Observation of Co/CoO nanoparticles below the critical size for exchange bias

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We compare the magnetic properties of pure and oxidized Co nanoparticles embedded in an amorphous Al_2O_3 matrix. Nanoparticles with diameters of 2 or 3 nm were prepared by alternate pulsed laser deposition in high vacuum conditions, and some of them were exposed to O_2 after production and before being embedded. The nanoparticles are organized in layers, the effective edge-to-edge in-depth separation being 5 or 10 nm. The lower saturation magnetizations per Co atom for the samples containing oxidized nanoparticles provide evidence for the formation of antiferromagnetic CoO shells in the nanoparticles. None of the samples with Co/CoO nanoparticles show exchange bias, while vertical hysteresis loop shifts and enhanced coercivities (as compared to samples with pure Co nanoparticles) are observed. This constitutes evidence for the nanoparticles size being in all cases smaller than the critical size for exchange bias. The difference in coercivity versus temperature dependences for the samples with pure and oxidized Co nanoparticles shows that the exchange anisotropy in Co/CoO nanoparticles appears at temperatures lower than 50 K. © 2007 American Institute of Physics. [DOI: [10.1063/1.2736303](https://doi.org/10.1063/1.2736303)]

I. INTRODUCTION

An exchange anisotropy appears in hybrid ferromagnetic (FM)-antiferromagnetic (AFM) systems when cooling down through the Néel temperature of the antiferromagnet.^{1–5} The anisotropy normally manifests as a horizontal shift of the magnetic hysteresis loop after field cooling. This shift (exchange bias) shows up due to an energy barrier, which appears at the FM-AFM interface after field cooling. The interfacial exchange interaction favors only one mutual orientation of the FM and the AFM spins. Therefore, the forward and backward FM spin reversal happens at different absolute values of the applied field, i.e., the hysteresis loop is biased.

Investigations of exchange anisotropy in systems of reduced dimensions may help to reveal the involved exchange mechanisms, provide insight in finite size effects,^{6–8} or shed light on interface effects.⁹ It is also very important for nanomagnetism applications. For example, it was shown that the exchange anisotropy stabilizes magnetic moments of FM nanoparticles, embedded in an AFM matrix,^{10,11} or agglomerated FM-core AFM-shell nanoparticles embedded in an amorphous matrix.¹² In these systems the stability of the FM magnetic moments is limited by the blocking temperature of exchange bias, rather than by the superparamagnetic block-

ing temperature of the FM nanoparticles.⁸ It was recently shown that for isolated FM-core AFM-shell nanoparticles there is a critical size, below which exchange bias does not exist.¹³ In this article we present a systematic study on the production and magnetic properties of pure Co and Co-core CoO-shell nanoparticles, embedded in an amorphous Al_2O_3 matrix as a function of nanoparticle diameter and edge-to-edge separation. The core-shell nanoparticles are zero-dimensional hybrid FM-AFM systems, and, therefore, allow studying the exchange anisotropy at the nanoscale.

II. SAMPLES PREPARATION AND CHARACTERIZATION

A. Pulsed laser deposition

The embedded Co nanoparticles were prepared by alternate pulsed laser deposition in high vacuum ($p < 10^{-7}$ mbar). An ArF excimer laser ($\lambda = 193$ nm, $\tau = 20$ ns full width at half maximum, repetition rate = 20 Hz, energy density = 1.9 J/cm^2) has sequentially been focused on high-purity Al_2O_3 and Co rotating targets. In order to prevent oxygen contamination, the Co target was ablated in vacuum during 30 s prior to deposition while having the substrates protected. The substrates were Si (100) wafers covered by a 700 nm thick amorphous SiO_2 layer, which were held at room temperature. The deposition sequence involved first the deposition of a 10 nm thick layer of amorphous (α -) Al_2O_3 .¹⁴ Subsequently, a layer of nanoparticles was produced followed by deposition of α - Al_2O_3 , this cycle being repeated

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FIG. 1. Cross-section schematic drawing of a sample. Five submonolayers of nanoparticles of diameter D are separated by layers of amorphous Al_2O_3 of thickness d .

five times. The deposition sequence is thus designed in order to have all nanoparticles nucleating on the same surface and all covered by protective $\alpha\text{-Al}_2\text{O}_3$ layers. A cross-section schematic drawing of the samples is shown in Fig. 1. Five layers of nanoparticles of diameter D are separated by an effective edge-to-edge in-depth distance d by layers of $\alpha\text{-Al}_2\text{O}_3$. The samples containing pure Co nanoparticles are designated as $dx Dy$, where x is the edge-to-edge nanoparticle in-depth separation, and y is the actual value of the nanoparticle diameter. Samples with edge-to-edge nanoparticle separations of 5 and 10 nm, and with nanoparticle diameters of 2 and 3 nm were produced.

