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Exchange bias and magnetization process of Co/CoO nanocomposite thin films

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Abstract

Co/CoO nanocomposite thin films have been deposited by RF sputtering with different oxygen partial pressures in the working atmosphere. It has been found that the magnetic properties (exchange bias (EB), coercivity and remanence) of the films are strongly dependent on the oxygen partial pressure. Film deposited with an O₂ pressure of 0.75×10^{-5} Torr shows a relatively high coercivity and high remanence ratio. Enhanced asymmetry of magnetization reversal appears in the film deposited with 1.5×10^{-5} Torr O₂ partial pressure. When the O₂ partial

pressure is increased to 3.0×10^{-5} Torr, a strong reduction in EB and coercivity have been obtained. The investigation of the microstructure using high-resolution transmission electron microscope reveals that the change in EB, remanence and coercivity is probably related to the competition between dipolar interaction and exchange coupling. Magnetoresistance and extraordinary Hall effect (EHE) of the Co/CoO nanocomposite films is also investigated in dependence on oxygen partial pressure. The film deposited under 2.3×10^{-5} Torr of oxygen exhibits strongly enhanced asymmetric magnetization reversal and unusually high EHE with a high coercivity.

Keywords: CoO/Co nanocomposite; Exchange bias; Asymmetric reversal; Magnetoresistance; Extraordinary Hall effect

1. Introduction

Exchange bias (EB), which was firstly found in the oxidized Co fine powders [1,2], refers to a shift (H_E) of hysteresis loop along field axis after field cooling (FC), resulting from the interaction between ferromagnet (F) and antiferromagnet (AF). This phenomenon has attracted extensive interest in research because of its potential applications in recording media, read heads, magnetic sensors and magnetoelectronic switching devices (spin valves) [3–6]. Several theoretical models have been proposed to explain the possible mechanisms [7–12]. Many phenomena related to EB have been investigated. For instance, the relationship between EB and its transport properties was analyzed by Hasegawa et al. [13]. Recently, the size dependence of F and AF has been extensively studied, driven by the interest in nanoscale structures [14–16].

With a reduction of the lateral size of F and AF, there is an increasing importance of magnetostatic interaction. It is known that coercivity and remanence of hysteresis loop of an F are strongly dependent on the magnetostatic interaction status within F. By controlling the structure or lateral size of F/AF, the parameters, such as EB, coercivity and remanence, can be tailored [15,16]. Hence, the investigation of the effect of dipolar interaction in F/AF systems would be of importance to understand the relationship between structure and these magnetic properties.

In this work, by controlling sputtering conditions and the argon-to-oxygen ratio, Co/CoO nanocomposite films with different ratios of Co and CoO were deposited. We report the structures and properties of the nanocomposite films after sputtering with oxygen partial pressures of 0, 0.75×10^{-5} , 1.5×10^{-5} , 2.3×10^{-5} and 3×10^{-5} Torr in the working atmosphere. High-resolution transmission electron microscopic (HRTEM) study reveals the possible mechanisms of the different features of EB. Magnetoresistance (MR) and extraordinary Hall effect (EHE) are investigated.

2. Experimental procedure

Co/CoO nanocomposite films were fabricated using an RF sputtering system (Discovery 18, Denton Vacuum) from a cobalt target of the purity of 99.97%. The base pressure was 2.5×10^{-6} Torr. The work pressure for the sputtering was 7.5×10^{-3} Torr. In order to obtain Co/CoO nanocomposite films, the working atmosphere had a variety of oxygen partial pressure levels by mixing a two-gas mixture of 5% O₂+95%

Ar (99.999%) with pure argon gas (99.999%). By carefully mixing the two gases with a digital flow meter, different oxygen partial pressures were obtained. The deposition power was 50W and deposition time was 800 s. All the films were deposited on the glass substrate with a size of 8×8 mm. The film thickness was approximately 50 nm, which was determined by means of scanning electron microscopy (Philips FEG-XL30) and surface profilometer (P-12, Tencor Instruments). The phases present in the Co/CoO nanocomposite films were characterized by means of X-ray diffraction (XRD) with a CuK_α radiation using a Philips PW-1729 diffractometer and of Raman spectroscopy using a Jobin Yvon Horiba LabRam HR800 Raman spectrometer. The hysteresis loops were determined using an Oxford Instruments maglab vibration sample magnetometer (VSM). A JEOL JEM3010 HRTEM was used to investigate the microstructure. The measurement of MR was carried out using the four-probe method. EHE was studied with the usual cross geometry. For the convenience of discussion, we denote the samples deposited with different oxygen partial pressures as S1, S2, S3, S4, S5, respectively as shown in Table 1.

