Surfactant-assisted atomic-level engineering of spin valves

Harsh Deep Chopra* and David X. Yang
Thin Films & Nanosynthesis Laboratory, Materials Program, Mechanical and Aerospace Engineering Department, State University of New York, Buffalo, New York 14260

P. J. Chen and W. F. Egelhoff, Jr.
Magnetic Materials Group, National Institute of Standards and Technology, Gaithersburg, Maryland 20899
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Surfactant Ag is successfully used to atomically engineer interfaces and nanostructure in NiO-Co-Cu-based bottom spin valves. At a Cu spacer thickness of 1.5 nm, a strong net ferromagnetic (or positive) coupling >13.92 kA/m (>175 Oe) between NiO-pinned and "free" Co layers leads to a negligible "giant" magnetoresistance (GMR) effect (<0.7%) in Ag-free samples. In contrast, the net ferromagnetic coupling could be reduced by a factor of 2 or more in spin valves deposited in the presence of ≈1–3 ML of surfactant Ag, and such samples exhibit more than an order of magnitude increase in GMR (8.5–13 %). Based on transmission electron microscopy (TEM), a large contribution to net ferromagnetic coupling in Ag-free samples could be directly attributed to the presence of numerous pinholes. *In situ* x-ray photoelectron spectroscopy and TEM studies show that surfactant Ag floats out to the surface during deposition of successive Co and Cu overlayers, leaving behind smooth interfaces and continuous layers that are less prone to intermixing and pinholes. The use of surfactants in the present study also illustrates their potential use in atomic engineering of magnetoelectronics devices and other multilayer systems.

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I. INTRODUCTION

Artificially modulated magnetic multilayers are under intense research scrutiny. In these systems, the discovery of several paradigms has added facets of understanding to the known body of knowledge of the physics of magnetism. Examples include perpendicular magnetic anisotropy in ferromagnetic films that are only a few ML thick and sandwiched between non-ferromagnetic metals (examples: Co/Pt, Fe/Cu, etc.);[1] the giant magnetoresistance (GMR) effect[2] (defined as a large change in electrical resistivity as a function of applied magnetic field) in magnetic multilayers which are comprised of ferromagnetic films separated by nonferromagnetic spacers (examples; Co/Cu, Fe/Cr, etc.), etc. Furthermore, mesoscopic scale magnetic order can be established in artificially modulated magnetic multilayers.[3]

During the growth of a multilayer, the surface free energy of one layer is usually higher than the other layer(s). This leads to a wetting of the low-surface-energy layer on the high-surface-energy layer, but agglomeration of the high-surface-energy layer on the low-surface-energy layer. In this context, the basic building block for a broad category of magnetic multilayers is a ML of a *high*-surface-energy transition metal (Ni, Fe, Co) on a *low* surface-energy noble metal (Au, Ag, Cu, etc.). The surface free energy of metals such as Cu(111) (1830 mJ/m^2), Au(111) (1500 mJ/m^2), or Ag(111) (1250 mJ/m^2) is significantly lower than that of Co(111) (3230 mJ/m^2), Fe(111) (2480 mJ/m^2), or Ni(111) (2450 mJ/m^2).[4] Therefore, in addition to agglomeration of magnetic transition metals over the noble metals, the noble atoms tend to segregate out onto Ni, Fe, or Co, giving rise to intermixing across the interface. The end result is a multilayer with rough, diffuse, and intermixed interfaces. An effective avenue that offers control over elementary deposition steps at the atomic level is through the use of surface modifiers, or simply, surfactants.[5,6] Since it was first suggested and experimentally demonstrated that adsorbate layers which float or segregate out to the surface during overlayer growth may be able to favorably alter epitaxial growth, a rapid development of this approach was witnessed in the field of semiconductors,[7–14] and metal-on-metal epitaxy.[5,6,15–20] More recently, surfactants were also shown to favorably alter elementary deposition steps in polycrystalline multilayers.[21–26]