The specimens containing oxidized nanoparticles are designated as $o dx Dy$, and were produced under identical conditions as the corresponding $dx Dy$ specimens except that the Co nanoparticles were exposed to oxygen for 20 s after production. The O_2 pressure was 7×10^{-4} mbar except for $o d5 D2$, which was higher (2×10^{-3} mbar). A summary of specimen features is included in Table I.

B. Transmission electron microscopy

A control specimen with Co nanoparticles of 3 nm diameter, having nanoparticles in layers 1–3 separated by 10 nm and in layers 3–5 separated by 5 nm, has been produced on Si substrates. The purpose was to determine the actual values from cross-section transmission electron microscopy (TEM) measurements. Figure 2(a) shows a TEM image of this speci-

FIG. 2. TEM images of control specimens having 3 nm diameter pure Co nanoparticles. (a) Cross-section image of specimen having nanoparticle layers separated 5 and 10 nm. (b) Plan-view image of sandwich film containing Co nanoparticles with expected diameter of 3 nm.

men, where it is clearly seen that the edge-to-edge in-depth separations of the nanoparticles correspond well to the intended values.

In order to determine the D value finally achieved, sandwich films containing a single nanoparticle layer embedded in $\alpha\text{-Al}_2\text{O}_3$ have been grown on carbon-coated mica substrates under the same conditions as the multilayer structures, both for pure Co and oxidized Co nanoparticles. TEM specimens have been straightforwardly prepared by floating the films off the substrate in de-ionized water and picking up on copper grids. Figure 2(b) shows a plan view of a sample containing pure Co nanoparticles with expected diameter of 3 nm. Since most of the nanoparticles are round and their dimensions are very similar in plan- and cross-section views, it can be concluded that the nanoparticles are spherical, they are well separated from each other, and their mean diameter is about 3 nm. Figures 3(a) and 3(b) show plan views of the sample with pure and oxidized 2 nm Co nanoparticles, respectively. While the exact structure of the oxidized nanoparticles is unknown, they can be considered as consisting of a pure Co core and a CoO shell (probably incomplete).

C. Rutherford backscattering spectrometry

Rutherford backscattering spectrometry using a 1.57 MeV He^+ beam was used to determine the Co content in the

TABLE I. Nanoparticle in-depth separation (d), nanoparticle diameter (D), and transient O_2 pressure (p), in the case of oxidized nanoparticles, relative to the samples considered in the present study.

Sample	d (nm)	D (nm)	p (mbar)
d5 D2	5	2	-
d10 D2	10	2	-
d5 D3	5	3	-
d10 D3	10	3	-
o d5 D2	5	2	2×10^{-3}
o d10 D2	10	2	7×10^{-4}
o d5 D3	5	3	7×10^{-4}
o d10 D3	10	3	7×10^{-4}

FIG. 3. Plan TEM images of control specimens with pure (a) and oxidized (b) Co nanoparticles with expected diameter of 2 nm.

samples, and to confirm the nanoparticles' in-depth separation. In the first case the detector was placed at an angle of 10° from the incident beam. The mean Co content of samples (o)dx D3 was 3.4×10^{16} atoms/cm², mean deviation -35% , while that of samples (o)dx D2 was 2.7×10^{16} atoms/cm², mean deviation -25% . In order to determine the in-depth nanoparticle separation the detector was placed at an angle of 75° from the incident beam. Using this geometry, the path of the detected backscattered He ions in the sample is larger than in the first case, which may allow the resolution of the different layers of the Co nanoparticles separated by the Al₂O₃ layers. Figure 4 shows RBS spectra for the samples (o)d10 Dy. The peaks corresponding to the individual layers of Co nanoparticles are observed for samples d10 D2, d10 D3, and o d10 D2. Such a "fine structure" also means that metal diffusion is not a significant process. The individual metal layers were only resolved for d10 Dy specimens, since for d5 Dy specimens the Co part of the spectrum was flat due to overlapping of neighboring peaks (because of the limited resolution of the spectrometer). Intensities of the individual Co peaks are nearly equal, thus indicating that the amount of Co in each layer of nanoparticles is approximately the same. Obviously, for the sample o d10 D2 the separation between first and second layers is larger than the separation between other layers, which is a production artifact.

