Surfactant induced symmetric and thermally stable interfaces in Cu/Co multilayers

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Received 31 July 2011, in final form 6 October 2011
Published 3 November 2011
Online at stacks.iop.org/JPhysCM/23/485003

Abstract
In this work we studied Ag surfactant induced growth of Cu/Co multilayers. The Cu/Co multilayers were deposited using Ag surfactant by the ion beam sputtering technique. It was found that Ag surfactant balances the asymmetry between the surface free energies of Cu and Co. As a result, the Co-on-Cu and Cu-on-Co interfaces become sharp and symmetric and thereby improve the thermal stability of the multilayer. On the basis of obtained results, a mechanism leading to symmetric and stable interfaces in Cu/Co multilayers is discussed.

1. Introduction

Control over the interface roughness in a thin film multilayer is an important issue. For example, in a multilayer structure stacked as A/B repeats, the inherent difference in the surface free energy ($\gamma$) of A and B leads to alternate smooth and rough interfaces. If A has larger surface free energy than B, then B will wet the surface of A, while A will make islands over the surface of B [1]. Therefore asymmetric interfaces occur in multilayers due to the difference in the $\gamma$ values of the elements [2]. In particular, in magnetic multilayers, smooth and symmetric interfaces are of much interest because of their technological applications [3–6]. The magnetic layers separated by a nonmagnetic spacer layer show the giant magnetoresistance (GMR) due to oscillatory exchange coupling between the magnetic layers as the thickness of the spacer layer is varied [7–10]. Thickness fluctuation of the spacer layer due to asymmetric interface roughness may affect the strength of exchange coupling [11] and in this condition the spin dependent scattering may be modified [12–14].

In the Cu/Co multilayer system the $\gamma$ values for Cu and Co are different. Although $\gamma$ depends on the crystallographic orientations, in case of polycrystalline structures, the average value of $\gamma$ is relevant. The experimentally observed values of $\gamma$ for polycrystalline Cu, Co and Ag are 1.8 J m$^{-2}$, 2.55 J m$^{-2}$ and 1.24 J m$^{-2}$, respectively [15, 16]. This difference in $\gamma$ leads to wetting of Cu-on-Co and de-wetting of Co-on-Cu, giving rise to a smooth Cu-on-Co and a rough Co-on-Cu interface. This was experimentally evidenced by Timothy et al via scanning tunnelling microscopy, demonstrating that Co makes islands over Cu, while Cu makes a smooth layer on Co in Cu/Co multilayers [17].

It has been demonstrated in the literature that by using a surface active species or so called surfactant the difference between the surface free energies can be minimized. Use of such surfactants in the crystal growth technology is well established [18]. However, in the case of thin films deposited in vacuum, surfactants have been used only in a few multilayer systems [1, 19–27]. Egelhoff and Steigerwald [2] studied the role of adsorbed gases (H, O, N, Co and S) in deposition of metal-on-metal epitaxial systems. These adsorbed gases float or segregate to the surface, balancing the surface/interface energy and strain during the growth. Gaseous surfactants such as oxygen also suppress the intermixing and increase the GMR by restricting pinholes in the Cu spacer layer [28–30].
However, due to enhanced mobility of gaseous surfactants, it is likely that they get trapped across the grain boundaries.

In the case of Ge/Si(100) multilayers, Copel et al have demonstrated that use of As surfactant triggers the layer-by-layer type growth and inhibits interdiffusion [1]. Hoegen et al have shown that Sb surfactant not only inhibits interdiffusion but results in a relaxed (strain free) and defect free Ge film on Si(111) [31]. In a theoretical study by Barabási the interaction of the surface with a surfactant was described [32]. In another theoretical work by Zhang and Lagally [33] the surfactant mediated layer-by-layer growth was described on the basis of atomic interactions. More recently, Egelhoff and co-workers have explored the effect of surfactants (e.g. In, Ag, O, Pb etc) in Cu based spin valve systems and demonstrated that surfactants improve the surface and interface properties as well as increasing the GMR value [19, 34, 35]. In another study, Camarero et al have demonstrated that Pb atoms used as surfactant suppress the twin formation, which increases the coupling between Co layers [23]. Theoretical studies also show that a monolayer of Pb used as a surfactant in a Cu/Co multilayer minimizes the difference in surface free energy of Cu and Co, inhibits the island formation and floats over the surface by an atomic exchange process [36, 37]. The use of Ag surfactant was also studied to examine the interfacial intermixing [38–40] and GMR in magnetic multilayers [41]. In the case of Ti/Ni multilayers, Ag surfactant was used to get smooth and symmetric interfaces [42].