Whereas it has been variously shown that deliberately adsorbed surfactants can be used to gain control over the growth of one layer over another, this is only a first step toward the goal of atomically engineered layered structures. Some way must be found to remove or displace the surfactant species as the growth proceeds. The best method in this regard is to float out the surfactant during overlayer growth. Soft metals with large atomic volume tend to exhibit rapid surface diffusion and low surface free energy, properties that favor their floating out to the surface during overlayer growth, and smoothing an otherwise rough surface. The large atomic volume favors the floating out process, since the incorporation of a large atom in a small lattice costs a great deal of energy in the form of lattice strain. Examples include Pb, In, Hg, Sb, Ag, As, Sn, etc. Whereas the results of surfactant Ag are presented in this paper, the effect of other surfactant species such as Au, Pb, Hg, In, etc. was previously reported elsewhere.[21–26]

The present study discusses the effect of surfactant Ag in favorably altering the nature of interfaces and magnetic properties in polycrystalline NiO-Co-Cu-based GMR bottom spin valves, the simplest magnetoelectronics devices. Preliminary results on the role of surfactant Ag in GMR spin valves were recently presented elsewhere.[26]

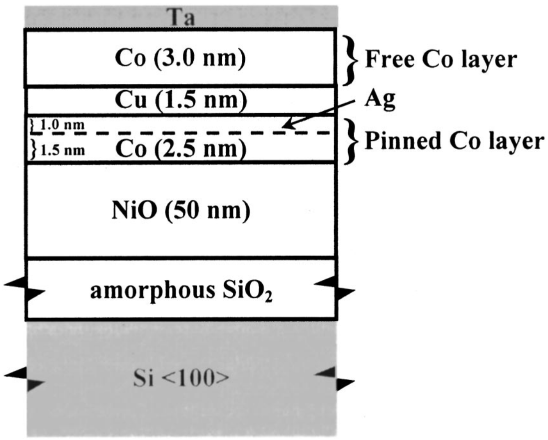

FIG. 1. Schematic of NiO-Co-Cu-based bottom spin valves investigated in the present study. Also shown is the position where surfactant Ag was deposited.

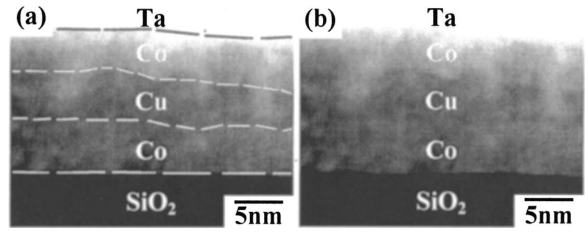

FIG. 2. (a) TEM micrograph of a Co (6.0 nm)/Cu (6.0 nm)/Co (6.0 nm) trilayer on oxidized silicon substrate showing sufficient contrast between the Co/Cu layers is possible under optimized imaging conditions. (b) The same TEM micrographs as in (a) but without the outlined interfaces to aid the viewer to follow the Co/Cu layers.

II. EXPERIMENTAL DETAILS

The dc magnetron sputtered NiO-Co-Cu-based bottom spin valves investigated in the present study were deposited on oxidized Si(100) substrates coated with NiO, and had the following configuration: Co (3.0 nm)/Cu (1.5 nm)/Co (2.5 nm)/NiO (50 nm); a protective Ta film ≈7.5–10.0 nm thick was deposited in order to prevent oxidation of the free Co layer. The configuration of the NiO-Co-Cu-based bottom spin valves is shown schematically in Fig. 1. To study the effect of surfactant Ag, spin valves were deposited with a different choice of interface where surfactant Ag was deposited. However, it was found that deposition of surfactant Ag in between the "pinned" Co layer was most effective in favorably altering the magnetic characteristics of spin valves, and this position is also indicated in Fig. 1. The Ag float-out to the surface during growth of successive Co and Cu overlayers was monitored by *in situ* x-ray photoemission spectroscopy (XPS). Samples were deposited without surfactant Ag as well as samples with ≈1 and ≈3 ML of Ag. Elaborate steps were taken to remove any contamination on the substrates prior to the film deposition. Further experimental details are given elsewhere.[24,27]