III. MAGNETIZATION MEASUREMENTS

The magnetization measurements were performed in-plane with a vibrating sample magnetometer in the tempera-

FIG. 4. RBS spectra of samples d10 D2 (a), d10 D3 (b), o d10 D2 (c), o d10 D3 (d).

FIG. 5. (Color online) Hysteresis loops after zero field cooling from 300 to 5 K for the samples d5 D3 and o d5 D3 (a), d10 D3 and o d10 D3 (b), d5 D2 and o d5 D2 (c), d10 D2 and o d10 D2 (d). Magnetizations are normalized to the amount of Co in the samples; the saturation magnetization of the oxidized sample is taken as unity.

ture range from 5 to 300 K. In Fig. 5 zero field cooled to 5 K hysteresis loops for the samples d5 D3 and o d5 D3 (a), d10 D3 and o d10 D3 (b), d5 D2 and o d5 D2 (c), d10 D2 and o d10 D2 (d), are compared. The magnetization values are normalized according to the amount of Co in the samples. The lower saturation magnetization per Co atom in the samples with oxidized nanoparticles provides clear evidence for the formation of the AFM CoO shell. The higher coercivity of samples with oxidized particles is also related to the oxide shell, since normally hybrid FM-AFM systems show an enhancement of the coercivity, in addition to the unidirectional anisotropy.^{15–17} For all pairs of samples with oxidized and nonoxidized nanoparticles, the coercivity of the former at 5 K is significantly higher than that of the latter. For sample o d5 D2 the normalized saturation magnetization is the lowest, consistent with the larger AFM CoO phase due to the higher O₂ pressure used when oxidizing the nanoparticles in this case, as discussed in Sec. II and seen in Table I.

In Fig. 6 the coercivity versus temperature dependences for samples d10 D3 and o d10 D3 are compared. For the sample with oxidized nanoparticles, the coercivity is close to

FIG. 6. (Color online) Coercivity vs temperature for samples d10 D3 (pure) and o d10 D3 (oxidized). Lines are guides to the eye.

zero down to about 50 K, and with further decrease of temperature the coercivity increases abruptly. For the sample with nonoxidized nanoparticles, there is a gradual increase of the coercivity with temperature decrease. Figure 7 represents the coercivity versus temperature dependences for samples d5 D3 and o d5 D3, and they demonstrate a behavior similar to the latter case: the coercivity of the sample with oxidized nanoparticles increases rapidly at temperatures below 50 K.

Such a difference in the coercivity versus temperature behavior in the samples containing oxidized and nonoxidized Co nanoparticles may be attributed to the establishment of an exchange coupling at the Co/CoO interface at temperatures below 50 K. Although this temperature is much lower than the blocking temperature of exchange bias for “thick” Co/CoO bilayers (which is slightly lower than the Néel temperature of bulk CoO (291 K)),² in very thin AFM layers or nanoparticles the blocking temperature and the Néel temperature are known to be lower than those of the bulk counterparts.^{8,12,18,19} The lower value of the coercivity of the oxidized samples above 50 K is due to the smaller size of the FM Co core than that in the corresponding nonoxidized samples and, therefore, a lower value of the magnetocrystalline anisotropy. The mechanisms of the coercivity increase for Co-core CoO-shell nanoparticles after establishing an AFM structure in CoO, are discussed in Sec. IV.

For samples (o)dx D2 the coercivity is nonzero only at the lowest temperature at which the measurements were performed (5 K), meaning that the superparamagnetic blocking temperature of 2 nm nanoparticles is very low. However, at 5 K the coercivity of the oxidized sample is higher than that of the nonoxidized sample: 670 against 380 Oe for samples o d10 D2 and d10 D2, and 460 against 270 Oe for the samples o d5 D2 and d5 D2, respectively. This is consistent with the results for larger nanoparticles, and shows that a Co-core CoO-shell structure is present in these samples.

In Fig. 8 the coercivity versus temperature dependences for two specimens containing nonoxidized nanoparticles but

with different edge-to-edge in-depth separation (d5 D3 and d10 D3) are compared. In spite of the identical particle size (3 nm), the coercivity of the sample with an interlayer distance of 5 nm is systematically lower than that for the sample with the 10 nm interlayer separation. The matrix material is amorphous Al_2O_3 , which renders interparticle exchange interactions unlikely. However, such coercivity difference can be explained by a stronger dipole-dipole interaction between Co nanoparticles in neighboring layers in sample d5 D3 than in sample d10 D3. The dipole-dipole interaction is long range, increases the coherence of the magnetization rotation, and decreases the coercivity.

