Effect of spacer material on the magnetic surface anisotropy in ultrathin Fe$_{70}$B$_{30}$ multilayer films

R. J. Hickon, G. T. Rado, and C. L. Chien
Department of Physics and Astronomy, The Johns Hopkins University, Baltimore, Maryland 21218

It has been found recently that the magnetic surface anisotropy $K_s$ in Fe$_{70}$B$_{30}$/Ag multilayer films decreases monotonically with magnetic layer thickness ($2L$) for $2L < 16.5$ Å. In order to determine possible effects of the spacer material on the surface anisotropy in the aforementioned system, Ag has been replaced with Al$_2$O$_3$ and ferromagnetic resonance (FMR) measurements have been made on these films. These Fe$_{70}$B$_{30}$/Al$_2$O$_3$ films were fabricated by magnetron sputtering and were characterized by x-ray-diffraction and vibrating sample magnetometer (VSM) measurements in addition to FMR. In the region where $K_s$ depends upon $2L$, the data is insufficient to confirm the thickness dependence of $K_s$ that was observed in Fe$_{70}$B$_{30}$/Ag, while in the region where $K_s$ is independent of $2L$, the values of $K_s$ deduced for Fe$_{70}$B$_{30}$/Ag and Fe$_{70}$B$_{30}$/Al$_2$O$_3$ are in good agreement. The latter is particularly interesting in light of the enormous difference in conductivity between Ag and Al$_2$O$_3$.

We recently reported$^1$ the observation of a thickness-dependent magnetic surface anisotropy constant $K_s$ in amorphous Fe$_{70}$B$_{30}$/Ag multilayer films. When the Fe$_{70}$B$_{30}$ layer thickness $2L$ was greater than 16.5 Å a constant value of 0.34 erg/cm$^2$ was observed, while for $2L < 16.5$ Å, the value of $K_s$ was observed to decrease monotonically. This led us to speculate that the magnetic surface anisotropy mechanism may be different in amorphous Fe$_{70}$B$_{30}$ than in crystalline materials where such a dramatic thickness dependence of $K_s$ has not been observed. Experiments and theoretical investigations have shown$^2$ that a particular crystal surface may possess a different surface anisotropy constant depending upon what material is used to cover or support it. It is interesting, therefore, to change the spacer material in our Fe$_{70}$B$_{30}$ multilayer films and determine whether this affects the magnetic surface anisotropy constant as in crystalline materials. We have chosen Al$_2$O$_3$, an insulator, as an alternative to Ag as a spacer layer. The very low conductivity of Al$_2$O$_3$ allows maximum contrast with Ag and also partially alleviates the limitation placed on the total thickness of our multilayers by the skin effect during microwave absorption experiments. It is also expected that Al$_2$O$_3$ will offer excellent protection for the Fe$_{70}$B$_{30}$ against oxidation.

The Fe$_{70}$B$_{30}$/Al$_2$O$_3$ films were fabricated by magnetron sputtering, the Fe$_{70}$B$_{30}$ being dc sputtered while the Al$_2$O$_3$ was rf sputtered. The pressure in the chamber prior to sputtering was in the range 4-14 × 10$^{-7}$ Torr, with an argon pressure of 4 × 10$^{-3}$ Torr being used for the actual sputtering. The films were deposited onto Kapton and glass substrates at room temperature. The substrate holder and shutter were computer controlled and were preprogrammed for each sputtering run. The number of bilayers deposited varied between 20 and 100 for the different runs and in each case the sputtering rate of the Fe$_{70}$B$_{30}$ was monitored with a crystal oscillator that was calibrated by making surface profilometer measurements on a specially grown thick Fe$_{70}$B$_{30}$ film. The Al$_2$O$_3$ layer was made identically in all the runs while the Fe$_{70}$B$_{30}$ thickness $2L$ was varied.

Low-angle x-ray-diffraction measurements were made on the multilayers deposited on glass. The spectra are shown in Fig. 1 and suggest a good periodic layer structure in all the samples. The spectrum for which $2L = 16.3$ Å is not as clean as the others and suggests some small variation of the bilayer period through the sample. It can be seen that in each spectrum there is an envelope that determines the relative intensity of different order peaks. The shape of the envelope, which is characteristic of a single bilayer, depends upon the thicknesses of the Fe$_{70}$B$_{30}$ and Al$_2$O$_3$ layers and their respective scattering factors. By measuring the separation of successive peaks in a spectrum $\delta(2\theta)$, one may determine the bilayer period of the sample with the formula $d = \lambda/2\delta(2\theta)$, where $\lambda = 1.5406$ Å is the wavelength of the x rays. Since the Al$_2$O$_3$ thickness was the same for all these samples, by plotting the bilayer period against the crystal oscillator value of $2L$ and measuring the intercept and slope, one can check the value of the Al$_2$O$_3$ thickness (20 Å) and the calibration of $2L$. High-angle x-ray diffraction confirmed that both the Fe$_{70}$B$_{30}$ and the Al$_2$O$_3$ were indeed amorphous.

