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Authors
McMorran, B. J.
Cochran, A. C.
Dumas, Randy K.
et al.

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Measuring the effects of low energy ion milling on the magnetization of Co/Pd multilayers using scanning electron microscopy with polarization analysis

B. J. McMorran,[1,a)] A. C. Cochran,[1] R. K. Dumas,[2] Kai Liu,[2] P. Morrow,[3] D. T. Pierce,[1] and J. Unguris[1]
[1]*Center for Nanoscale Science and Technology, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA*
[2]*Department of Physics, University of California, Davis, California 95616, USA*
[3]*Materials Science and Engineering Laboratory, National Institute of Standards and Technology, Gaithersburg, Maryland 20899, USA*

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The dependence of the magnetization profile of Co/Pd multilayer films with very thin individual layers, Co(0.4 nm)/Pd(0.6 nm), on the energy of ion milling is investigated using scanning electron microscopy with polarization analysis (SEMPA). The effect of Ar ion milling on the Co/Pd magnetization angle distribution is compared for ion milling at 50 eV, 1 keV, and 2 keV. We find that 1 and 2 keV Ar ion milling causes a measurable change in the out-of-plane magnetization angle distribution as material is removed, but ion milling with 50 eV Ar ions does not significantly alter the magnetization. This enables quantitative imaging of all three vector components of the surface magnetization of the Co/Pd multilayer films with 20 nm lateral spatial resolution using SEMPA.
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The out-of-plane magnetization of Co/Pd or Co/Pt multilayers has been studied extensively,[1] and these important materials continue to garner much attention due to the promise they hold for future high density data storage technologies[2,3] and magnetoelectronics. However, complete characterization of the *magnetization vector* within these structures, crucial for understanding their behavior under applied fields, has been difficult because the sensitivity of most magnetization imaging techniques is limited to a single component of the magnetization. Scanning electron microscopy with polarization analysis (SEMPA) (Ref. 4) can image all three vector components of magnetization, but requires a clean, uncoated ferromagnetic surface because of the SEMPA 1 nm probing depth enforced by the shallow escape depth of polarized secondary electrons.[5] For many samples surface cleaning can be accomplished with *in situ* Ar ion milling at 1–2 keV energy. However, ion bombardment can alter the magnetic structure of Co/Pd multilayers by disordering the thin film interfaces that give rise to the out-of-plane anisotropy.[6,7] This disordering effect itself is a subject of interest (for reviews see Refs. 8 and 9), motivated by the possibility of patterning magnetic structures with perpendicular anisotropy by selected exposure to high energy (10–100 keV) ions and locally modifying the magnetic properties.

In this work, we use SEMPA to explore the effects of ion bombardment on Co/Pd multilayer magnetization using lower energy ions. In particular, our goal was to find *in situ* cleaning procedures that enable SEMPA imaging without altering the magnetic structure of these delicate samples. We show that low-energy (50 eV) plasma-generated Ar ions can be used to clean and mill through the multilayers with minimal damage to the magnetic structure. This allows complete, quantitative imaging of the in-plane and out-of-plane magnetic configurations in these multilayer films using SEMPA.

The Co/Pd multilayer in this study consists of 60 bilayers of Co(0.4 nm)/Pd(0.6 nm) deposited on a 20 nm Pd seed layer and topped with a 5 nm Pd capping layer. The multilayer was dc magnetron sputtered onto a Si substrate using an Ar working pressure of 0.7 Pa. This sample is similar to the "low pressure" sample discussed in Ref. 10. To produce a distribution of magnetic domains, the sample was first subjected to a +1.5 T saturating magnetic field normal to the surface, and then to a −0.1 T reversal field.

Two different ion sources, both installed on the SEMPA microscope, were used for the work described herein. For higher ion energies (>500 eV), a conventional sputter ion gun with a hot filament ionization source was used. This ion gun delivers a focused beam of Ar ions directed 40° from normal to the sample surface. The beam diameter was about 200 μm and the raster area was 1 mm^2. For lower ion energies, down to 50 eV, we used Ar ions from a microwave coupled plasma source. This broad beam ion source delivered a beam of Ar ions normal to the sample surface. In this paper we present measurements for 1 and 2 keV ions from the sputter ion source and for 50 eV ions from the plasma source. The average milling rate at 50 eV was 2.5 nm/min, at 1 keV it was 4.4 nm/min, and at 2 keV it was 7.0 nm/min.

Our SEMPA microscope has two orthogonal polarization analyzers which can each measure two polarization vector components, thus providing sensitivity to all three components of the magnetization vector. One analyzer can simulta-

a)Electronic mail: mcmorran@nist.gov.