The magnetoresistance measurements were made *in situ* using the four-point probe dc mode method. The multiplicative conversion factor from four-point resistance to sheet resistance is of the order of 4, but depends on the actual dimensions of the sample. The structure investigations were performed on a JEOL-2010 high-resolution transmission electron microscope operating at 200 keV. Observations were made on cross-sectioned samples prepared by ion milling in a cold stage using Ar^+ ions (3.5 keV and 1 mA). The cross-section profiles were viewed along the reference Si (110)-zone axis. Due to a close proximity of the elements Co ($Z=27$) and Cu ($Z=29$) in the Periodic Table, images recorded near the optimum Scherzer focus generally give a low composition contrast between adjacent Co and Cu layers. Nonetheless, under optimized imaging conditions (by viewing at defocused values, which increases the scattering factor contrast between the two elements, and due to the presence of Fresnel fringes at the Co-Cu interfaces),[28] the Co and Cu layers can be contrasted,[24,26,27,29] *especially if the individual layer thickness is sufficiently large*. This is shown in a cross-section transmission electron microscopy (TEM) micrograph in Fig. 2 for a Co (6.0 nm)/Cu (6.0 nm)/Co (6.0 nm) trilayer deposited on an oxidized silicon substrate. In case of *thin* Co/Cu layers, as is the case in spin valves, the present study shows that great care must also be taken to ensure that the Co-Cu interfaces are precisely aligned parallel to the incident electron beam. Even small deviations (±1°–2°) from precise sample orientation results in smearing-out of the contrast between the Co/Cu layers, making subsequent analysis difficult or impossible.

III. EXPERIMENTAL RESULTS AND DISCUSSION

Figures 3(a) and 3(b) show the high- and low-field GMR loops of a bottom spin valve sample without Ag, respectively. The low-field GMR loop in Fig. 3(b) shows that the pinned and free Co layers exhibit a large ferromagnetic coupling, which is at least equal to +13.92 kA/m (+175 Oe). (The actual coupling strength should be greater than +13.92 kA/m although its precise value could not be determined due to an overlap between the switching fields for the free and pinned Co layers). This large ferromagnetic coupling precludes the manifestation of any significant GMR effect in the sample, which is less than 0.7% in Fig. 3(a). Figures 3(c) and 3(d) show the high- and low-field GMR loops of bottom spin valve sample with 1 ML of surfactant Ag, respectively. In sharp contrast to the Ag-free sample, the low-field GMR loop of the 1-ML Ag-containing sample in Fig. 3(d) shows that Ag succeeds in reducing the net ferromagnetic coupling by a factor of 2.5 to +5.65 kA/m (+71 Oe). This observed reduction in net ferromagnetic coupling allows sufficient switching of the free Co layer parallel and antiparallel with respect to the NiO-pinned Co layer, giving rise to a large GMR value of 8.5% [Fig. 3(c)], which is more than an order of magnitude higher than in the Ag-free sample. Figures 3(e) and 3(f) show the high- and low-field GMR loops of a sample containing 3-ML Ag, respectively. Figure 3(e) shows that in the presence of 3 ML of surfactant Ag the GMR is further increased to 13%. However, note that there is also a small increase in net ferromagnetic coupling to +7.71 kA/m (+97 Oe) *with respect to the 1-ML Ag sample* in Fig. 3(d), although this value is still roughly half the coupling value in

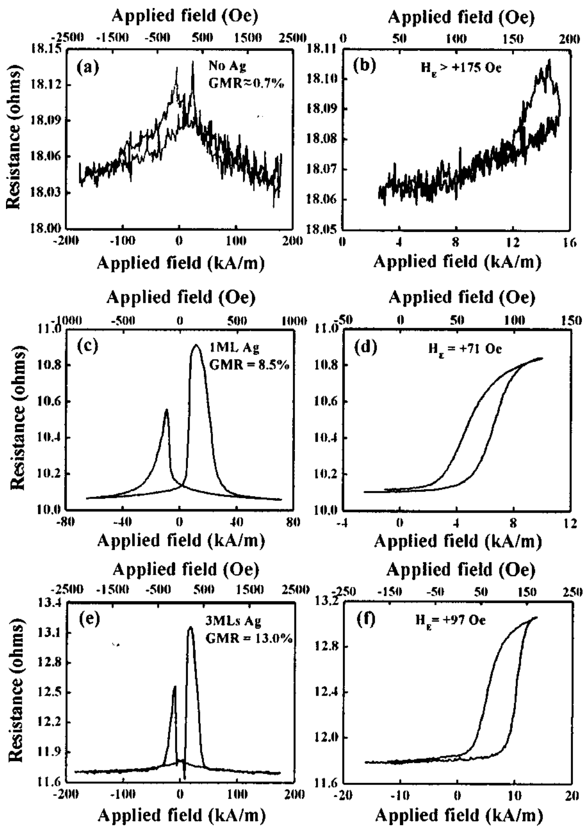

FIG. 3. (a) and (b) High- and low-field GMR loops of an Ag-free spin valve, respectively. (c), and (d) High- and low-field GMR loops of a 1-ML surfactant Ag-containing bottom spin valve, respectively. (e), and (f) High- and low-field GMR loops of a 3-ML surfactant Ag-containing bottom spin valve, respectively.

