Perpendicular anisotropy in Fe/Ag multilayers

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[Ag(2.6 nm)/Fe(x nm)]10 multilayers (0.2 ≤ x ≤ 1) have been prepared on Si(111) substrate by vacuum evaporation of 57Fe enriched iron from a heated tungsten crucible in a base pressure of 10⁻⁷ Pa. Ag was deposited by electron beam from a cold copper crucible. For ex-situ transmission Mössbauer spectroscopy measurements the samples were capped with a thick capping layer (55 nm Ag and 100 nm B). The deposited layers were removed from the substrate by using scotch tape. Field cooled (FC) and zero field cooled (ZFC) magnetization curves were measured by a superconducting quantum interference device (SQUID) magnetometer.

The samples show superparamagnetic behavior in the whole Fe thickness range. This is indicated by the deviation of the FC and ZFC magnetization curves, as shown in Fig. 1 for a few representative samples. The blocking temperature, as defined by the temperature of the maximum in the ZFC magnetization, is 34 K for x = 0.2 (well reproducing the value of Ref. [2]) and rapidly increases with increasing x until x = 0.5. The broadening of the distribution of the size of the superparamagnetic grains with increasing x can also be inferred from the ZFC curves. Samples with x ≥ 0.6 are also superparamagnetic, as it is indicated by in-field Mössbauer measurements not shown here, but for the majority of the Fe atoms the blocking temperature (T_B) is well above room temperature. The low temperature anomaly of the FC-ZFC curves of these samples (for x = 0.7 see Fig. 1) can be attributed to an about 10–20% of the Fe atoms forming smaller grains with T_B below room temperature.

The direction of the spontaneous magnetization changes from in plane to out of plane at around x = 0.6 in [Ag(2.6 nm)/Fe(x nm)]10 multilayers (0.2 ≤ x ≤ 1) prepared on Si(111) substrate by vacuum evaporation. Transmission Mössbauer spectroscopy measurements of removed samples with a thick capping layer are compared to conversion electron Mössbauer spectroscopy measurements of samples on the Si substrate with a thin capping layer. The stress arising because of the application of a thick capping layer and the removal of the samples from the substrate is shown to have negligible effect on the spontaneous magnetization. The results support that the appearance of the perpendicular anisotropy below x = 0.6 is an intrinsic property of Fe/Ag multilayers.
Figure 1: Temperature dependence of the magnetization of the $x = 0.4$ (diamonds), 0.5 (triangles), and 0.7 (squares) samples measured in 10 Oe magnetic field after cooling the sample in zero (full symbols) and 10 Oe (open symbols) applied field. Note the smaller magnetization scale for $x = 0.4$ and 0.5.

Since the magnetic anisotropy might also be influenced by extrinsic stress, we examined the dependence of the direction of the spontaneous magnetization on the temperature, on the thickness of the capping layer and on the sample removal. For the latter two purposes in the case of $x = 0.4$ and 0.7 two identical multilayer samples have been prepared in the same run. One of them was covered with a thick capping layer (55 nm Ag and 100 nm B) and the other one with a thin capping layer (5 nm Ag). The samples with thin capping layers were examined by conversion electron Mössbauer spectroscopy (CEMS) at 80 K and 300 K.

Mössbauer spectra of the as received samples with thin capping layer (these samples were not removed from their substrates) and of the removed samples with thick capping layer are shown in Figs. 2 and 3 for $x = 0.4$ and $x = 0.7$, respectively, measured at two different temperatures. The superparamagnetic blocking temperature is around 150 K for $x = 0.4$ and above room temperature for $x = 0.7$. For this reason the Mössbauer spectrum of the $x = 0.7$ sample exhibits magnetic splitting at 80 K and 300 K, while the sample of the $x = 0.4$ consists of paramagnetic lines at 300 K. The CEMS and the transmission spectra of samples differing in the capping layer thickness agree very well and show a similar distribution of the hyperfine parameters. Small differences of the 80 K spectra around the zero velocity arise from Fe impurities in the beryllium window of the cryostat used for the transmission measurements. The average hyperfine splitting, the width of the distribution and the average isomer shifts values were calculated by the NORMOS program and are deployed in Table 1. In measurements at elevated temperatures the distribution of the
The intensity of the 2nd and 5th lines of the magnetic sextet with respect to that of the 3rd and 5th lines is
\[ I_2 = 4 \sin^2 \theta/(1 + \cos^2 \theta). \] The \( \gamma \)-ray directions are perpendicular to the sample planes both for the CEMS and the transmission measurements. In the case of a hyperfine field distribution an average value of \( I_{25} \) can be given by supposing a uniform direction of all the magnetic moments. \( I_{25} = 0.9 \) and
\[ x = 0.4 \text{ and } 3.8 \text{ at } 80 \text{ K (see Table 1)} \] indicates that the average magnetization direction is close to perpendicular and in plane (\( \theta = 36^\circ \) and \( 80^\circ \)) for \( x = 0.4 \) and 0.7, respectively, for the CEMS and the transmission spectra as well. Stresses due to the applied capping layer and the sample removal procedure do not affect the direction of the spontaneous magnetization.

The \( I_{25} \) amplitudes determined by transmission measurements in a broad temperature range below \( T_N \) for several samples are shown in Fig. 4. It should be noted that for each sample the spectra were evaluated as a sum of magnetic and non-magnetic components, the ratio of latter gradually decreasing with decreasing temperature. For samples of \( x = 0.4, 0.5, \) and 0.7, a slight decrease of the average \( I_{25} \) of the magnetic components can be observed with decreasing temperature. It hardly exceeds the uncertainty arising from the correlation of line width and amplitude values and might be due to stress induced by heat dilatations of the different layers. Nevertheless, the observed temperature dependence cannot explain the rather sharp change in the anisotropy direction as a function of layer thickness. The largest temperature variation of \( I_{25} \) is observed below 150 K for \( x = 0.6 \), which is probably close to the width where the crossover between the anisotropy directions occurs and thus the most sensitive to thickness fluctuations of the layers.

In conclusion, these studies undoubtedly show that the appearance of the perpendicular anisotropy at around \( x = 0.6 \) is an intrinsic property of our Fe/Ag multilayers. Preliminary results on granular alloys of similar Fe concentration and blocking temperature prepared by co-evaporation of the elements indicate that the out of plane spontaneous magnetization is related to the layered structure. The observed epitaxial growth of the layers [6] is suggested to explain the phenomena.

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**References**