Vibrating sample magnetometer (VSM) measurements were made at room temperature on the samples deposited on Kapton substrates. The thicknesses studied were again $2L = 2.6, 6.3, 16.3, 20.9, 54.2,$ and 106 Å. From Fig. 2(a) it can be seen that even with the maximum available field of 14 kOe applied in the plane of the sample, it was impossible to saturate the 2.6- and 6.3-Å samples. The other four samples did saturate, however, and the room-temperature saturation magnetization was determined to be 793, 1121, 1018, and 1061 emu/cm$^3$ for the $2L = 16.3$, 20.4, 54.2, and 106-Å samples, respectively. In Ref. 1 the analytical form $M(T) = [942 - 434 \times (1/2L)]$ emu/cm$^3$ for $2L > 6.8$ Å and $M(T=300 K) = [594 - 343 \times (1/2L)]$ emu/cm$^3$ for $2L < 6.8$ Å was determined for Fe$_{70}$B$_{30}$/Ag. It can be seen that our measured values for Fe$_{70}$B$_{30}$/Al$_2$O$_3$ all lie within...
20% of those determined for Fe$_{70}$B$_{30}$/Ag. The discrepancy can be accounted for by a large experimental error resulting from, first, the difficulty in determining the volume of the samples and, second, the small moments (of the order of 10$^{-3}$ emu) of the samples. Magnetization curves with the magnetic field applied perpendicular to the plane of the sample are shown in Fig. 2(b). Again the 2$L = 2.6$- and 6.3-Å samples cannot be saturated but the 2$L = 16.3$- and 20.9-Å samples do exhibit a clear saturation or “knee” field, $H_{\text{knee}}$ less than 14 kOe. We may write $H_{\text{knee}} = 4\pi M - (4K_M/2L)$. Using the analytical form for $M$ from Ref. 1, we calculate $K_M = 0.32$ and 0.31 erg/cm$^2$ for 2$L = 16.3$ and 20.9 Å, respectively. Both these values are within 10% of the value of 0.34 erg/cm$^2$ determined for Fe$_{70}$B$_{30}$/Ag.$^1$ The curves for the 2$L = 56.4$- and 106-Å samples are more rounded, saturation being barely achieved with the maximum applied field. An accurate value of $K_M$ cannot be determined for these samples.

Ferromagnetic resonance (FMR) measurements were performed on the samples deposited on the Kapton substrates at 9.52 GHz with the applied field both parallel and perpendicular to the plane of the sample and at 23.9 GHz with the applied field in the plane of the sample. The resonance fields are plotted as a function of $1/2L$ in Fig. 3. The solid curves are the best fit curves for the Fe$_{70}$B$_{30}$/Ag system.$^1$ These were calculated from the theory of Zhang and Rado$^5$ and assume analytical forms for $M(T = 300$ K) and $K_M$ as a function of $2L$.$^1$ It can be seen that they agree well with the Fe$_{70}$B$_{30}$/Al$_2$O$_3$ data for $2L > 10$ Å. For $2L < 10$ Å, the linewidths of the resonances become very large and both the parallel and perpendicular resonance fields converge towards $\omega/\gamma$, where $\omega$ is the circular frequency and $\gamma = 18.38$ MHz/Oe. If we were to calculate $K_M$ for these resonance fields we would obtain values that decrease monotonically with $2L$. However, when we recall that in the VSM study it was impossible to saturate the samples for which $2L < 10$ Å, it seems likely that the magnetic layers in these films are not completely continuous for $2L < 10$ Å although the low-angle x-ray data suggests that there is still a clear periodic composition modulation. That is, the Fe$_{70}$B$_{30}$ must still reside in well-defined planes, but need not occupy the entire area of each plane. We must stress that this is in marked contrast to Fe$_{70}$B$_{30}$/Ag where linewidths remained small$^6$ and saturation was always achieved$^6$ for even the smallest values of $2L$. Of course, if the magnetic layers are not continuous and the infinite thin-film geometry is lost then $K_M$ cannot be determined.
In summary, we have fabricated Fe$_{70}$B$_{30}$/Al$_2$O$_3$ multilayer films with magnetic layer thicknesses in the range 2.6 Å < $2L$ < 106 Å. For $2L$ < 10 Å the magnetic layers appear not to be continuous and so the behavior of $K_s$ cannot be determined there. For $2L$ > 10 Å, both VSM and FMR experiments are consistent with the values of $M$ and $K_s$ determined for the Fe$_{70}$B$_{30}$/Ag system. For the region 10 Å < $2L$ < 16.5 Å the data is insufficient to confirm the thickness dependence of $K_s$ that was observed previously. However for $2L$ > 16.5 Å a constant value of $K_s = 0.34$ erg/cm$^2$ accounts well for FMR measurements on both the Fe$_{70}$B$_{30}$/Al$_2$O$_3$ and Fe$_{70}$B$_{30}$/Ag systems. In light of the vast difference in conductivity between the two spacer materials and the fact that they appear to affect the growth of continuous Fe$_{70}$B$_{30}$ layers differently, this is somewhat surprising. Neither this insensitivity of $K_s$ to the spacer material nor the strong thickness dependence of $K_s$ found in Fe$_{70}$B$_{30}$/Ag have been found in crystalline materials. This again suggests that the surface anisotropy mechanism in amorphous and crystalline materials may indeed be of a different nature.

This work was supported in part by National Science Foundation Grant No. DMR 88-22559 and by the U.S. Naval Research Laboratory.