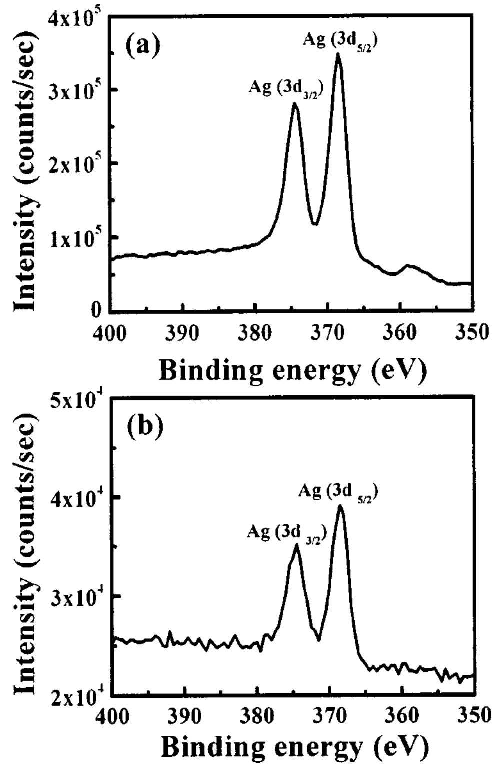

FIG. 4. (a) Reference profile of the Ag 3*d* XPS intensity for 0.4-nm Ag on Co. (b) Profile of the Ag 3*d* XPS intensity for segregated Ag on the "pinned" Co layer in a spin valve with 1-ML Ag. See Fig. 1 for the position where 1-ML Ag was initially deposited prior to its segregation through overlayers of Cu and Co in the spin valve.

the Ag-free sample in Fig. 3(b). As discussed below, the origin of this increase in coupling is due to Ag-modified interface topography that is conducive to Néel's so-called "orange-peel" effect.[30]

The XPS studies showed that surfactant Ag floats out to the surface during overlayer growth, although some Ag is incorporated into the Co and Cu overlayers. The XPS profiles of the Ag float-out are shown in Fig. 4. Figure 4(a) shows a reference XPS profile for 2 ML of Ag on the surface of a 1.0-nm Co film. As seen from Fig. 4(a), 2 ML of Ag correspond to approximately 380 kcps intensity; therefore, 1-ML Ag corresponds to 190 K cps. With respect to this reference XPS profile, Fig. 4(b) shows the XPS spectrum obtained from the amount of Ag that has floated out to the surface of the spin valve (in Fig. 1) having 1 ML of surfactant Ag. Figure 4(b) shows that following the deposition of 1-ML Ag in between the "pinned" Co layer, its subsequent float-out following the deposition of the remaining "pinned" Co layer, the Cu spacer layer, and the "free" Co layer gives an intensity for Ag of about 32 kcps. This indicates that $\frac{1}{6}$-ML Ag floats out to the surface. This effect of incorporation of a small amount of Ag in *each* layer was a small or nominal increase in the thickness of various layers of the spin valves. Thus, with respect to the Ag-free sample, the thickness of pinned Co layer (based on XPS measurements) was changed from 2.5 to 2.54 nm in the 1-ML Ag-containing sample, and to 2.6 nm in the 3-ML Ag-containing sample. Similarly, the Cu spacer thickness was slightly increased from 1.5 nm in an Ag-free sample to 1.54 nm in a 1-ML Ag sample, and to 1.62 nm in the 3-ML Ag-containing sample. The free Co layer thickness was also increased slightly from 3.0 nm to 3.08 and 3.25 nm in 1- and 3-ML Ag-containing samples, respectively. The remaining Ag floats out to the surface, over which the protective Ta layer was deposited. For the above-described small or nominal change in the Cu thickness, it is important to note that the typical coupling strength in Ag-free samples (deposited under similar growth conditions) as a function of Cu spacer thickness lies between +15.91 kA/m (+200 Oe) and +9.94 kA/m (+125 Oe) for Cu spacer thickness between 1.5 and 1.7 nm, respectively. Therefore, the observed large reduction in net ferromagnetic coupling in Ag-containing samples (even after taking into account an increase in the Cu layer thickness due to some Ag incorporation) can be directly attributed to the salubrious effect of surfactant Ag.