The field cooling was performed in a field of 10 kOe from 300 to 5 K. As was discussed in the Introduction, field cooling is needed to induce an exchange anisotropy in hybrid FM-AFM systems. For all samples with pure Co nanoparticles the field cooled hysteresis loop corresponds to the zero field cooled loop. However, for samples with Co/CoO nanoparticles no exchange bias was found after field cooling. A significant vertical shift of the hysteresis loop was observed instead. In Fig. 9 the zero field cooled (ZFC) and field cooled (FC) hysteresis loops for the samples of dx Dy are compared. All field cooled loops differ from zero field cooled loops only by a vertical shift, while no horizontal shift is observed. The zero field cooled loops are perfectly symmetric. This symmetry proves absence of exchange biased nanoparticles in the sample, since otherwise asymmetric steps or different magnetization reversal slopes for the right and left parts of the hysteresis loop would be observed. There is no evident correlation between the value of the vertical shift, and the nanoparticles size or the interlayer distance.

IV. DISCUSSION

Absence of exchange bias also has been observed by Skumryev *et al.* in isolated Co-core CoO-shell nanoparticles in Al_2O_3 matrix.¹⁰ Lund *et al.*²⁰ have shown that the total anisotropy energy of the AFM part is crucial for exchange bias to appear in FM-AFM bilayers. A model which describes magnetic behavior of hybrid FM-AFM nanoparticles has been developed in Ref. 13. Here, we summarize its key aspects.

FIG. 7. (Color online) Coercivity vs temperature for samples d5 D3 (pure) and o d5 D3 (oxidized). Lines are guides to the eye.

FIG. 8. (Color online) Coercivity vs temperature for samples d5 D3 ($d=5$ nm) and d10 D3 ($d=10$ nm). Lines are guides to the eye.

The considered model assumes that there are three essential energies, a competition between which determines the magnetic response of the system. These are the effective Zeeman energy of the FM part $E_{Z\text{eff}}$, the anisotropy energy of the AFM part E_A , and the exchange energy at the FM-AFM interface E_{int} . If $E_{\text{int}} < E_{Z\text{eff}}$, there are two possibilities. For $E_{\text{int}} < E_A$, the FM spins will be rotated while AFM spins will not, and exchange bias will be observed. For $E_A < E_{\text{int}}$, there will be no exchange bias. AFM spins will be rotated coherently with the FM spins, and the coercivity will be larger than that for a pure FM particle of the same size. For the case of $E_{Z\text{eff}} < E_{\text{int}}$ there are two possibilities as well. For $E_{Z\text{eff}} < E_A$, the Zeeman energy is not high enough either to overcome the interfacial energy barrier or to rotate the AFM spins. The FM part will stay “frozen” in an external field, and after field cooling this will show up as a vertical magnetization shift. For $E_{Z\text{eff}} > E_A$, the Zeeman energy is high enough to rotate both FM and AFM spins. Thus, there are three possible states in the system that are summarized in Table II and illustrated schematically in Fig. 10: exchange bias (EB) when $E_{\text{int}} < E_{Z\text{eff}}$ and $E_{\text{int}} < E_A$; AFM spin reversion (AR) when $E_A < E_{\text{int}}$ and $E_A < E_{Z\text{eff}}$; and frozen FM state, leading to the vertical shift (VS) after the field cooling for the case of $E_{Z\text{eff}} < E_{\text{int}}$ and $E_{Z\text{eff}} < E_A$.¹³ It is noteworthy that this vertical shift is due not to uncompensated AFM spins, but to the frozen FM spins. If the uncompensated AFM spins contributed to the vertical shift, one would assume that the AFM spin structure stays stable during the FM magnetization reversal. In that case exchange bias would be observed, thus contradicting our observations.

In the case of a spherical FM core-AFM shell particle with radius R and FM core radius r , the considered energies can be written as

$$E_{\text{int}} = 4\pi\sigma r^2, \quad (1)$$

with σ an empirical exchange coupling constant;

$$E_A = \frac{4\pi}{3}K_A(R^3 - r^3), \quad (2)$$

where K_A is the volume anisotropy constant of the antiferromagnet;

FIG. 9. (Color online) Zero field cooled (ZFC) and field cooled (FC) at 10 kOe hysteresis loops for the samples with oxidized Co nanoparticles: o d5 D3 (a), o d10 D3 (b), o d5 D2 (c), and o d10 D2 (d). None of the samples demonstrates exchange bias after field cooling, but a significant vertical shift of the hysteresis loops.

