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Study of Exchange-Bias Effects for Antiferromagnetic-Nonmagnetic Nanocomposite Materials

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Abstract. We have investigated the magnetic properties of $[MnO]_x[MoO_{2+\delta}]_{1-x}$ ($0.5 \le x \le 0.7$) composite films, which have a structure that comprises randomly oriented MnO nanocrystallites of about 10 nm diameter. After field cooling through a critical temperature T_c, these films exhibit exchange-bias effects. This phenomena is attributable to the pinning effect of uncompensated magnetic moments at the surface of MnO nanocrystallites by the antiferromagnetic (AF) domain walls in the core. The formation energy of the AF domain wall for the film $x = 0.7$ is larger than for the film with $x = 0.5$.

Keywords: antiferromagnetic domain wall, exchange-bias, manganese oxide, molybdenum dioxide, nanocrystallite

The nature of magnetism in nanosized composite materials has become a very active area of research because of their unique magnetic properties as well as their technological applications. We have reported on the structural and magnetic properties of $[MnO]_x[MoO_{2+\delta}]_{1-x}$ ($0 \le x \le 1$) composite films.[1,2] The film structures depend markedly on the Mn contents; the films with $x \le 0.1$, $0.2 \le x \le 0.4$, and $0.5 \le x \le 0.7$ have MoO_2 polycrystalline structures, $MoO_{2+\delta}$ and MnO amorphous structures, and MnO nanocrystallites, respectively. X-ray diffraction (XRD) analysis and high-resolution transmission electron microscopy (HRTEM) show that the films with $0.5 \le x \le 0.7$ have a structure that comprises randomly oriented MnO nanocrystallites of about 10 nm diameter in the Mn-Mo-O complexes. These nanocrystallite films show so-called mixed-magnetic behavior in the temperature dependence of their magnetization. Magnetization curves of these films exhibit a hysteresis loop below a critical temperature T_c.

Recently, we have shown that the film with $x = 0.5$ exhibits an exchange-bias effects,[3] *i.e.* a loop shift $|H_E|$ (exchange-bias field) and coercivity H_C (the half width of the loop at $M = 0$) enhancement, after field cooling through T_c. In the present report, we have explored the magnetic field cooling effects of the $[MnO]_x[MoO_{2+\delta}]_{1-x}$ ($x = 0.7$) composite film which is a higher Mn content than the film with $x = 0.5$, for the purpose of investigating the magnetic interaction between uncompensated magnetic moments at the surface of the MnO nanocrystallite and the antiferromagnetic (AF) moments of the core.

$[MnO]_x[MoO_{2+\delta}]_{1-x}$ ($0.5 \le x \le 0.7$) composite films, which had typical nanocrystalline features, were prepared using a magnetron sputtering system enhanced with an inductively coupled rf plasma (MPS-HC3; ULVAC Technologies, Inc.).[1, 2] Measurements of magnetization M as a function of magnetic field H for the composite films were carried out using a SQUID magnetometer (MPMS *XL*-7; Quantum Design). Magnetization curves (*M-H*) at 2 K were measured in magnetic fields up to 70 kOe. Magnetic fields were applied parallel to the in-plane direction of the film. For the zero-field-cooled (ZFC) case, the sample was cooled to 2 K in zero field. For the field-cooled (FC) case, cooling was performed from 330 K to a measured temperature in a cooling field H_{FC}; the cooling rate was 1 K/min.

Figure 1 shows the *M-H* curves for the film with $x = 0.7$ at $T = 2$ K; solid- and open-circles correspond to the ZFC and the FC ($H_{FC} = 70$ kOe) condition, respectively. The *M-H* curve under the ZFC condition (solid-circle), appears to be almost linear; it shows a very small hysteresis loop. Under the FC condition (open-circle), an asymmetric magnetization curve shift from the origin and increased magnetic susceptibility are observed. Similar asymmetric magnetization curves have been observed in many exchange-bias systems such as Co-CoO particles,[4] ferromagnetic/antiferromagnetic bi- or multi-layer,[5] and antiferromagnetic NiO[6] nanoparticle systems. Exchange-bias is one of the phenomena associated with the inter-