Figure 5 shows typical cross-section TEM micrographs of Ag-free spin valves. Figures 5(a) and 5(c) show micrographs with outlined Co-Cu interfaces in the Ag-free spin valve. Figures 5(b) and 5(d) show the same micrographs as in Figs. 5(a) and 5(c), respectively, but without the outlined interfaces to aid the reader to follow the position of the Co/Cu layers. The micrographs in Figs. 5 show that the Cu layer

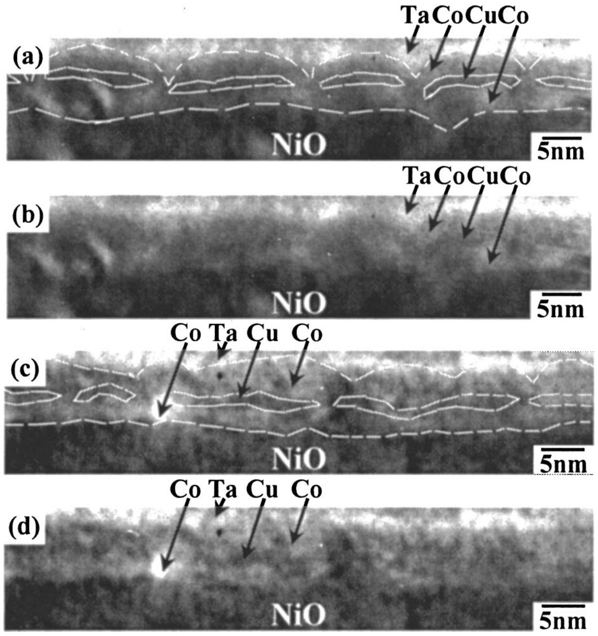

FIG. 5. (a) and (c) TEM micrographs of an Ag-free spin valve showing numerous pinholes. (b) and (d) Same TEM micrographs as in (a) and (c), respectively, but without the outlined interfaces.

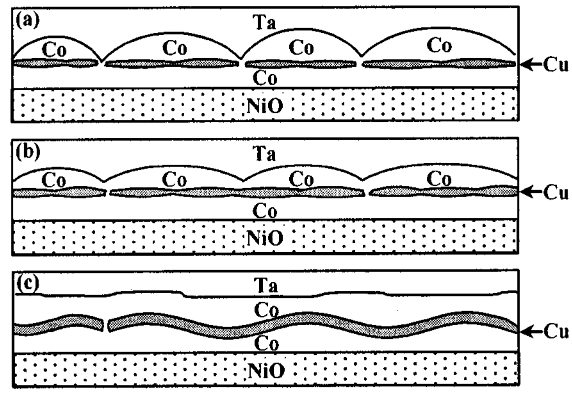

FIG. 6. Schematics of layer profiles (a) in Ag-free spin valves, (b) in 1-ML Ag samples, and (c) in 3-ML Ag samples. The schematics are based on observations of numerous TEM micrographs of Ag-free and Ag-containing samples.