However, detailed studies on the Ag surfactant mediated growth in Cu/Co multilayers are still lacking. In the present work, we studied the effect of Ag surfactant in a Cu/Co multilayer. It was also investigated how this addition of Ag surfactant affects the structural and magnetic properties of Cu/Co multilayers. We used the neutron reflectivity (NR) technique, which is nondestructive and measures the interface roughness with an accuracy of less than an angstrom length. It was found that addition of Ag surfactant makes Cu-on-Co and Co-on-Cu interfaces, which are otherwise rough and asymmetric, smooth and symmetric. This leads to reduced interdiffusion and thereby improved thermal stability of Cu/Co multilayers prepared using Ag surfactant. The obtained results are presented and discussed in the following sections.

2. Experimental details

The ion beam sputtering (IBS) technique was used to deposit the Cu/Co multilayers with and without Ag surfactant. The Ar$^+$ ions of energy 1.00 keV were produced using a radio-frequency (RF) ion beam source (Veeco 3cm RF source). The ion beam was neutralized using an RF generated electron flood source. The ion beam of size 3 cm was kept incident at an angle of 45° with respect to a target. The targets were mounted on a rotary motion feedthrough, which can hold up to four different targets. The targets were sputtered alternately to deposit multilayer structures. The samples were prepared without any surfactant as a reference (sample A) and with Ag surfactant (sample B) added on top of a Cu buffer layer deposited on a Si(100) substrate at room temperature (without intentional heating). The nominal structures of samples are given below:

(A) Cu (10 nm)/[Cu (3 nm)/Co (2 nm)]$^{10}$
(B) Cu (10 nm)/Ag (0.2 nm)/[Cu (3 nm)/Co (2 nm)]$^{10}$.

Here, the Cu layer thickness of 3 nm corresponds to the third (and weakest) AF peak in the oscillatory exchange coupling. At this thickness it is expected that magnetoresistance (MR) will be small compared to the first or second maximum for Cu layer thickness of 1 nm or 2 nm, respectively [9]. The Cu layer thickness of 3 nm was chosen as at lower thicknesses the Bragg peak in the neutron reflectivity (NR) will appear at high $q_z$ values, and due to limited flux of neutron sources it may be difficult to make NR measurements in a reasonable time.

Prior to the deposition of samples the base pressure in the deposition chamber was about 2 $\times$ 10$^{-8}$ mbar and during deposition the pressure was about 5 $\times$ 10$^{-4}$ mbar due to flow of Ar gas (purity 99.9995%) in the source and neutralizer. The deposition rates achieved were 5.8 nm min$^{-1}$, 3.2 nm min$^{-1}$ and 7.4 nm min$^{-1}$ for Cu, Co and Ag, respectively. X-ray and neutron reflectivity measurements were carried out to measure the bilayer thickness and interface roughness of the samples. X-ray reflectivity (XRR) measurements were carried out using x-rays of wavelength 1.54 Å generated using a laboratory source. The NR measurements were carried out at the SuperADAM instrument at ILL, Grenoble, France, using neutrons of wavelength 4.4 Å. X-ray diffraction (XRD) measurements were carried out with 1.54 Å x-rays in the $\theta$–2$\theta$ geometry using a standard diffractometer (Bruker D8 Advance) equipped with a fast 1D detector based on silicon drift technology (Bruker LynxEye).

Longitudinal magneto-optical Kerr effect (MOKE) hysteresis loops were measured under the field always directed in the plane of the sample. The magnetoresistance (MR) measurements for all the samples were carried out at room temperature using the four point probe method. The direction of the current flowing in the sample was along the direction of the magnetic field (parallel to the surface of the sample). Here, the MR is defined as

$$MR = \frac{R_0 - R_{sat}}{R_0}$$

where $R_0$ is the resistance in the absence of magnetic field and $R_{sat}$ is the resistance under the magnetic field in which the samples are magnetically saturated.