3. Results and discussion

Fig. 1 shows the XRD spectra of the Co/CoO films deposited with different oxygen partial pressures from 0 to 3.0×10^{-5} Torr. The spectra were collected at room temperature. Film S1 shows a mixed structure of HCP and FCC-Co phases. With increasing oxygen pressure, the Co diffraction peaks become weaker and disappear in films S4 and S5, indicating the reduction of metallic Co. CoO phase is invisible in the XRD spectrum of S2. When the oxygen pressure increases to 1.5×10^{-5} Torr (S3), small and broad CoO (200) peak appears, showing the formation of

CoO phase. Three strongest peaks of the CoO phase appear clearly in the XRD spectrum of the film S5. Metallic Co phase cannot be found when the oxygen partial pressure is higher than 1.5×10^{-5} Torr. It is to note that Co_3O_4 phase cannot be found in the XRD spectra, which is consistent with a previous investigation [17].

Fig. 2 illustrates the Raman spectra of the Co/CoO films. The Raman spectrum of S1 shows no peaks, apparently due to the weak polarizability of metallic Co, implying that the sample is in pure metallic form. Peaks appear in the spectra of the films S2–S5. The three strong peaks at 462, 507, 667 cm^{-1} are the characteristic peaks of CoO. These measurements indicate that CoO is formed at O_2 partial pressure level as low as 0.75×10^{-5} Torr. This is in contradiction to the XRD measurements shown in Fig. 1. It is probably due to the small amount of CoO in the film sputtered with 0.75×10^{-5} Torr oxygen (S2). These observations imply that Raman spectroscopy is very sensitive in detecting small amount of oxides.

Fig. 3 shows the TEM micrographs of Co/CoO nanocomposite films. Micrograph (a) shows the structure of sample S2. This sample contains fine Co crystallites with a size of 4–5 nm. CoO crystallites appear as thin layers along the grain boundaries of metallic Co grains. Micrograph (c) shows the structure of film S5. In this sample, Co grains are totally isolated by CoO network. The average grain size of metallic Co is in the range of 5–8 nm. Micrograph (b) shows the structure of sample S4. This sample has a mixed structure between the cases shown in micrographs 3a and c. In some areas, Co particles appear to cluster together and interconnect with CoO appearing as thin layers along grain boundaries. In other areas, isolated Co (4–6 nm) particles are surrounded by relatively larger CoO

grains, as observed in Fig. 3c. S3 also shows an inhomogeneous structure similar to S4 (Fig. 3b). The major difference is that the amount of CoO grains is less than that in the S4.

Fig. 4a shows HRTEM micrograph of S2. Based on lattice analysis, most of the grains are identified as FCC-Co and HCP-Co, in good agreement with the XRD results shown in Fig. 1. The FCC-CoO phase is found in grain boundaries between Co grains. This structure is similar to the $\text{Co}_{\text{core}}/\text{CoO}_{\text{shell}}$ composite structure proposed by Meiklejohn and Bean [1]. As evident in Fig. 4b, relatively large CoO grains appear together with a few isolated Co grains. Samples S3 and S4 exhibit microstructures featuring a mixture of those shown in Fig. 4a and b, as observed in the TEM image in Fig. 3b.

The results presented above, including XRD, Raman spectrometry, TEM and HRTEM, indicate that small Co grains are formed with CoO layers along grain boundaries when the amount of CoO is low, e.g., S2. With increasing the amount of CoO, CoO tends to form relatively large grains and metallic Co grains are isolated.

Fig. 5 gives an illustration of VSM hysteresis loops of the Co/CoO nanocomposite films. The measurement was performed at 80K after a FC under 1.59×10^3 kA/m from room temperature (295 K). S1, which is of pure metallic Co, does not show any EB due to the lack of AF CoO. S2 shows a coercivity enhancement and a large shift of hysteresis loop with a high remanence ratio. When the partial pressure of oxygen is increased to 1.5×10^{-5} Torr (S3), the coercivity increases further. However, the remanence of the hysteresis loop decreases significantly. At the same time,

the magnetization behavior shows the enhanced asymmetry in magnetization reversal. For S4, the asymmetry of magnetization reversal becomes more significant. S5 possesses low values of coercivity and remanence. The coercivity of the film is smaller than that of as-deposited film without oxygen (S1).

