Measuring the effects of low energy ion milling on the magnetization of Co/Pd multilayers using SEMPA

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The dependence of the magnetic properties of Co/Pd multilayer films with very thin individual layers, Co(0.4nm)/Pd(0.6nm), 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 keV and 2 keV Ar ion milling causes a measureable 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 distribution. This enables quantitative imaging of all three vector components of the surface magnetization of the Co/Pd multilayer films with 20 nm spatial resolution using SEMPA.

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 magneto-electronics. However, complete characterization of all three directional components of the magnetization 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 vector. scanning electron microscopy with polarization analysis (SEMPA) [4] can image all three vector components of magnetization, but requires a clean, uncoated ferromagnetic surface because of SEMPA's 1 nm probing depth which is limited 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 [8,9]), motivated by the possibility of patterning magnetic structures in Co/Pd films by locally modifying the magnetic properties using patterned masks or focused beams, and medium to high energy (10^1 to 10^3 keV) ions.

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 cleaninig 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 nanostructure 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 identical to the "low pressure" sample discussed in [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. The samples were imaged in the remanent state.

Two different ion sources, both installed on the SEMPA microscope, were used for the work described here. 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 20 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 keV and 2 keV ions from the sputter ion source and for 50 eV ions from the plasma source. The 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, providing sensitivity to all three components of the magnetization vector. One analyzer can simultaneously 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. Fig. 2a 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. Similarily, Fig. 2b 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. Derivation of angles in the y-z plane is also complicated by slight image shifts between the x-y and x-z images. To quantify the degree of perpendicular magnetization we extract a histogram from Fig. 2b which plots the distribution of image pixels as a function of magnetization angles in the x-z plane, as shown in Fig. 2c. 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.

To determine the sensitivity of the magnetic structure to ion milling, a series of SEMPA images were 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 indicate no change in the domain structure just after the Pd cap is removed. Large magnetic domains are seen to break up into maze domains after more than 5 to 10 bilayers have been removed, but no change in the distribution of magnetization angles is observed during these transitions. Areas with maze domains remain unchanged during ion 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. 2c which were calculated for each SEMPA measurement and plotted as a function of milling depth. The histograms in the figure are color-coded to convey the distribution of magnetization angles.

The data in left side of the plots in Fig. 3 shows 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 distributuion 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 is consistent with a decrease in perpendicular anisotropy due to ion beam induced interfacial disorder in the multilayer structures. At 50 eV the disorder is small enough that it does not affect the perpendicular alignment.

For ion milling at all three energies, the magnetization at the surface eventually lays down almost completely in-plane after about 45 nm of the 60 nm thick multilayer stack has been sputtered away (right side of Fig. 3 plots). We do not know if this in-plane structure is the result of ion milling, however it is interesting to note that similar effects have been observed with higher energy ions. In Co thin films a similar transition has been observed after bombardment by 10 keV [11] and 30 keV He^+ [6,12,13], ~30 keV Ga+ [14-20], and by other ion species at much higher energies [7,21-23]. These studies all find that the magnetization transitions from out-of-plane to in-plane at a comparable fluence to ours (10^{15} to 10^{16} ions/cm^2). At high energies the transition is attributed to the gradual erosion of the well-defined interfaces between Co and nonmagnetic (Pd, Pt, or Au) layers and the resulting destruction of the interfacial anisotropy [8,12,24]. 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.

In conclusion, we have demonstrated that low energy (50 eV) argon ions can be used to clean delicate Co/Pd magnetic multilayer surfaces without significantly altering the magnetization. Removal of larger amounts of multilayer material with 50 eV ions can cause small changes in remanent domain structure, but the distribution of magnetization angles is undisturbed. These results point to the possibility of using ultra low energy ion milling in combination with SEMPA for magnetic depth profiling, similar in principle to compositional depth profiling [25]. 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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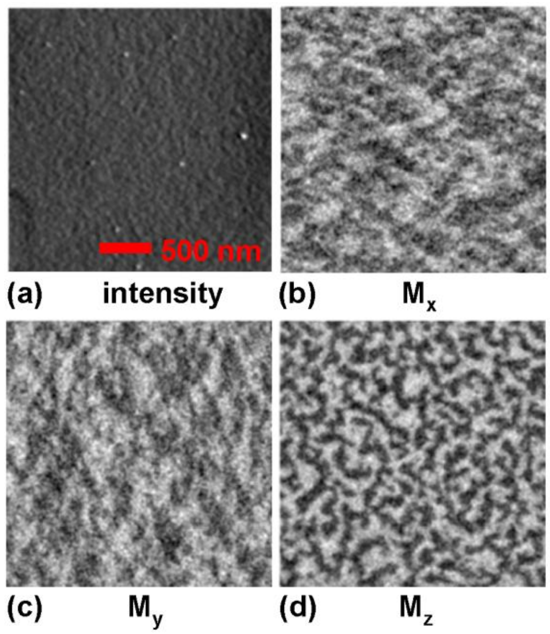