3. Results

3.1. X-ray and neutron reflectivity measurement

Figure 1 shows the XRR pattern of the as-deposited samples A and B. The XRR pattern shows a Bragg peak around $q_z = 0.14$ Å$^{-1}$, corresponding to the bilayer period of 4.9 nm, which is close to the nominal value of 5 nm. As can be seen here the intensity at the Bragg peak is greater for sample B, indicating that the interface roughness may be smaller with addition of Ag surfactant. Since the contrast between Cu and Co for the x-rays is rather poor, we performed neutron reflectivity (NR) measurements to get further insight. In the
Figure 1. X-ray reflectivity of Cu/Co multilayers deposited without surfactant (A) or with Ag surfactant (B). The patterns on the y-axis have been shifted for clarity. The inset shows the reflectivity at the Bragg peak.

In the case of neutrons the contrast between Cu and Co is much larger as compared to x-rays.

The NR patterns were recorded using a 2D position sensitive detector (PSD) and are shown in figure 2 as a function of \( p_i + p_f \) and \( p_i - p_f \) with \( p_i(0) = 2\pi \sin \theta_i(0)/\lambda \), normal to the surface component of the incoming (outgoing) wavevector, \( \theta_i(0) \) the angle of the incidence (scattering) and \( \lambda \) the wavelength of the neutrons [43]. As can be seen from the PSD images, the Bragg peak appears as a bright spot. The brightness of the Bragg reflected region is more intense for sample B as compared to sample A. This shows that with addition of Ag surfactant the reflectivity at the Bragg peak \( (R) \) is enhanced as also observed in XRR data.

A cut across the line for \( p_i = p_f \) gives the specular reflectivity which is plotted in figure 3(a) for samples A and B as a function of \( q_z \). \( q_z = p_i + p_f \). Here it may be noted that in the NR pattern we did not observe the magnetic Bragg peak which is expected due to antiferromagnetic (AF) coupling [44]. This may be due to the fact that the thickness of the Cu layer corresponds to the third AF peak, which is weakest in the oscillatory exchange coupling. Further, since we have only 10 repeats of Cu/Co bilayers, it is expected that the intensity of the AF peak will be too small to measure experimentally in this case. This is further confirmed by the MR measurements (shown later), where the typical values of MR are about 1% in the as-deposited samples. Also as discussed later, magnetic domains are distributed randomly in virgin samples, therefore misalignment of domains may also cause absence of the AF peak.

The NR measurements were always performed on virgin samples which were never exposed to external magnetic field. In order to fit the data we assume that all Cu-on-Co and Co-on-Cu interfaces are similar and the net magnetization of the sample is zero when no external magnetic field is applied. Under the saturation magnetic field, neutrons of spin up (down) are parallel (antiparallel) to the magnetic induction of the sample. In the absence of a magnetic field, the magnetic domains are expected to be randomly oriented and in this situation the total magnetic moment may be zero and as a result the magnetic scattering length density (SLD) is zero. However, if the size of magnetic domains is typically of the order of the neutron coherence length or more, two critical edges are expected in unpolarized neutron reflectivity \( (R) \) data. The coherence length of the neutrons in our case is of the order of 100 \( \mu m \). If the sizes of the magnetic domains are larger than the neutron coherence length, neutron reflectivity will be essentially an incoherent addition of spin up and spin down neutrons, giving rise to two critical angles as observed in the case of 100 nm thick polycrystalline iron films [45]. However, if the Co domain sizes are significantly smaller than the neutron coherence length, the magnetic contribution to the neutron scattering length will average out to zero. It is reported in the literature that in the case of Cu (2 nm)/Co (2 nm) multilayers the typical size of magnetic domains is about 1 \( \mu m \) [44, 46]. Therefore to fit the NR data we assume that the net magnetic moment of the sample is zero. We also compared polarized neutron reflectivity (PNR) measurements with unpolarized NR measurements to verify Co magnetization. Here we found that the average of spin
The parameters varied in the fitting of the data are bilayer thickness (\(d\)) and the interface roughness (\(\sigma\)) of the Cu-on-Co interface and the Co-on-Cu interface, respectively, when no surfactant was used. With addition of Ag surfactant, the interface roughnesses of the two interfaces increases from 0.23% to 0.36% when Ag surfactant is added. Therefore, the values of roughnesses may be taken as a lower limit.