sandwiched between the Co layers is barely continuous in the Ag-free sample. This shows that the lower-surface-energy Cu layer can remain discontinuous even when it is deposited over the high-surface-energy Co layer having a rough surface through roughness-induced pinholes.[31] (In addition, intermixing at the Co/Cu interfaces was apparent from a thinner Cu layer in the sample). As a result, the Ag-free sample contains numerous pinholes, thereby bridging the NiO-pinned and free Co layers together. It is this bridging that gives rise to a very large contribution to the net coupling in the Ag-free samples, as seen from the low-field GMR loop in Fig. 3(b). The TEM results also show that the Co and Cu layers in the Ag-free spin valves do not grow epitaxially. Instead, the Co and Cu layers consist of (apparently) randomly oriented nanocrystallites in successive metal layers. Based on examination of numerous TEM micrographs, a typical schematic of the profiles of the Co and Cu layers in the Ag-free samples is shown in Fig. 6(a). Also, from an analysis of TEM micrographs, roughly 2% of the Cu layer was estimated to be discontinuous. (A quantitative estimation of pinhole coupling strength as a function of pinhole population is beyond the scope of this work, and was reported elsewhere).[31] Figure 7 shows typical TEM micrographs of 1-ML Ag-containing spin valves, with Figs. 7(b) and 7(d) being their respective counterparts without the outlined interfaces. In contrast to the highly discontinuous nature of the Co/Cu layers in the Ag-free samples, the micrographs in Figs. 7 show that the Cu spacer layer in 1-ML Ag sample is more continuous, and contains fewer pinholes. As Ag floats out to the surface, it leaves behind a smoother surface of a pinned Co layer over which the Cu layer is deposited. Based on an observation of TEM micrographs, the degree of intermixing at the Co/Cu interfaces in the 1-ML Ag sample is reduced with respect to the Ag-free sample. Note that the location where the surfactant Ag is deposited is an important consideration in favorably modifying the structure and magnetic properties of spin valves. Surfactant Ag was most effective when it was *inserted* within the pinned Co layer, and less effective when it was deposited *over* it, or at the Cu-free Co interface. At first, it would appear that Ag should be deposited on Cu prior to the deposition of the free Co layer. However, it is important to note that *a discontinuous Cu layer is a prerequisite to the formation of pinholes*. Due to a small Cu spacer thickness of 1.5 nm (approximately 5–6 ML of Cu) used in the present study, if the Cu layer is deposited over a rough pinned Co layer, a discontinuous Cu

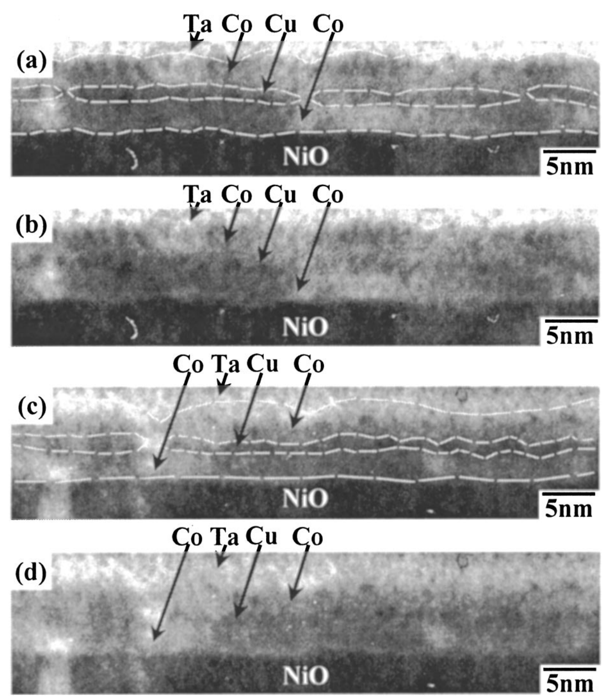

FIG. 7. (a) and (c) TEM micrographs of a 1-ML surfactant Ag spin valve showing almost continuous Co/Cu layers with few pinholes in comparison to Ag-free spin valves. (b) and (d) Same TEM micrographs as in (a) and (c), respectively, but without the outlined interfaces.

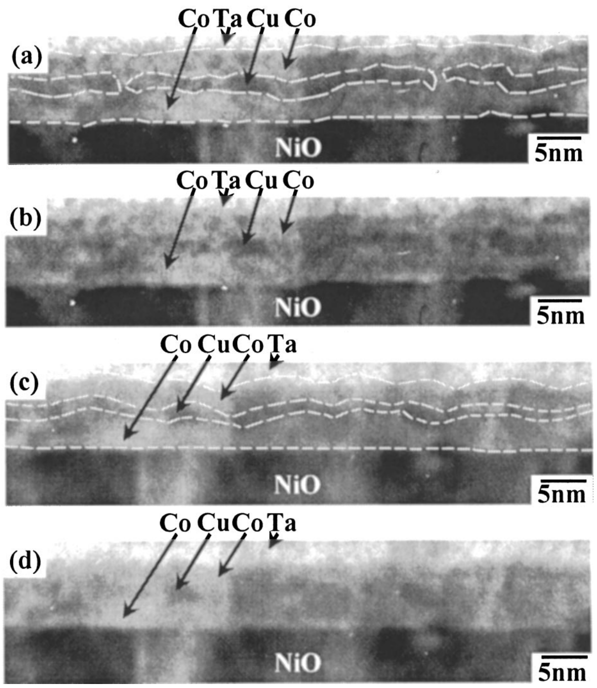

FIG. 8. (a) and (c) TEM micrographs of a 3-ML surfactant Ag spin valve showing highly continuous Co/Cu layers with only occasional pinholes. (b), and (d) Same TEM micrographs as in (a) and (c), respectively, but without the outlined interfaces.