For S2, metallic Co grains are connected with discontinuous CoO network present along the grain boundaries. Therefore, there are strong dipolar interactions between Co grains and also strong coupling between F Co and AF CoO. The relatively large EB, coercivity and remanence are due to the balance between dipolar interaction and exchange coupling. It is to note that this sample possesses a high remanence ratio of >90% and H_{c-} of approximately 2.39×10^2 kA/m. These properties are certainly interesting for magnetic recording media.

In contrast, S5 consists of isolated Co particles and relatively large CoO grains. The dipolar interaction between Co particles is expected to be weak. The relatively large sizes of CoO grains may lead to a significant reduction of EB in this sample. It is to note that the reduction of EB may also be attributed to spin configuration of the Co/CoO interfaces [18].

A complex magnetization behavior has been found in samples S3 and S4. As shown in Fig. 3b, some Co particles are connected to each other with thin CoO layers along their grain boundaries and some other Co particles are separated by relatively large CoO grains. The dipolar interaction between Co particles is expected to be reduced since the average number of Co neighbors is reduced. As reported by Girgis et al. [16], the reduction in magnetostatic interaction may result in an asymmetry of hysteresis loop

and a reduction of remanence. The asymmetry in magnetization reversal is evident in the hysteresis loop of S4, which may be due to the magnetization reversal changing from domain nucleation to domain wall motion [19]. In addition, some Co particles, although in small quantity, have been found to be isolated. These isolated Co particles may have an additional contribution in the reduction of coercivity and remanence, as observed in S5. The complex hysteresis of samples S3 and S4 may be attributed to the combination of two effects: asymmetry due to reduction of dipolar interaction and low coercivity together with low remanence of isolated Co particles. The physics needs to be further investigated.

Fig. 6 shows the hysteresis loop of S2 taken at 295K, with 1.59×10^3 kA/m FC at 80 and 4 K. The hysteresis loop measured at 295 K has a low coercivity since there is no exchange coupling between F and AF above Néel temperature of CoO. The remanence is quite high in this film. The remanence of the hysteresis loop measured at 80 K is approximately equal to the saturation magnetization. The left coercivity of the hysteresis, H_{c-} , increases to 2.39×10^2 kA/m, as discussed in the previous part. Compared with the hysteresis loop taken at 80 K, H_{c-} increased to 5.92×10^2 kA/m at 4K. The increase is due to the increase of magnetic anisotropy (here exchange coupling is shown by the enhanced EB). It should be noted that a clear asymmetry appears at 4K accompanied with a reduction of remanence. This behavior is possible due to the competition between dipolar interaction and exchange coupling since the dipolar interaction between metallic Co particles may not have a significant change from 80 to 4K. This work has shown that high coercivity and high remanence may be achieved in a crystallographic isotropic F/AF nanocomposite with a good balance between dipolar interaction and

exchange coupling. Remanence enhancement has been reported in nanocomposite magnets [20,21].

Fig. 7 shows the EB of the resultant Co/CoO films. S2 has an EB of 54kA/m at 80K. Then, EB decreases quickly with increasing temperature. Relatively large EB is observed in S3 and S4. As reported in Ref. [22], EB is strongly dependent on grain size. Low values of EB are found for S5 in the whole range of temperatures, confirming the low exchange coupling in this sample due to the isolation of the Co particles.

Fig. 8 shows the ratio of EB and coercivity (H_E/H_c) as a function of oxygen pressure, where H_c is defined as $H_c = (H_{c+} + H_{c-})/2$. H_{c+} and H_{c-} are coercivity values in the positive and negative field directions. It is well known that H_E/H_c is one of the important parameters in these materials. A large ratio is desirable for applications, such as recording media and spin valves. It is evident in Fig. 8 that H_E/H_c has a nearly linear relationship with oxygen partial pressure, indicating an increase in H_E/H_c with increasing amount of CoO.

Fig. 9 shows the dependence of magnetization and resistivity of the Co/CoO nanocomposite films on the oxygen partial pressure. It is seen that the magnetization is nearly linear to the oxygen partial pressure (Fig. 9b). Given the facts that CoO is antiferromagnetic below its Néel temperature (291 K) and that only Co contributes to the magnetization, the linear relationship indicates a constant rate of oxidation of Co with oxygen partial pressure. Fig. 9a shows the resistivity as a function of oxygen partial pressure. The resistivity increases with increasing oxygen partial pressure and it increases faster than the magnetization in dependence on oxygen

partial pressure. It is known that resistivity is attributed to metallic (Co) particles, conductivity of CoO and possible tunneling effect by overriding the oxide barrier. When CoO phase increases to a critical volume x_c , the resistivity is expected to increase fast as tunneling effect becomes dominant [23] since Co particles are gradually isolated by CoO crystals, as demonstrated in Fig. 3c.

