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# Magnetization reversal studies in structurally tailored cobalt nanowires



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#### ABSTRACT

Cobalt nanowires (NWs) having *hcp* crystal structure are structurally tailored for different preferred orientations (PO) of (0002), (1010), (1120) and (1011) by varying bath temperature and bath concentration in commercially available 50 nm pore diameter polycarbonate (PCT) and 20 nm pore diameter anodic alumina (AAO) membranes. The magnetization studies show orientation dependent competition of magneto-crystalline anisotropy with shape anisotropy. The large effective anisotropy,  $K_{eff}$  (along longitudinal direction) of  $1.42 \times 10^6$  erg/cc is observed in (0002) PO NWs, which changes sign ( $-1.50 \times 10^6$  erg/cc) in (1010) PO NWs. The angular dependence of coercivity [ $H_c(\theta)$ ] in (0002) oriented Co NWs exhibits a non-monotonic behavior in both the 50 nm and 20 nm samples. The fitting of  $H_c(\theta)$  data reveals that the magnetization reversal mechanism initially takes place by curling and subsequently changes to coherent rotation mode after a certain transition angle, which is higher in case of denser NW array. This increase in transition angle can be attributed to the increased magneto-static interactions in the AAO membrane array having  $10^3$  times higher NW areal-density than that in PCT membrane array. © 2013 Elsevier B.V. All rights reserved.

#### 1. Introduction

Magnetic nanowires (NWs) have drawn immense interest due to their application in reprogrammable magnonic crystal devices, magnetic sensors and spintronics application based GMR and TMR devices [1,2]. Cobalt has the strongest uniaxial magnetocrystalline anisotropy (MCA) along the *c*-axis of its closed packed *hcp* unit cell whose energy  $(4.5 \times 10^6 \text{ erg/cc})$  is comparable to the shape anisotropy (SA) energy  $(6 \times 10^6 \text{ erg/cc})$  of infinitely long cylindrical NW. Magnetization in cobalt NWs has been recently studied because of its tailoring of the magnetic crystal anisotropy energy in these nanostructures via growth conditions [3-6]. Different techniques are used to fabricate NWs. Among these, the electro-deposition provides a simple and controllable method to deposit NWs with different structural orientations in commercially available anodic alumina (AAO) [3,4] and polycarbonate (PCT) [5,6] membranes. Another important anisotropic contribution in a NW-array is magneto-static interactions among the NWs which is dependent on the pore density of the membrane. Tailoring of the magnetic anisotropy in such a system therefore can play an important role in developing various technological applications.

Magnetization behavior in such oriented cobalt NWs is of particular interest since one nanowire could behave as a "single domain" entity and its magnetization reversal could be similar to the coherent rotation, i.e., all spins rotating in unison as in single domain ferromagnetic particles. Apart from this, there are other complex magnetization reversal mechanisms that could be operative due to nucleation of a vortex or transverse domain at the ends of the NW. The nucleated domain then propagates along the length of the NW as the field is swept and results in spin reversal. The reversal mechanism mode could be curling (for vortex domain nucleation) or transverse (for transverse domain nucleation). The operative magnetization reversal mechanism is mainly derived from the study of the dependence of nucleation field (which is the field at which a change in magnetization just starts in a saturated single-domain state) [7] or from the dependence of coercive field (which is the field at which the magnetization changes sign) with the angle  $\theta$  between the applied magnetic field (*H*) and NW-axis.

In this communication, we present the study of magnetic behavior in structurally tailored cobalt NWs of (0002), (10 $\overline{10}$ ), (11 $\overline{20}$ ) and (10 $\overline{11}$ ) preferred orientations (PO) in two different membranes viz., 50 nm *dia* polycarbonate and 20 nm *dia* anodic alumina. We have used the angular dependence of coercivity  $H_C(\theta)$  to study the magnetization reversal mechanism in the two sets of NWs having different areal-densities, AAO membrane having 10<sup>3</sup> times higher density than PCT. The results are explained in conformity with competition of different anisotropy contributions.

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Two types of commercially available porous membranes, namely (1) polycarbonate membrane with nominal pore diameter of 50 nm and a pore density of  $10^9 \text{ cm}^{-2}$  and (2) anodic alumina membrane with 20 nm pore diameter and pore density of 10<sup>12</sup> cm<sup>-2</sup> were coated with 200 nm gold on one side that serves as cathode for electro-deposition in the pores. Different crystallographic orientations of cobalt NWs were obtained by using two different bath temperatures of 50 °C and 60 °C and two different concentrations of electrolytic bath with compositions: 25 mM CoSO<sub>4</sub> · 7H<sub>2</sub>O & 0.1 M H<sub>3</sub>BO<sub>3</sub> and 500 mM CoSO<sub>4</sub> · 7H<sub>2</sub>O & 0.4 M H<sub>3</sub>BO<sub>3</sub>. The bath pH was maintained at 4.5 by either by adding few drops of H<sub>2</sub>SO<sub>4</sub> or NaOH. The electro-deposition is carried out at a constant potential of -1.0 V in a three electrode cell using computer controlled Potentiostat (Model CHI-1100A). The cell consists of a standard calomel (Hg/ HgCl<sub>2</sub>/KCl) reference electrode and a 2 cm  $\times$  1.5 cm Pt sheet as the counter electrode. Deposition was done for the predetermined time to obtain a wire length of about 1200 nm. The structural measurements of the synthesized nanowires were subsequently done by using Philips X'Pert PRO X-ray diffractometer (40 mA, 45 kV) with Cu-K $\alpha$  radiation ( $\lambda$  = 1.5406 Å) in Bragg—Brentano geometry. The Xray diffractograms were recorded on the NWs embedded in membrane itself. The scanning electron microscope (SEM) images of the nanowires were obtained by a ZEISS EVO-50 microscope. On selected samples high-resolution transmission electron microscope (HRTEM) images are acquired using JEOL JEM 2100F microscope equipped with a field emission gun operated at 200 kV. Three samples of the Co NWs deposited in 50 nm PCT and two samples of Co NWs deposited in 20 nm AAO of different PO of hcp-Co were selected for the study. Magnetization measurements were then done with vibrating sample magnetometer (VSM).