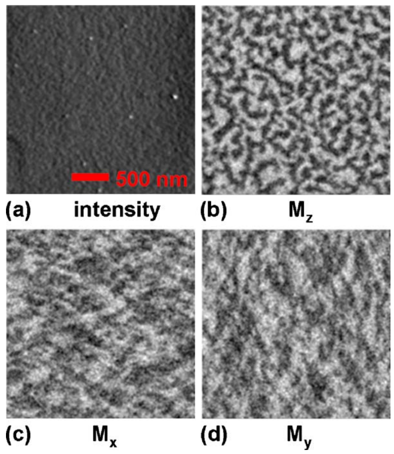

FIG. 1. (Color online) SEMPA measurements of a Co/Pd multilayer surface display (a) the surface topography, (b) the out-of-plane surface magnetization, and [(c) and (d)] the in-plane surface magnetization. This image was recorded after 52 nm of material (including capping layer) was removed.

neously measure the topography and both in-plane magnetization components (M_x and M_y) in a single scan. The other, accessed by simply deflecting the secondary electrons by 90°, simultaneously measures the topography, one in-plane component (M_x), and the out-of-plane component (M_z). An example of a SEMPA image of a Co/Pd multilayer film at remanence is shown in Fig. 1. The complete set of images in Fig. 1 representing all three magnetization vector components required two consecutive scans, one for M_x and M_y and one for M_x and M_z.

The direction and magnitude of the magnetization vector can be derived from the components, as shown in Fig. 2. Figure 2(a) is the direction of the in-plane magnetization [$\arctan(M_y/M_x)$]. The angles are represented by the colors shown in the inset colorwheel. Similarly, Fig. 2(b) is the direction of the magnetization in the x-z plane [$\arctan(M_z/M_x)$]. The y-z plane image is similar, since there is no preferential alignment of the magnetization in the x-y plane. The magnetization tilt angle from the surface normal, $\arctan(M_z/\sqrt{M_x^2+M_y^2})$, was also computed in some cases, but this required the exact registration of two separate images. The resulting distribution of angles was similar to that obtained using x-z images alone. We therefore used the distribution of angles in the x-z plane to quantify the degree of perpendicular magnetization, as shown in Fig. 2(c). In this case, the histogram shows that the magnetization is primarily out-of-plane, with the magnetization mostly aligned along the perpendicular, 90° and 270°, directions. After examining all three components of the magnetization, no evidence was found for the cone state observed in previous SEMPA studies of Co/Pt multilayers.[11]

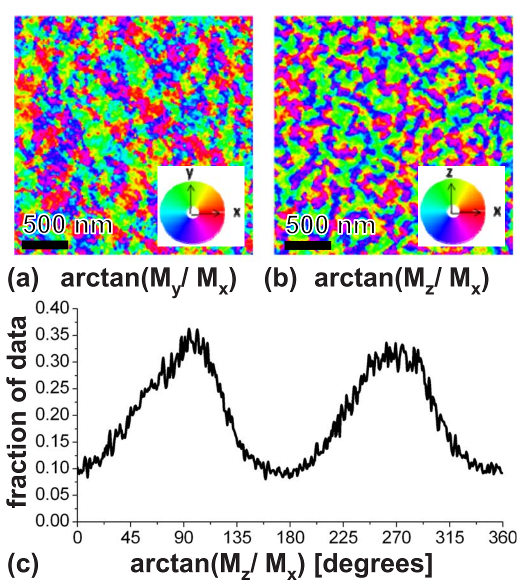

FIG. 2. (Color online) Images of (a) the in-plane and (b) out-of-plane magnetization angle are calculated from magnetization component images (Fig. 1). (c) A histogram of image (b) shows the fraction of image pixels with a particular magnetization angle in the x-z plane and indicates that the magnetic domains point mostly out of plane.

To determine the sensitivity of the magnetic structure to ion milling, a series of SEMPA images was acquired while milling completely through a Co/Pd multilayer film. There were 18 milling steps at 50 eV, 15 steps at 1 keV, and 13 steps at 2 keV, and the associated mill depths were determined with a precision of ± 2 nm from the mill times and rates. The SEMPA measurements began after the ion milling removed the outermost contamination and Pd capping layers, and ended with the exposure of the underlying Pd seed layer, determined by Auger electron spectroscopy. SEMPA images show a mixture of labyrinth domains and larger (>500 nm) domains just after the Pd cap is removed. Little change is seen in this domain structure during 50 eV ion milling. After more than five to ten bilayers have been removed by 1 and 2 keV ion milling, the large magnetic domains begin breaking up into labyrinth domains, which then remain unchanged during further milling. The evolution of the magnetization orientation during ion milling through the multilayer is summarized in Fig. 3. This figure consists of a series of histograms similar to Fig. 2(c) which were calculated for each SEMPA measurement and plotted as a function of milling depth. The histogram levels are color coded to convey the distribution of magnetization angles.