MR is an important phenomenon in magnetic thin films. A shift of MR curve similar to EB has been observed in Co/CoO bilayer by Gredig et al. [24]. In this study, MR measurement was also carried out. The shift as observed in bilayer materials is expected to present in these composite films. Fig. 10 shows the MR effect at 295 and at 80K after FC and zero field cooling (ZFC) for the films with 0 and 1.5×10^{-5} Torr O_2 , respectively (S1 and S3). From the MR curves, it is seen that there is no shift in the films with ZFC at 80 and 295 K. The film with 1.59×10^3 kA/m FC shows an apparent shift which is similar to EB at 80K. Gredig et al. [24] have described the relation of the EB and MR. However, the “EB” of MR in our study does not agree with theirs. The graph also shows that there is a positive MR effect in the small magnetic field which is similar to pure Co films, but the amplitude is small [25]. The MR effect of S2, S4 and S5 was also measured. S2 and S4 have similar shape of MR curves to S3. For S5, there is no positive MR effect that is similar to that of certain composition of granular films [26].

EHE has attracted extensive investigation for its potential applications and interesting subjects of fundamental research [27]. Fig. 11 shows the EHE effect of films S1 and S4. The hysteresis loop measurement was carried out by sweeping the field between 1.59×10^3 and $-1.59 \times$

10³ kA/m. For S1, Hall resistivity ρ_H is small at room temperature and is further reduced at 80 K as EHE is temperature-dependent. The EHE hysteresis loop of S1 is soft as it is expected for a soft magnet. For S4, as shown in Fig. 11b, ρ_H is very large, in the order of 0.5–1 Ωm ($\times 10^{-6}$) and 100 times greater compared with the EHE effect of pure Co film (Fig. 11a). At 80K, EHE has a relatively large coercivity in both hysteresis loops after FC and ZFC. Fig. 11b also shows that EHE hysteresis loop is shifted toward lower ρ_H after an FC at $H = 1.59 \times 10^3$ kA/m. No shift is observed in the hysteresis loop after ZFC. The shift may be related to the inhomogeneous structure as observed in Figs. 3 and 4. It is to note that EHE is relatively small in S2, S3 and S5. It is known that Hall resistivity has two components: ordinary Hall effect and EHE. EHE can be much larger than ordinary Hall effect in magnetic films [28]. EHE has been found to be strongly dependent on the microstructure. Recently, large EHE has been reported in nanocomposite films, such as Ni/SiO₂ and Co/SiO₂ [28,29]. In this work, relatively high EHE was observed in F and AF Co/CoO nanocomposite (S3). The mechanism of EHE in nanocomposite films is not well understood. It may be associated with metal–insulator transition [29].

4. Conclusions

In conclusion, the structure, magnetic and transport properties of Co/CoO nanocomposite films deposited with different partial pressures of oxygen have been investigated. The microstructure and properties are strongly dependent on the oxygen pressure during deposition. The film with 0.75×10^{-5} Torr O₂ (S2) shows a relatively high coercivity and high remanence at 80K after FC, probably due to a good balance between

dipolar interaction and exchange coupling. A clear asymmetry of magnetization reversal has been observed in the film of 2.3×10^{-5} Torr O₂ (S4). The sample with asymmetry (S4) exhibits an unusually high EHE (large EHE effect, high coercivity and shifted hysteresis loop). Our structural and magnetic examinations support that the competition of dipolar interaction and exchange coupling is responsible for the asymmetry of magnetization reversal.

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- Fig. 11 EHE of S1 at 80 and 295K (a). S4 at 295K, with ZFC and FC at 80K (b).

Sample	Oxygen partial pressure (Torr) $\times 10^{-5}$	Working pressure (Torr) $\times 10^{-3}$	Sputtering time (s)	Sputtering power (W)
S1	0	7.5	800	50
S2	0.75	7.5	800	50
S3	1.5	7.5	800	50
S4	2.3	7.5	800	50
S5	3.0	7.5	800	50

We denote the five sample as S1, S2, S3, S4, S5 deposited with an oxygen partial pressure of 0, 0.75×10^{-5} , 1.5×10^{-5} , 2.3×10^{-5} and 3×10^{-5} Torr, respectively.

Table 1

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6

Fig. 7

Fig. 8

Fig. 9

Fig. 10

Fig. 11