#### 3. Results and discussion

#### 3.1. Structural characterization

Fig. 1(a) and (b) shows the X-ray diffraction (XRD) patterns of five samples with NWs embedded in the membrane itself. Since the nanowire axis is perpendicular to the plane of the membrane, the observed peaks in the diffractograms would come from the cobalt planes lying parallel to the membrane plane. Fig. 1(a) shows XRD patterns of three NW samples namely PCT-S1, PCT-S2 and PCT-S3 deposited in 50 nm PCT under different conditions. The sample PCT-S1 (shown in red), was deposited from 25 mM cobalt bath at a temperature of 60 °C which shows the highest intensity peak at

44.5° that corresponds to the (0002) hcp-Co peak. Though it has small contribution from Au under-layer peak at 44.2° but its strong intensity at 44.5° is assigned to come mainly from cobalt deposited in the membrane after taking into account the intensity of the other weak *fcc*-Au peaks at 64.5° and 77.5° due to the gold under-layer. The (0002) PO growth suggests that c-axes of the unit cells in various grains are parallel to NW-axis. Sample PCT-S2 was deposited from 25 mM cobalt bath at 50 °C and its XRD (shown in blue) shows the dominant presence of  $(11\overline{2}0)$  and  $(10\overline{1}0)$  peaks of *hcp*-Co along with weak Au peaks. This observation of  $(11\overline{2}0)$  and  $(10\overline{1}0)$ PO growth thus suggests that the *c*-axes of the unit cells in the various grains are perpendicular to NW-axis. The two different set of planes along which the PO growth occurs in this case means a relative rotation of hexagonal-prism unit cells about the *c*-axis. On the other hand the sample PCT-S3 (shown in black) deposited from concentrated 500 mM cobalt bath at 60 °C shows the high intensity peak at  $47.5^{\circ}$  that corresponds to the  $(10\overline{1}1)$  hcp-Co peak. The other weak peaks at 44.2°, 64.5° and 77.5° correspond to the *fcc*-Au peaks. It may be noted that fcc (200) peak of Au occurs at 44.2° and *hcp*-Co (0002) peak occurs at 44.5°. The inset of Fig. 1(a) presenting the expanded view near  $2\theta$ -range of 44—45° reveals that the observed diffraction peak in case of PCT-S1 is due to Co and that in case of PCT-S3 is due to Au. Thus all the nanowire samples possess well defined PO character. This has been confirmed by detailed HRTEM study of samples PCT-S1 and PCT-S2 reported previously [6], in which deposition-mechanism controlled PO effects were concluded. HRTEM study of PCT-S3 sample is presented later in Fig. 4.

Fig. 1(b) shows X-ray diffractograms of two samples namely AAO-S1 and AAO-S2 deposited in 20 nm AAO membranes. Sample AAO-S1 (shown in red) is deposited from 500 mM cobalt bath at 60 °C which shows the prominent peak at 44.5° that corresponds to the (0002) *hcp*-Co planes along with the presence of weak *fcc*-Au peaks due to under-layer. Thus samples PCT-S1 and AAO-S1 possess the same preferred orientation. The sample AAO-S2 (shown in blue) deposited from 500 mM cobalt bath at 50 °C shows (1010) and (1120) PO growth, like the sample PCT-S2.

Fig. 2 (a) and (b) respectively shows the SEM images of Co nanowires of sample PCT-S1 after dissolving the PC membrane in dichloromethane and sample AAO-S1after dissolving AAO membrane in 0.1 M NaOH solution. The length and diameter of observed NWs are 1.2  $\mu$ m and ~80 nm in PCT-S1 and 1.6  $\mu$ m and ~25 nm in AAO-S1. Fig. 3(a) and 4(a) shows the TEM images of the NW samples AAO-S2 and PCT-S3. HRTEM images of the AAO-S2 NW taken at two different locations of the same NW are shown in Fig. 3(b) and (c). The white arrow in these HRTEM images indicates the NW-axis. The lattice spacing of the planes seen in these images is 0.24 nm, which is twice that of the lattice spacing of the (11 $\overline{2}$ 0) planes, i.e. 0.12 nm, and thus clearly establishes the PO growth of (11 $\overline{2}$ 0) planes



D. Kaur et al. / Journal of Magnetism and Magnetic Materials 344 (2013) 72-78



Fig. 1. XRD patterns of cobalt nanowires samples (a) PCT-S1, PCT-S2 and PCT-S3 deposited in 50 nm PCT membrane and (b) AAO-S1 and AAO-S2 in 20 nm AAO membrane. The peaks labeled with "" are due to the Au under-layer. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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