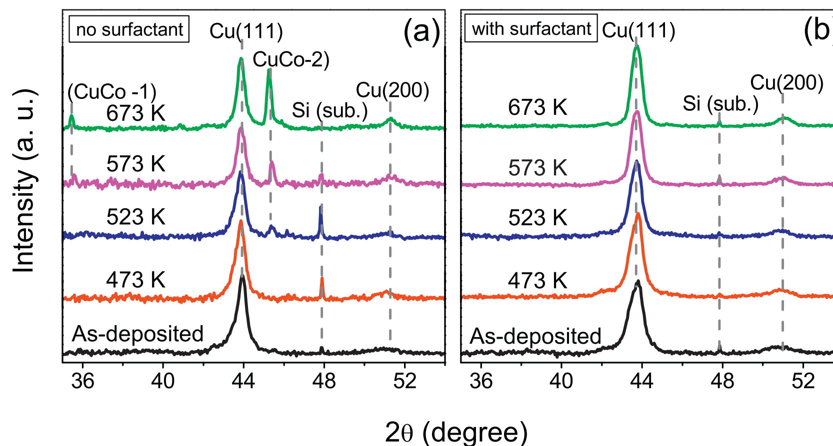

Fig. 3. XRD pattern of Cu/Co multilayers prepared without surfactant (a) and using Ag surfactant (b) in the as-deposited state and after annealing at different temperatures.

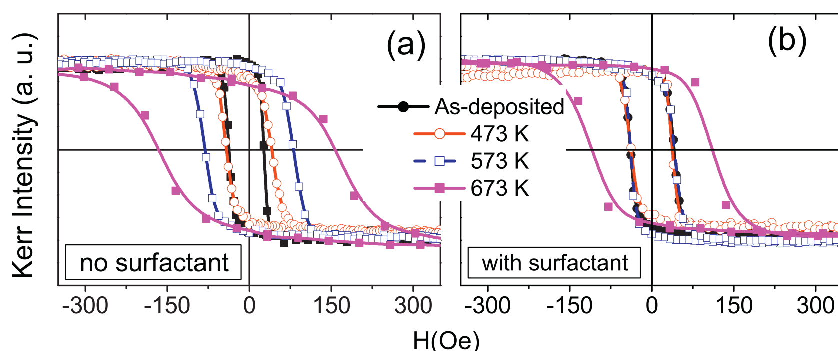

Fig. 4. MOKE signal of Cu/Co multilayers prepared without surfactant (a) and using Ag surfactant (b) in the as-deposited state and after annealing at different temperatures. The scattered points are measured data and solid lines are guide to eye.

CuCo-2 in Fig. 3(a) are obtained when the multilayers are prepared without Ag surfactant and annealed above 523 K. However the XRD results in the samples prepared using the Ag surfactant are completely different. As can be seen from Fig. 3(b) there is no additional peak appearing in the XRD pattern of the Cu/Co multilayers prepared using the Ag surfactant even after when annealed at 673 K. This shows that by adding the Ag surfactant in the Cu/Co multilayers, intermixed CuCo phases do not grow. The XRR results of the sample prepared with Ag surfactant also confirm the observed XRD results as no interdiffusion takes place in the samples prepared using the Ag surfactant.

3.3. MOKE measurements

Fig. 4 shows the MOKE results of both samples. As can be seen from the figure the coercivity of the as-deposited and that of annealed at 473 K, are almost equal at about 40 Oe for both samples. This result can be understood with the XRR and XRD results where there is no change in the structure of the multilayer at 473 K. At the higher annealing temperatures of 573 K and 673 K, the coercivity of the sample prepared without surfactant increases to 80 Oe and 165 Oe, respectively. On the other hand there is no increase for the sample deposited using the Ag surfactant at 573 K, though at 673 K it increases to 110 Oe. An increased coercivity is indeed undesirable from the applications point of view as high sensitivity read head based devices require small coercivity (magnetically soft material) for switching the field. The observed MOKE results can be well correlated to the XRR and XRD results, and can be understood in terms of increased interdiffusion in the sample prepared without surfactant. As interdiffusion occurs due to thermal annealing, Co and Cu atoms get intermixed. This results in isolation of Co atoms and magnetoelastic coupling decreases between them which results in an increased value of coercivity as observed in the literature [42].

The Cu–Co system is immiscible in equilibrium. It has a positive heat of formation of +10 KJ/mol and therefore does not form any intermetallic compounds. Formation of intermixed metastable phases and associated structural transition that occur in Cu–Co thin films are due to the difference in the surface free energy of Cu and Co [1–3]. In case of nm range thin films the volume fraction of surface is very high and in this situation the asymmetry in the surface free energy may be the driving force for formation of intermixed phases. Schmid et al. show that when a Co thin film, grown on a Cu substrate, is annealed, a layer of Cu comes over the Co surface [43]. The mechanism of such diffusion in thin films has been explained in terms of the difference in the surface free energy [17,18,23,25]. By adding a surfactant the difference in the surface free energy of Co and Cu is expected to be minimized. Our XRR results clearly show a symmetric Cu-on-Co and Co-on-Cu interface with addition of the Ag surfactant. The high temperature annealing of the samples show almost no interdiffusion when Ag surfactant was added. This confirms that the difference in the surface free energy should get balanced by adding Ag surfactant. If this is the case no intermixed phase is expected as observed from the present XRD measurements.

4. Conclusion

We observed that addition of Ag surfactant in Cu/Co multilayers results in the smoother and symmetric interfaces. Thermal stability measurements show that the addition of the Ag surfactant inhibits the interfacial alloying across the interfaces in Cu/Co multilayers. When no surfactant was used, metastable CuCo phases grow at the interfaces due to interdiffusion caused by a difference in the surface free energy of Cu and Co. The surfactant atoms balance the difference in the surface free energy, resulting in suppression of interdiffusion. The MOKE measurement demonstrates that the Ag surfactant helps in minimizing the coercivity in Cu/Cu multilayers.

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