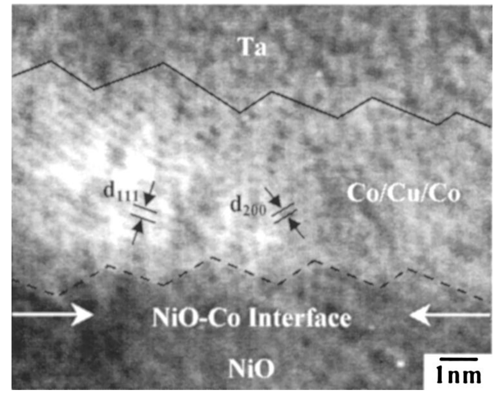

FIG. 9. HRTEM micrograph showing topographically correlated Co-Cu interfaces in a 3-ML surfactant Ag-containing sample. The topographic correlation arises due to an agglomeration of surfactant Ag, and its subsequent float out to the surface.

layer results. Any subsequent use of surfactants is then unable to prevent the direct contact between the free Co layer and the pinned Co layer, notwithstanding the degree of smoothness of the free Co layer induced by surfactant Ag. To ensure this does not happen, Ag was *inserted* within the pinned Co layer. When Ag is inserted within the pinned Co layer, it continues to float out during the growth of the remaining pinned Co overlayer, leaving behind a smooth surface over which a continuous Cu is deposited. Subsequently, as the Ag floats out, it further promotes a smoother growth of the high surface energy Co layer over Cu. In contrast, when it is deposited on top of the pinned Co layer, only the topography of the Cu-free Co interface is changed. Similar to the Ag-free sample, TEM results show that Co and Cu layers in the 1-ML Ag samples do not grow epitaxially. A schematic of the typical profiles of the Co and Cu layers in the 1-ML Ag samples is shown in Fig. 6(b). Figure 8 shows typical TEM micrographs of spin valve samples with 3-ML Ag; again Figs. 8(b) and 8(d) show the same micrographs as in Figs. 8(a) and 8(c), respectively, but without the outlined interfaces. Figures 8 show that unlike the Ag-free samples, the 3-ML Ag-containing samples contain only occasional pinholes. A typical schematic of the profiles of the Co and Cu layers in the 3-ML Ag samples is shown in Fig. 6(c).

The results presented above show that the observed reduction in net ferromagnetic coupling in both the 1- and 3-ML Ag samples in comparison to the Ag-free samples can be explained in terms of a large reduction in the number of pinholes in the Ag-containing samples. However, there are important differences in terms of contribution to net coupling from different mechanisms between zero Ag, 1-ML Ag, and 3-ML Ag samples. The 3-ML Ag sample has the highest GMR and a somewhat higher coupling ($+7.71$ kA/m or $+97$ Oe, 13% GMR) compared to the 1-ML Ag sample ($+5.65$ kA/m or $+71$ Oe; 8.5% GMR), but both samples have much lower coupling in comparison to the Ag-free sample (>13.92 kA/m or >175 Oe; $<0.7\%$ GMR). The 3-ML Ag sample has very little pinhole coupling, and the net coupling receives a contribution from oscillatory exchange[32] and Néel's "orange-peel" coupling,[30] which requires topographically correlated interfaces. Highly correlated Co-Cu interfaces in the 3-ML Ag samples are clearly evident in the medium magnification TEM micrographs in Figs. 8, as well as the high-resolution TEM (HRTEM) micrograph in Fig. 9. The TEM studies show that the average grain size λ in the films is ≈ 30 nm and the average roughness A is ≈ 0.5–0.6 nm in the 3-ML Ag sample. Using these values, the calculated value of average coupling strength due to the orange-peel effect in the film is $0.012\ \mathrm{mJ\,m^{-2}}$. This coupling, expressed in terms of a fictitious coupling field h acting on the free Co layer of thickness t, corresponds to an *added* shift in the GMR loop equal to $+2.3$ kA/m ($+29$ Oe). The remaining contribution to the net coupling in this sample is due to oscillatory exchange. In case of the 1-ML Ag sample, Fig. 7 shows that the interfaces are not topographically correlated (a key to the manifestation of the orange-peel effect). Therefore, the net coupling in the 1-ML Ag sample is due to oscillatory exchange coupling and contributions from occasional pinholes. In the case of the Ag-free spin valve, the pinholes makes the majority contribution to the net coupling. Due to the intermixed and diffuse interfaces in the Ag-free sample, the oscillatory coupling is likely to be weak. (The effect of interface roughness and intermixing on the strength of oscillatory exchange coupling is evident from a higher GMR in the 3-ML Ag sample compared to the 1-ML Ag sample). Unlike the estimation of the strength of the orange-peel coupling, unfortunately, it is very difficult to precisely estimate the strength of oscillatory exchange coupling in polycrystalline samples having rough interfaces. Similarly, an estimation of pinhole coupling requires a systematic variation of the pinhole population as a function of the Cu spacer thickness, which is beyond the scope of this paper, and discussed elsewhere.[31]