FIG. 1. SEMPA measurements of a Co/Pd multilayer surface display (a) the surface topography, (b) and (c) the in-plane magnetization, and (c) the out-of-plane magnetization. This image was recorded after 53 nm of material (including capping layer) had been removed.

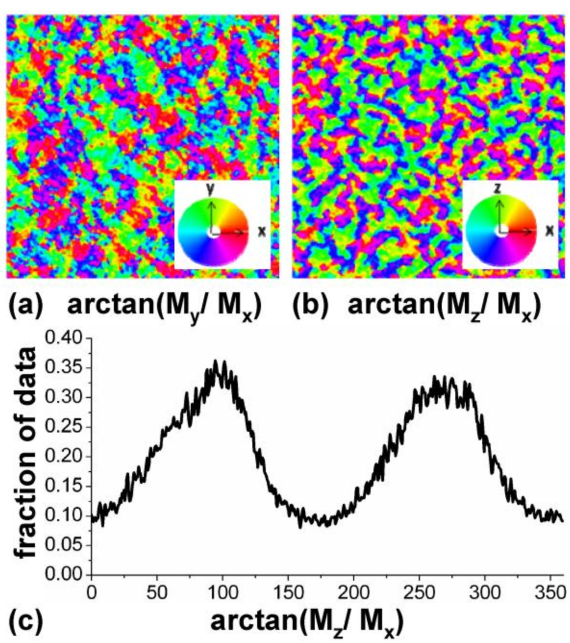

FIG. 2. (Color online) Images of (a) the in-plane and (b) out-of-plane magnetization angle are calculated from magnetization component images (Fig. 1). (c) A histogram of image (b) shows that the magnetic domains point mostly out of plane.

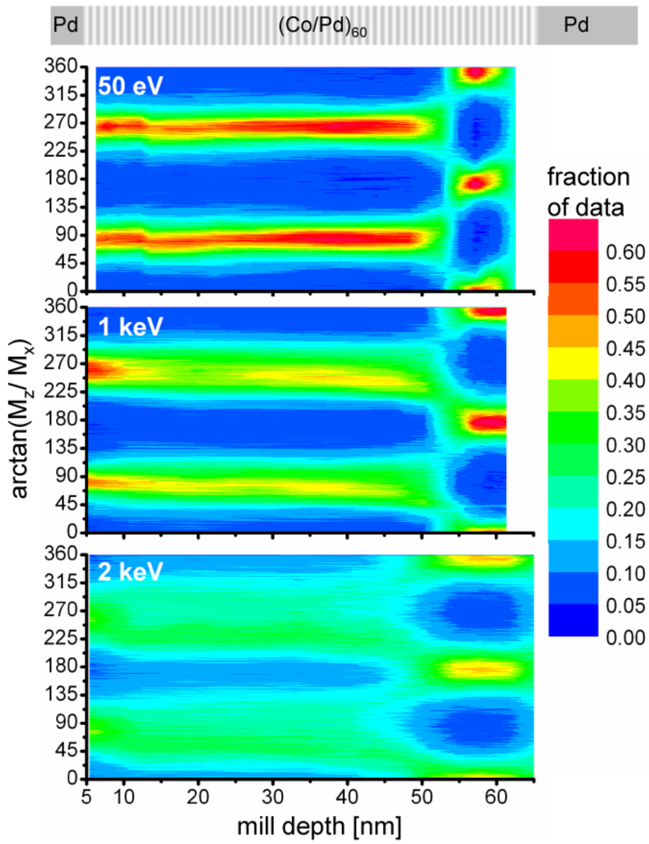

FIG. 3. (Color online) Magnetization orientation as a function of mill depth using 50 eV (top plot), 1 keV (middle), and 2 keV Ar ions (bottom plot). A schematic of the multilayer is shown at the very top to guide the eye. These contour plots are composed of histograms of the magnetic polarization angle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