The left side of the plots in Fig. 3 show dramatic differences between the various ion milling energies. At 50 eV the perpendicular magnetization orientation is largely unaffected as more multilayer material is sputtered away, as can be seen by the horizontal red stripes in the top plot of Fig. 3. In contrast, ion milling with 2 keV ions has significantly reduced the perpendicular alignment even after just milling through the 5 nm Pd capping layer. For 1 keV Ar ion milling the magnetization starts out preferentially aligned out-of-plane after removing the Pd capping layer, but the distribution broadens and becomes more in-plane after milling through just 10 nm of multilayer. The decrease in perpendicular alignment and the spread in the distribution of magnetization orientation at higher ion energies are consistent with a decrease in perpendicular anisotropy due to ion beam-induced interfacial disorder in the multilayer structures. For ion milling at 50 eV the narrower angular distribution indicates that the ion-induced disorder is much smaller.

For ion milling at all three energies, the magnetization at the surface eventually lies down completely in-plane after about 45 nm of the 60 nm thick multilayer stack has been

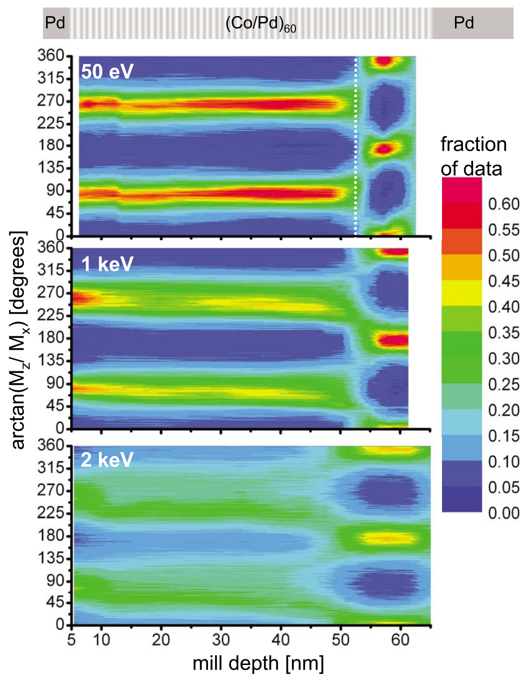

FIG. 3. (Color online) Magnetization angle distribution as a function of mill depth using 50 eV (top plot), 1 keV (middle), and 2 keV Ar ions (bottom plot). A schematic of the multilayer is shown at the very top to guide the eyes. These contour plots are composed of histograms of the magnetic polarization angle in the x-z plane recorded during ion milling [the histogram in Fig. 2(c) is indicated by a white dotted line in the top plot]. Ion milling with 50 eV ions does not change the initial out-of-plane magnetization distribution, as indicated by the two red horizontal stripes at 90° and 270°, while milling with higher energy ions increases the width of the angular distribution as the magnetization tilts more in-plane. At all milling energies, the magnetization is seen to lay down completely in-plane after most of the bilayers have been removed (right side of plots).

sputtered away (right side of Fig. 3 plots). However, the perpendicular anisotropy should persist even in the thin film limit,[12,13] which is inconsistent with the observed in-plane magnetization near the end of the milling process. It is interesting to note that in Co thin films a similar transition from out-of-plane to in-plane magnetization has been observed after bombardment by 10 keV (Ref. 14) and 30 keV He^+,[6,15,16] ~30 keV Ga+,[17,18] and by other ion species at much higher energies.[7,17–19] These studies all find that the magnetization transitions from out-of-plane to in-plane at a comparable ion fluence to ours (10^{15}–10^{16} cm^{-2}) and attribute this change to the destruction of the interfacial anisotropy[8,15,20–22] caused by erosion of the well-defined interfaces between Co and nonmagnetic (Pd, Pt, or Au) layers. Similar processes are probably present in this work at lower ion energies, although a direct comparison of fluences is complicated by the different ion/solid interactions at these very different energies. The in-plane surface magnetization is most likely a result of the microstructural change caused by the milling.