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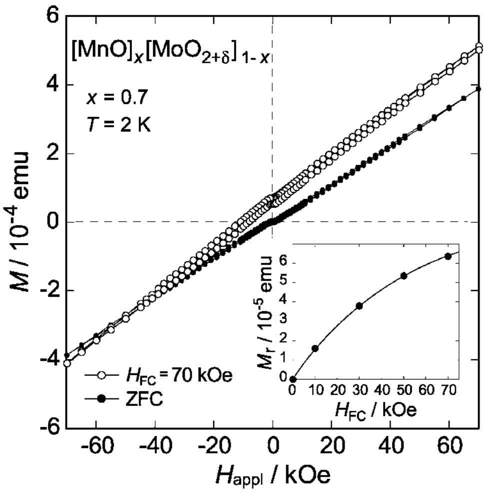

FIGURE 1. *M-H* curve for field-cooled $[MnO]_x[MoO_{2+\delta}]_{1-x}$ (x = 0.7) composite film. The inset shows the cooling field H_{FC} dependence of remanent magnetization M_r that is the average of the upper and lower intercepts on the M axis.

facial exchange coupling between ferromagnetic and antiferromagnetic spin structures.[4]

In our previous paper on the film with $x = 0.5$ we have pointed out that the AF domain walls have been formed in the MnO nanocrystallite core.[3] The AF domain structure is different in the magnetized and demagnetized processes. The difference of each domain formation energy is an origin of asymmetric *M-H* curves. We also attribute asymmetric *M-H* curves in Fig. 1 to the pinning of uncompensated magnetic moments at the surface of MnO nanocrystallites by the AF domain walls in the core. The concept of the AF domain wall model has been proposed by Mauri *et al.*.[7]

The inset in Fig. 1 shows the cooling field H_{FC} dependence of the remanent magnetization M_r that is the average of the upper and lower intercepts on the M axis. M_r increases with increasing H_{FC}. This result indicates that the surface moments of the MnO nanocrystallites frozen in the direction of H_{FC} increase with increasing H_{FC} after field cooling.

Figure 2 shows the comparison of the absolute value of the exchange-bias field $|H_E|$ plotted against T / T_c for the film with $x = 0.5$ (open-circles and dashed line) and $x = 0.7$ (solid-circles and line); the T_c values for the films with $x = 0.5$ and 0.7 are 24 K and 77 K, respectively. The $|H_E|$ for both of these films decrease rapidly with increasing temperature. The $|H_E|$ values of the $x = 0.7$ film are larger than those of the $x = 0.5$ film and vanish at higher temperature. These results indicate that the formation energy of the AF domain wall for the film with $x = 0.7$ is larger than for the film with $x = 0.5$. We assume that the formation energy of the AF domain wall is dependent on the volume of the MnO nanocrystallites. From XRD analysis using the

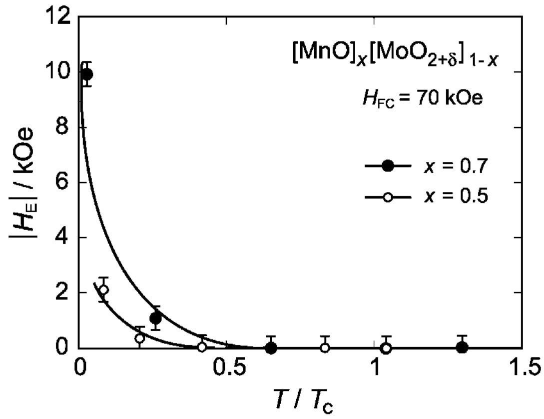

FIGURE 2. Temperature dependences of $|H_E|$ as measured at H_{FC} = 70 kOe for $[MnO]_x[MoO_{2+\delta}]_{1-x}$ (x = 0.5, x = 0.7) composite films.

Scherrer's equation, the size of MnO nanocrystallites, which is about 10 nm diameter, does not depend on Mn contents. Thus, it is expected that the number density of MnO nanocrystallites in $[MnO]_x[MoO_{2+\delta}]_{1-x}$ ($0.5 \le x \le 0.7$) composite films increases with increasing x. Thus, for the film with $x = 0.7$, it is necessary to consider the AF interaction between MnO nanocrystallites which are coupled magnetically to each other below T_c. It is also possible that the effective AF ordered region is larger than 10 nm leading to a large formation energy for an AF domain wall and a high critical temperature T_c for the film with $x = 0.7$.

In conclusion, we have measured magnetic field cooling effects for a $[MnO]_x[MoO_{2+\delta}]_{1-x}$ (x = 0.7) composite film. After field cooling through T_c, the observed asymmetric magnetization curve shifted from the origin is attributed to the pinning effect of uncompensated magnetic moments at the surface of MnO nanocrystallites by the AF domain walls in the core. The formation energy of the AF domain wall for the film with $x = 0.7$ is larger than for the film with $x = 0.5$.

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