Finally, previous experiments on the surfactant-assisted epitaxial growth of transition metals on noble metals under ultrahigh-vacuum conditions provided some clues as to the promotion of a faceted surface morphology in 3-ML Ag samples and its absence in 1-ML Ag samples.[5,33] These experiments showed that the background oxygen atoms in the deposition chamber (for example, oxygen atoms knocked off from the chamber walls) could have a profound effect on the mechanism by which Ag floats out to the surface. Due to the strong affinity of oxygen to magnetic transition metals, the oxygen atoms cause an agglomeration of surfactant Ag, in order to attach themselves to the underlying magnetic transition atoms. This leads to an islanding of the surfactant Ag, and subsequent wavy characteristics of the Co/Cu overlayers as Ag floats out to the surface. In contrast, these experiments show an interesting behavior when only 1 ML of surfactant is present. In this instance, instead of an agglomeration of the surfactant over the transition magnetic atoms, results suggest that Ag floats out uniformly *along with 1 ML of the magnetic transition metals*. A similar behavior may be found in the present films, although as in previous experiments conclusive experiments would be very difficult due to an inability to eliminate the background oxygen atoms completely.

IV. SUMMARY

Surfactant Ag was used to alter the growth mode in NiO-Co-Cu-based bottom spin valves favorably. It was found that at a Cu thickness of 1.5 nm, a large ferromagnetic coupling at >13.92 kA/m($+175$ Oe) exists between pinned and free Co layers in the Ag-free spin valves, and a small GMR effect of less than 0.7%. Based on TEM studies, a large contribution to the net ferromagnetic coupling in Ag-free samples could be directly attributed to the existence of numerous pinholes. Results show that 1 and 3 MLs of Ag deposited in between the pinned Co layer succeeds in reducing the net ferromagnetic coupling by roughly a factor of 2 or more. This reduction in the net ferromagnetic coupling is sufficient to enable the free Co layer to switch between being parallel and antiparallel with respect to the pinned Co layer. As a result Ag-containing samples exhibit an order of magnitude or more increase in GMR (8.5% in 1-ML Ag samples and 13% in 3-ML Ag samples, in comparison to Ag-free samples). The existence of a large number of pinholes in the Ag-free samples could be directly attributed to an inability of the 1.5-nm-thick Cu layer to cover the rough surface of the pinned Co layer completely, giving rise to roughness induced pinholes. *In situ* x-ray photoelectron spectroscopy results show that, in Ag-containing samples, surfactant Ag floats out to the surface during deposition of successive Co and Cu overlayers, leaving behind smooth surfaces and continuous layers that are less prone to intermixing and pinholes. The use of surfactants in the present study illustrates their potential use in the atomic engineering of thin films, to favorably alter the physical properties not only in GMR films, but also in magnetoelectronics devices and other multilayer systems that are currently the focus of intense research activities due to a fundamental interest in their electron transport properties, interesting magnetic behavior, and myriad technological applications.

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*Corresponding author.
Electronic address: hchopra@eng.buffalo.edu

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