Figure 3(b) shows the SLD profile of samples A and B, obtained from the fitting of NR data. For a rough interface the SLD profile will not be sharp and a fairly gradual change of SLD will occur at an interface. As can be seen from figure 3(b), the SLD profiles were asymmetric and broad when no surfactant was used. With addition of Ag surfactant the profiles become sharp and symmetric. This clearly shows that the addition of Ag surfactant in the Cu/Co multilayer results in an appreciable decrease of interface roughnesses and also the Co-on-Cu and Cu-on-Co interfaces become symmetric.

### Table 1

| Sample | No surfactant | Ag surfactant |
|--------|--------------|---------------|
| \(R_{\text{Bragg}}\) (%) | 0.23 ± 0.01 | 0.36 ± 0.01 |
| \(\ell\) (nm) | 4.9 ± 0.1 | 4.9 ± 0.1 |
| \(\sigma_{\text{Co-on-Cu}}\) (nm) | 0.36 ± 0.05 | 0.11 ± 0.05 |
| \(\sigma_{\text{Cu-on-Co}}\) (nm) | 0.18 ± 0.05 | 0.10 ± 0.05 |
| \(\sigma_{\text{substrate-film}}\) (nm) | 0.4 ± 0.2 | 0.4 ± 0.2 |

The thermal stability of the Cu/Co multilayer was studied by making XRR, XRD, MOKE and MR measurements in samples A and B after annealing them in a vacuum furnace (base pressure \(\sim 1 \times 10^{-6}\) mbar). The annealing of the samples was performed between 373 and 673 K with a step of 100 K for 1 h at each temperature. It was found that, up to a temperature of 473 K, the properties of samples remain almost the same as the as-deposited ones; however, above this temperature, the samples prepared using Ag surfactant were found to be more stable as compared to samples prepared without any surfactant. The results are presented in the following subsections.

### 3.2. Thermal stability of Cu/Co multilayer

#### 3.2.1. X-ray reflectivity measurements

The XRR patterns of samples A and B are shown in figure 4 for the as-deposited samples and after annealing them at 573 and 673 K. In sample B, after annealing at 573 K, \(R\) is at the level of the as-deposited state, and decreases marginally when annealed at 673 K. However, in the case of sample A there is a significant decrease in \(R\) even at 573 K, and when annealed at 673 K \(R\) reduces to background levels. This result clearly demonstrates that the thermal stability of the Cu/Co multilayer is improved with Ag surfactant. A reduction in \(R\) is basically due to
interdiffusion at the interfaces. When thermal annealing of the samples was performed, it was assumed that the topological roughness of the samples should not increase, and with an increase in the temperature intermixing at the interfaces should occur, therefore obtained intermixing (due to a decay in $R$) is labelled as interdiffusion length.

From the measured x-ray reflectivity data, the interdiffusion in both samples can be quantified and the decay in $R$ can be used to calculate the diffusion coefficient using the expression $I(t) = I(0) \exp(-8\pi^2 D t/\ell^2)$; here, $I(0)$ is the intensity before annealing and $I(t)$ is the intensity after annealing time $t$ at temperature $T$, $\ell$ is the bilayer periodicity. With known diffusion coefficient ($D$), the one-dimensional interdiffusion length ($L$) can be calculated with the expression $L^2 = 2Dt$ in the direction normal to the surface of samples [51]. The interdiffusion lengths obtained in this way are about 0.55 nm after annealing at 573 K for sample A. In the case of sample B, no measurable interdiffusion can be observed at 573 K. At a still higher annealing temperature of 673 K, the Bragg peak disappears completely in sample A whereas in sample B this peak remains intact. It is interesting to see that in sample A a new peak appears at $2\theta = 45.6^\circ$ (shown as a star in figure 5), which does not correspond to any known phases of Cu or Co. As CuCo is an immiscible system, this peak cannot be assigned to a known alloy of CuCo. In order to confirm our results, we repeated this experiment by preparing a new Cu/Co multilayer sample both with and without surfactant and observed the same results as shown in figure 5. It has been reported in the literature that an intermixed fcc-structured metastable phase of CuCo may form upon solid-state interfacial reaction after annealing at moderate temperature around 573 K [52].