In conclusion, we demonstrated that low energy (50 eV) argon ions can be used to clean delicate Co/Pd magnetic multilayer surfaces with no apparent alteration of the magnetic configuration. Of course some surface damage may occur during the initial cleaning before the first SEMPA image. Removal of larger amounts of multilayer material with 50 eV ions causes only minor changes in remanent domain structure and leaves the distribution of magnetization angles undisturbed, which suggests that any surface damage due to this process remains constant with mill depth and does not appear to affect the magnetization measurement. These results point to the possibility of using ultralow energy ion milling in combination with SEMPA for magnetic depth profiling, similar in principle to compositional depth profiling.[23] Of course magnetic depth profiling would require accounting for the effects of an altered magnetostatic environment caused by the removal of magnetic overlayers. In general, low energy ion milling with 50 eV ions will enable high resolution SEMPA measurements of all three components of the magnetization direction in various Co/Pd multilayer structures.

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[1] P. F. Carcia, A. D. Meinhaldt, and A. Suna, Appl. Phys. Lett. **47**, 178 (1985).
[2] B. D. Terris, T. Thomson, and G. Hu, Microsyst. Technol. **13**, 189 (2007).
[3] I. Yoo, D. K. Kim, and Y. K. Kim, J. Appl. Phys. **97**, 10C919 (2005).
[4] M. R. Scheinfein, J. Unguris, M. H. Kelley, D. T. Pierce, and R. J. Celotta, Rev. Sci. Instrum. **61**, 2501 (1990).
[5] D. L. Abraham and H. Hopster, Phys. Rev. Lett. **58**, 1352 (1987).
[6] C. Chappert, H. Bernas, J. Ferré, V. Kottler, J. P. Jamet, Y. Chen, E. Cambril, T. Devolder, F. Rousseaux, V. Mathet, and H. Launois, Science **280**, 1919 (1998).
[7] J. Q. Xiao, K. Liu, C. L. Chien, L. F. Schelp, and J. E. Smith, J. Appl. Phys. **76**, 6081 (1994).
[8] J. Fassbender, D. Ravelosona, and Y. Samson, J. Phys. D **37**, R179 (2004).
[9] J. Fassbender and J. McCord, J. Magn. Magn. Mater. **320**, 579 (2008).
[10] B. J. Kirby, S. M. Watson, J. E. Davies, G. T. Zimanyi, K. Liu, R. D. Shull, and J. A. Borchers, J. Appl. Phys. **105**, 07C929 (2009).
[11] R. Frömter, H. Stillrich, C. Menk, and H. P. Oepen, Phys. Rev. Lett. **100**, 207202 (2008).
[12] O. Hellwig, A. Berger, and E. E. Fullerton, J. Magn. Magn. Mater. **290–291**, 1 (2005).
[13] J. E. Davies, O. Hellwig, E. E. Fullerton, M. Winklhofer, R. D. Shull, and K. Liu, Appl. Phys. Lett. **95**, 022505 (2009).
[14] M. Urbaniak, F. Stobiecki, D. Engel, B. Szymański, and A. Ehresmann, Acta Phys. Pol. A **115**, 326 (2009).
[15] T. Devolder, J. Ferré, C. Chappert, H. Bernas, J. P. Jamet, and V. Mathet, Phys. Rev. B **64**, 064415 (2001).
[16] D. T. Smith, L. Chang, J. O. Rantschler, V. Kalatsky, P. Ruchhoeft, S. Khizroev, and D. Litvinov, IEEE Trans. Magn. **45**, 3554 (2009).
[17] T. Aign, P. Meyer, S. Lemerle, J. P. Jamet, J. Ferré, V. Mathet, C. Chappert, J. Gierak, C. Vieu, F. Rousseaux, H. Launois, and H. Bernas, Phys. Rev. Lett. **81**, 5656 (1998).
[18] J. Jaworowicz, A. Maziewski, P. Mazalski, M. Kisielewski, I. Sveklo, M. Tekielak, V. Zablotskii, J. Ferré, N. Vernier, A. Mougin, A. Henschke, and J. Fassbender, Appl. Phys. Lett. **95**, 022502 (2009).
[19] D. Weller, J. E. E. Baglin, A. J. Kellock, K. A. Hannibal, M. F. Toney, G. Kusinski, S. Lang, L. Folks, M. E. Best, and B. D. Terris, J. Appl. Phys. **87**, 5768 (2000).
[20] G. S. Chang, A. Moewes, S. H. Kim, J. Lee, K. Jeong, C. N. Whang, D. H. Kim, and S.-C. Shin, Appl. Phys. Lett. **88**, 092504 (2006).
[21] S. Kavita, V. R. Reddy, S. Amirthapandian, A. Gupta, and B. K. Panigrahi, J. Phys.: Condens. Matter **21**, 096003 (2009).
[22] T. Devolder, Phys. Rev. B **62**, 5794 (2000).
[23] A. W. Czanderna, T. E. Madey, and C. J. Powell, *Beam Effects, Surface Topography, and Depth Profiling in Surface Analysis* (Plenum, New York, 1998).