$$E_{Z_{\text{eff}}} = \frac{4\pi r^3}{3}(\mu_F H - K_F) = \frac{4\pi r^3}{3}\varepsilon_Z(H), \quad (3)$$

with the energy density $\varepsilon_Z(H) = (\mu_F H - K_F)$, μ_F is a specific magnetic moment, K_F a volume anisotropy constant of the FM part, and H the applied magnetic field. The critical size for exchange bias corresponds to the case when all these energy terms are equal to each other.¹³ The critical radius for EB is

$$R_c = \frac{3\sigma}{\varepsilon_Z} \left(1 + \frac{\varepsilon_Z}{K_A}\right)^{1/3}. \quad (4)$$

The obtained critical size for exchange bias is thus a function of the applied field. This dependence is plotted in Fig. 11 for the Co/CoO case. The constants used here are $\sigma = 3 \text{ erg/cm}^2$,²¹ $K_A = 2.7 \times 10^8 \text{ erg/cm}^3$, and $K_F = 2 \times 10^6 \text{ erg/cm}^3$.²² For a field of 10^4 Oe the critical diameter is $2R_c = 12 \text{ nm}$, well above the mean diameter of the nanoparticles studied in this work. For a nanoparticle smaller than the critical size, the exchange bias is forbidden for *any* ratio of the FM to the AFM part in the particle. In other words, while for the shaded region in Fig. 11 the exchange bias is strictly forbidden, for the region above this curve the bias exists only for certain FM to AFM parts ratios. For a nanoparticle of 3 nm diameter one should apply an external field of about 50 kOe in order to get the possibility to observe

exchange bias at a certain FM-AFM ratio in the particle. The necessity of applying such high fields makes potential applications of hybrid nanoparticles extremely difficult, since it requires massive and expensive superconducting magnets. Moreover, this can be meaningless,^{23–25} since the AFM spin-flop transition field is size dependent,^{23–25} and therefore the AFM structure in the thin shells can be destroyed by relatively small fields of several tesla.²⁵

Dipole-dipole interactions are not considered in the above-described model, since they are generally weaker than other energy terms involved. Taking into account dipole-dipole interactions will lead to a shift of the value of the critical size for a system of isolated nanoparticles. However, it does not change the fact that the critical size exists both for a single FM-core AFM-shell nanoparticle and for a system of such nanoparticles, isolated from each other by an amorphous insulating matrix.

V. CONCLUSIONS

In this article the production and magnetic properties of pure and oxidized Co nanoparticles embedded in an amor-

TABLE II. Energy conditions and corresponding states in a hybrid FM-AFM nanoparticle after field cooling and applying the opposite field below the Néel temperature of the antiferromagnet.

Energy conditions	State
$E_{\text{int}} < E_{Z_{\text{eff}}}$ and $E_{\text{int}} < E_A$	Exchange bias
$E_A < E_{\text{int}}$ and $E_A < E_{Z_{\text{eff}}}$	AFM spin reversal
$E_{Z_{\text{eff}}} < E_{\text{int}}$ and $E_{Z_{\text{eff}}} < E_A$	Ferromagnet pinning

FIG. 10. (Color online) Schematic of the ferromagnet and the antiferromagnet spin reversal. The initial state, after field cooling, is shown on the left side. On the right side the three possible variants of spin reversal are shown.

FIG. 11. Critical diameter for exchange bias as a function of the applied field for single domain Co-core CoO-shell particle. In the region under the curve the exchange bias is forbidden for any ratio of the FM to the AFM part in a hybrid particle.

phous Al_2O_3 matrix were described. The nanoparticles are well isolated from each other by an amorphous nonconducting matrix, which eliminates any interparticle exchange interactions. A comparison of zero field cooled hysteresis loops for similar samples with oxidized and nonoxidized nanoparticles provides clear evidence for the formation of an AFM CoO shell in the oxidized Co nanoparticles. While for samples with pure Co nanoparticles the low-temperature coercivity increases with the particle size and interlayer distance increase, we do not observe such a trend for the samples with Co/CoO nanoparticles. This shows that the coercivity of the samples with oxidized Co nanoparticles is governed by the interfacial exchange interactions and anisotropy of the antiferromagnetic part, rather than by anisotropy of the Co part and interparticle dipole-dipole interactions. The coercivity versus temperature behavior for the samples with oxidized nanoparticles shows that the blocking temperature of thin CoO shells in the samples is less than 50 K. This is much less than the blocking temperature of “thick” Co/CoO bilayers. Such a difference is a consequence of finite-size effects in ultrathin CoO shells. For all samples with oxidized nanoparticles, no exchange bias was observed after field cooling, while vertical hysteresis loop shifts were always observed. This provides evidence for the nanoparticle sizes being below the critical size for exchange bias.

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