From the interdiffusion measurements using XRR, we observed that the interdiffusion in the sample prepared without any surfactant was significantly enhanced as compared to the sample prepared using Ag surfactant. An enhanced interdiffusion may give rise to an intermixed CuCo phase as observed in our case. As pointed out by Li et al [52], the origin of the intermixed CuCo phase may be excess surface free energy. When a sample is prepared using Ag surfactant, the surface free energies of Cu and Co are balanced and in this situation the excess free energy for formation of an intermixed CuCo phase may not be available. Therefore our results demonstrate that by balancing the surface free energy using Ag surfactant the thermal stability of Cu/Co multilayers can be improved significantly.

3.2.2. X-ray diffraction measurements. The XRD patterns of Cu/Co multilayers prepared with and without Ag surfactant in the as-deposited state and after annealing at temperatures of 573 and 673 K are shown in figure 5. The XRD patterns of the as-deposited samples show a sharp peak around $2\theta = 43.6^\circ$ and a broader peak around $2\theta = 50.5^\circ$ corresponding to Cu(111) and Cu(200) reflections, respectively. The typical grain size obtained from the peak width of Cu(111) is about 15 nm. After annealing at 573 K, peaks shift towards the higher angle side in both samples A and B, which indicates a reduction in the inter-atomic distance. Further annealing at 673 K results in complete suppression of the Cu(111) peak for sample A, whereas in sample B this peak remains intact. It is interesting to see that in sample A a new peak appears at $2\theta = 45.6^\circ$ (shown as a star in figure 5), which does not correspond to any known phases of Cu or Co. As CuCo is an immiscible system, this peak cannot be assigned to a known alloy of CuCo. In order to confirm our results, we repeated this experiment by preparing a new Cu/Co multilayer sample both with and without surfactant and observed the same results as shown in figure 5. It has been reported in the literature that an intermixed fcc-structured metastable phase of CuCo may form upon solid-state interfacial reaction after annealing at moderate temperature around 573 K [52].

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3.2.3. MOKE and magnetoresistance measurements. Figure 6 shows MOKE hysteresis loops of samples A and B. The AF coupled region of the multilayer contributes to magnetoresistance (MR) and is quantified by the AF fraction
Figure 5. XRD pattern of Cu/Co multilayers prepared without surfactant (A) and using Ag surfactant (B) at different annealing temperatures.

Figure 6. MOKE hysteresis loop of sample A and sample B at different annealing temperatures.

(AFF) given by AFF = 1 − (M_r/M_s), with M_r and M_s being remanent and saturation magnetization, respectively [53–55]. The AFF for sample A is about 7%, and in sample B it increases to a value of about 12%. After annealing at 573 K, these values become about 14% and 20% for sample A and sample B, respectively. In sample A no MOKE signal could be detected after annealing at 673 K, which may be due to complete intermixing of Cu and Co layers as seen from our results. However, in sample B, the AFF after annealing at 673 K is about 21%.

Figure 7 shows the obtained MR of samples prepared without surfactant (figure 7(A)) and with Ag surfactant (figure 7(B)). In the case of sample A the value of MR increases after annealing at 573 K and is suppressed completely at 673 K, whereas in the case of sample B the value of MR increases at both 573 K and 673 K. The values of resistivity at room temperature (zero field) of samples A and B are 9.4 Ω cm and 10.2 Ω cm, respectively. Under the saturation magnetic field these values for samples A and B become 9.3 Ω cm and 10.07 Ω cm, respectively. The values of resistivity for samples A and B are almost the same and the observed MR is due to the change in resistivity when magnetic field is applied.

The resulting MR is basically due to competitive spin dependent scattering (SDS) and AF coupling processes. The contributions of SDS and AF coupling can be separated by normalizing MR to AFF [56, 57]. Figure 8 shows a variation of MR/AFF at different annealing temperatures. The data shown at 300 K correspond to as-deposited samples. For sample A, MR/AFF is higher as compared to sample B. A higher value of MR/AFF indicates that the contribution of
Figure 7. MR of sample A and sample B at different annealing temperatures.

Figure 8. Magnetoresistance normalized to the antiferromagnetic coupling fraction for the multilayer prepared without surfactant (□) and with Ag surfactant (■) at different annealing temperatures. The solid lines are a guide to the eye.

