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## Characterizing magnetic interactions in Ni nanowires by FORC analysis

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#### Abstract

Polycrystalline Ni nanowires with different diameters were electrodeposited in nanoporous anodized alumina membranes. First-Order Reversal Curves (FORCs) were measured and FORC distributions were calculated. They clearly showed an asymmetric behavior with a strong maximum at negative interaction fields, evidencing the dominant demagnetizing interactions which depend on the geometry of the nanowires.

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#### 1. Introduction

Self-organized arrays of ferromagnetic nanowires show potential feasibility for applications in perpendicular magnetic recording media and high-sensitivity magnetic sensors [1], and are well suited for both experimental and theoretical study of magnetism at nanoscale [1–4]. These systems can be easily synthesized through the method of two-step anodization [5] and subsequent chemical electrodeposition [2,3].

One phenomenological approach that has been topic of renewed interest in magnetic systems is the First-Order Reversal Curves (FORC) analysis [6–11]. FORCs can be experimentally obtained as follows: (a) first, one has to saturate the sample with a field  $H_{\rm max}$ ; (b) then, set the field H to a return value  $H_{\rm r} < H_{\rm max}$ ; (c) then, increase H back to  $H_{\rm max}$ , measuring the magnetization; and (d) and repeat steps (b) and (c) for decreasing values of  $H_{\rm r}$ , while  $H_{\rm r} \ge -H_{\rm max}$ .

The FORC distribution is, then, defined by  $\rho(H_r, H) \equiv -0.5\{\hat{\partial}^2 M(H_r, H)/\hat{\partial} H_r \hat{\partial} H\}$  and performed on the dataset of the measured FORCs. The FORC diagram is a contour plot of  $\rho$  on a 45° rotated coordinate system  $\{H_c = (H - H_r)/2, H_b = (H + H_r)/2\}$ . In these coordinates,

 $\rho$  can be compared to the statistical distribution of particles from the Preisach model [12], which describes the field-induced reversal of an ensemble of single-domain particles.

Thus, the FORC diagram provides a detailed characterization of the hysteretic response of a system because it evidences dominant magnetic interactions, magnetic aftereffects and the annihilation of memory during the demagnetization process.

In this work, a set of samples of Ni nanowires grown in the nanoporous anodized alumina membranes were magnetically characterized by FORC analysis.

#### 2. Experimental details

Thin (0.2 mm) Al foils with 99.997% purity were pretreated in vacuum at  $600\,^{\circ}\text{C}$  for 2 h; washed in acetone; electropolished in a  $\text{H}_2\text{SO}_4 + \text{H}_3\text{PO}_4 + \text{H}_2\text{O}$  solution; etched in a 3 M NaOH solution and rinsed in deionized water. Later, the foils were anodized in a constant voltage cell containing an aqueous solution of 0.3 M oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ), at 4 °C, using a graphite electrode as cathode. The first anodization was carried out for 1 h and the second for 40 min. Several samples were prepared with anodization voltage  $V_{\rm an}$  varying between 15 and 30 V. The electrodepositions were carried out in a solution of 0.1 M NiSO<sub>4</sub> · 6H<sub>2</sub>O + 45 g/LH<sub>3</sub>BO<sub>3</sub>, at 55 °C, for 2 min, under 20 V, at 100 Hz.

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The structural characterization was done by Scanning Electron Microscopy (SEM) and X-ray Diffractometry (XRD), with Cu  $K_{\alpha}$  radiation. The magnetic characterization was accomplished at 300 K in a 7T-SQUID, with magnetic field applied perpendicularly to the membrane surface.

#### 3. Structural and magnetic characterization

SEM images of the samples were obtained and treated in a suitable image processing software, from where we calculated the mean diameter  $d_{\rm m}$ , the mean interpore distance  $D_{\rm int}$  and the density of pores. A linear dependence of  $d_{\rm m}$  and  $D_{\rm int}$  with  $V_{\rm an}$  was observed, with  $d_{\rm m}$  between 30 and 90 nm and  $D_{\rm int}$  between 40 and 110 nm, in agreement with the literature [13,14]. The mean density of pores varies between 10<sup>9</sup> and 10<sup>10</sup> pores/cm<sup>2</sup>. The shape of the pores also varies with  $V_{\rm an}$ . Samples made at 30 and 40 V have a more regularly oriented hcp patterns. Below  $V_{\rm an} = 30 \,\mathrm{V}$ , the pore arrays are predominantly random, whereas above 40 V the pores tend to deform. It is well known that the regularity of the pore array depends on voltage and duration of the first anodization [15-17] and that for high  $V_{\rm an}$  the pores tend to diverge from the circular shape [18]. On the other hand, the length of the pores depends solely on the duration of the second anodization [13]. We estimate that our samples have lengths between 5 and 15 μm, yielding an aspect ratio greater than 50.

The XRD patterns confirm the presence of polycrystalline Ni. The (111) peak of Ni was not detected due to its coincidence with the strong (200) peak of Al. The grain sizes of Ni were calculated for each crystallographic direction and they were seen to increase with  $d_{\rm m}$ , in the range of 10–22 nm, indicating that the geometry of the pores regulates the grain size of Ni. In this range, the crystallites must be exchanged-coupled single-domain particles, with random orientation of easy axes [2]. Such a magnetic structure can allow non-uniform modes of magnetization reversal [19,20].

The shape uniaxial anisotropy in our samples was confirmed by measuring hysteresis loops in the directions perpendicular and parallel to the wires. An outstanding increase of coercive field  $H_{\rm C}$  and remanence ( $M_{\rm R} > 0.5 M_{\rm S}$ ), in comparison to bulk Ni, was observed. It is in accordance with Ref. [19]. To obtain the FORC distributions, approximately 20 FORCs with  $H_{\rm max} = 3\,\rm kOe$  were measured for each sample (see Fig. 1a). The FORC distributions were calculated by the method described by Heslop and Muxworthy [11], applying the "extended" FORC definition [7], normalized by the saturation magnetization  $M_{\rm S}$  of each sample and plotted in the  $\{H_{\rm c}, H_{\rm b}\}$  coordinates. Fig. 2b displays typical results for the sample with  $d_{\rm m} = 38\,\rm nm$ .

#### 4. Results and discussion

In the classical Preisach model, a distribution of noninteracting single-domain particles is considered, where

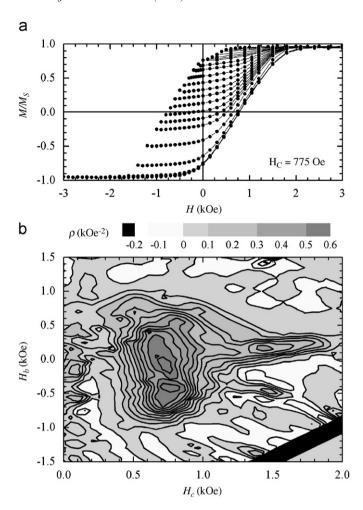


Fig. 1. (a) FORCs and (b) FORC diagram for the sample with  $d_{\rm m}=38\,{\rm nm}.$ 

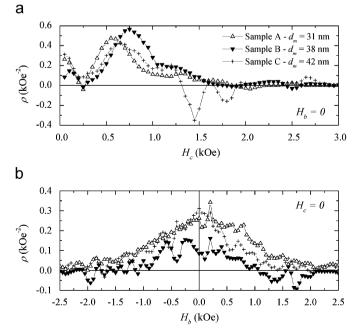


Fig. 2. FORC distribution of all samples at (a)  $H_b = 0$  and (b)  $H_c = 0$ .

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