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Thickness dependence of the magnetic and electrical properties of Fe:SiO₂ nanocomposite films

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Nanocomposite Fe₈₀(SiO₂)₂₀ films with thickness from 150 to 5000 Å have been prepared by rf magnetron sputtering from a composite target. The crystallites in the Fe₈₀(SiO₂)₂₀ films have a bcc structure with the average size of 46–66 Å which was determined by transmission electron microscopy. As indicated by the thickness dependence of resistivity, the stacking and connectivity of the crystallites depend on the thickness of the films. The magnetic properties also depend on the microstructure which changes with the thickness of the films. The magnetic coercivity of the films increases with the thickness of the film, reaches a maximum, and then decreases. The maximum coercivity of 400 Oe at 300 K and 1200 Oe at 5 K was observed for a film with a thickness of about 700 Å.

I. INTRODUCTION

Nanocomposite materials have the form of small crystallites dispersed in a matrix which may be insulating or metallic. The magnetic properties of nanocomposite materials depend on the microstructure of the films which can be controlled by either changing the size of crystallites and/or the separation distance between the crystallites. It has been shown that the crystallites size and the intercrystallite distance of the nanocomposite Fe:SiO₂ system can be systematically changed by varying the Fe volume fraction.^{1–6} The average crystallite size in the films for a fixed volume fraction can also be varied by changing the substrate temperature.⁷ For the films with lower metal volume fraction the crystallites are isolated whereas for the films with higher metal volume fraction the crystallites are well connected.⁸ For the Fe:SiO₂ films with a Fe volume fraction near 50% the crystallites begin to form a percolating network. As the thickness of the film is decreased, the number of crystallites connecting together in the film-normal direction decreases and a change in the electrical and magnetic properties is expected. In this work we investigated the thickness dependence of magnetic and electrical properties of the Fe:SiO₂ films.

II. EXPERIMENTAL PROCEDURE

The nanocomposite Fe:SiO₂ films were prepared by rf magnetron sputtering from a composite target. The sputtering targets were prepared by sintering a mixture of pressed Fe and SiO₂ powders. The sputtering gas was 5 mTorr of Ar, and the films were deposited on glass substrates at room temperature. The films used in this study have a target composition of Fe₈₀(SiO₂)₂₀ (55 vol. % of Fe). The film thickness ranged from 150 to 5000 Å. The microstructure of the films was studied by using a JEOL 2010 high-resolution transmis-

sion electron microscope (HRTEM) and electron diffraction. The magnetization and the coercive force were measured using a commercial alternating-gradient force magnetometer and a SQUID magnetometer. The resistivity was measured in a standard four-point probe configuration.

III. RESULTS AND DISCUSSION

The electron-diffraction pattern of the Fe₈₀(SiO₂)₂₀ films with a thickness of 700 Å is shown in Fig. 1. The electron-

FIG. 1. Electron-diffraction pattern of Fe₈₀(SiO₂)₂₀ film with a thickness of 700 Å.

FIG. 2. HRTEM image for the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ film with a thickness of 700 Å.

diffraction pattern of the films show a α -Fe structure with the (110), (200), (211), etc. rings present. The d spacing of the (110) peak as deduced from x-ray diffraction is 2.03 Å, which is close to the lattice parameter of α -Fe. The electron-diffraction pattern of the film can be indexed to α -Fe and there is no evidence of contribution from any Fe oxide phases; this is also confirmed by Mössbauer spectroscopy. The hyperfine field as calculated from the Mössbauer spectra is 330 kOe, which is close to the value for α -Fe. The morphology of the crystallites as revealed by the HRTEM image for the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films with a thickness of 700 Å is shown in Fig. 2. The average particle size of the Fe crystallites is 46–66 Å and the Fe crystallites are mostly isolated in an amorphous SiO_2 matrix. The Fe crystallites have a clear boundary with the SiO_2 matrix. The fringes on the crystallites represent the lattice spacing of Fe.

The thickness dependence of the resistivity for the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films is shown in Fig. 3. It is seen that the resistivity decreases as the film thickness increases and above 700 Å the resistivity decreases drastically, indicating that the films form a better connecting network of Fe crystallites. The plane view HRTEM image shows that the Fe crystallites are isolated in the SiO_2 matrix, so the drastic change in resistivity for the films with thickness above 700 Å

FIG. 3. Resistivity vs thickness for the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films.

FIG. 4. Magnetic coercivities measured at 300 and 5 K vs thickness for the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films.

may be likely due to the change in stacking and connection of the Fe crystallites. The inset of Fig. 3 shows the resistivity versus film thickness for 700–4273 Å thick films. As the film thickness increases from 700 to 4273 Å, the resistivity changes from 5.6×10^{-2} to $1.8 \times 10^{-4} \Omega \text{ cm}$.

The magnetic coercivity measured at 300 and 5 K of the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films versus thickness is shown in Fig. 4. The magnetic properties depend on the thickness of the film. It is observed that as the film thickness increases the magnetic coercivity increases, goes through a maximum, and then decreases. The maximum coercivity of about 400 Oe at 300 K and 1200 Oe at 5 K was obtained for a film with a thickness of about 700 Å. As the film thickness increases, the change in magnetic coercivity measured at 5 K is more pronounced compared to the magnetic coercivity measured at 300 K for the films with thicknesses less than 1200 Å.

Figure 5 shows the dependence of magnetic coercivity with temperature for $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films with different thicknesses. The magnetic coercivity decreases as the temperature

FIG. 5. Magnetic coercivities vs temperature of the $\text{Fe}_{80}(\text{SiO}_2)_{20}$ films with different thicknesses.

increases. The temperature dependence of coercivity with a film thickness more than 1000 Å is almost linear, whereas the film with a thickness of 285 Å has a $T^{1/2}$ dependence. The $T^{1/2}$ dependence of coercivity for a thin film is characteristic of a system of superparamagnetic particles below the blocking temperature.⁹ As the film thickness increases the number of connected crystallites also increases, i.e., the interaction between the crystallites increases and one observes a change in the magnetic properties of the films.

IV. SUMMARY

We have prepared Fe:SiO₂ nanocomposite films with different thicknesses. The size of the α -Fe crystallites for the Fe₈₀(SiO₂)₂₀ films ranges from 46 to 66 Å. The resistivity of the Fe₈₀(SiO₂)₂₀ films decreases with increasing film thickness. Above 700 Å the resistivity drops drastically, indicating that the crystallites form a better connecting network in the thick films. The magnetic coercivity for the Fe₈₀(SiO₂)₂₀ films depends on the microstructure which changes with the thickness of the films.

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