SDS is larger in sample A, as compared to sample B. This may be due to higher interface roughness in sample A.

Within samples A or B, there is no change in MR/AFF up to an annealing temperature of 573 K. However, it increases in sample B after annealing at 673 K, whereas it could not be measured for sample A. Here, it may be noted that in sample B the AFF only increases marginally (from 20% to 21%) when the annealing temperature is increased from 573 to 673 K. From XRR measurements we observed that some interdiffusion takes place in sample B at 673 K. In this condition, the SDS may be enhanced, giving rise to an increase in MR/AFF.

4. Discussions

From the results presented in section 3, the effect of Ag surfactant in the Cu/Co multilayer may be summarized as (i) reduction of interface roughness leading to symmetric Co-on-Cu and Cu-on-Co interfaces, (ii) suppression of interdiffusion and (iii) improvement in the thermal stability of the multilayer.

As mentioned before, $\gamma_{\text{Cu}}$ at 1.8 J m$^{-2}$ is significantly smaller compared to $\gamma_{\text{Co}}$ at 2.55 J m$^{-2}$. This difference in the surface free energy leads to asymmetric Co-on-Cu and Cu-on-Co interfaces. When Cu is deposited on Co, it will wet the surface of Co as the surface free energy of Cu is smaller. On the other hand, when Co is deposited on Cu, it will de-wet or agglomerate on Cu. This situation will lead to a sharper Cu-on-Co interface as compared to the Co-on-Cu interface. Ag surfactant with $\gamma_{\text{Ag}} = 1.24$ J m$^{-2}$ will help in removing the asymmetry due to its very low surface free energy as compared with Cu and Co. In this condition, when the growth of the multilayers takes place, the upcoming atoms basically see the lower surface free energy of surfactant atoms instead of the other element of the multilayer. This leads to wetting of the upcoming layer. If the surfactant atoms float off to the surface, the deposition of the next element will also see the surface free energy of the surfactant. In this situation the layer-by-layer type growth is induced, resulting in a smooth and symmetric interface in a multilayer [1, 32].

In our case the obtained results indicate that addition of Ag surfactant is altering the growth mode of the Cu/Co multilayer. The surfactant atoms placed once on the Cu buffer layer balance the surface free energy of Cu and Co, resulting in symmetric Cu-on-Co and Co-on-Cu interfaces. As expected, in the absence of Ag surfactant, the interface roughness of the Co-on-Cu interface is larger as compared to the Cu-on-Co interface. An asymmetry in the interface...
roughness may result in a strained interfacial region [31], which will act like nucleation centres when the multilayer is annealed at higher temperatures. This will result in enhanced interdiffusion as observed in the case when Ag surfactant was not used. On the other hand, since the addition of Ag surfactant removes the asymmetry of the interface roughness, such strained regions may be minimized to a large extent, resulting in stable interfaces. The reduction of interdiffusion length may be therefore understood in terms of smooth and sharp interfaces formed by the addition of Ag surfactant. The XRD results obtained in this work also support this argument, as in the absence of Ag surfactant an intermixed CuCo structure [52] is formed, while no such structure can be observed when Ag surfactant is added.

5. Conclusions

In the present study we observe that the addition of Ag surfactant to Cu/Co multilayers results in increased (x-ray and neutron) reflectivity at the Bragg peak due to smoother interfaces. The depth profile of the multilayer becomes symmetric with addition of Ag surfactant as it balances the asymmetry between the surface free energy of Cu and Co. The thermal stability measurements of Cu/Cu multilayers prepared without surfactant indicate formation of an intermixed CuCo structure. With addition of Ag surfactant to Cu/Co multilayers, the thermal stability of multilayers improves due to a suppression of interdiffusion.

Acknowledgments

We acknowledge DST, Government of India, for providing financial support to carry out NR experiments under its scheme ‘Utilization of international synchrotron radiation and neutron scattering facilities’. A part of this work was performed under the Indo-Swiss Joint Research Programme with grant no INT/SWISS/JUAF(9)/2009. We are thankful to Dr P Chaddah for continuous support and encouragement and acknowledge Mr S R Potdar for the help provided in sample preparation.

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