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Magnetic viscosity in iron-rhodium nanowires

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ABSTRACT

Noble/transition bimetallic nanowires of nominal composition $Fe_{90}Rh_{10}$ are AC electrodeposited into 20 nm diameter hexagonally self-assembled alumina nanopores. Nanowires about 18 nm in diameter and 1 µm long are polycrystalline and multiphase. Wires contain α -Fe grains and very small grains of the ClCs-type α' -Fe(Rh) phase. The room temperature magnetization mechanism and the thermal stability of nanowire magnetic configurations are further investigated by measuring the dependence of the coercive field on the applied field sweeping rate R. From these data a mean fluctuations field value of $\mu_0 H_{FR} = (9.0 \pm 0.5)$ mT is obtained (at coercivity) and an effective activation magnetic moment $\mu_{ac} = 5400 \ \mu_B$ is estimated, with μ_B the Bohr magneton. At the coercive field (about 45 mT) the resulting activation lengths become $l_{AC} \approx 6.4$ and 6.7 nm for α -Fe and α' -Fe(Rh), respectively. Assuming an effective magnetic anisotropy, considering magnetostatic shape contributions in addition to the magnetocrystalline one, the domain wall thickness δ_w in α -Fe grains and in the α' -Fe(Rh) become $\delta_{wFe} = 13.4$ nm and $\delta_{wFeRh} = 10.9$ nm respectively. These values are comparable to the activation lengths estimated at the coercive field in each phase. These facts strongly indicate that irreversible polarization reversal in these nanowires takes place by local curling, involving localized nucleation modes.

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1. Introduction

In many applications of magnetic nanowires, as magnetic sensors and catalysts [1], the properties of interest are directly related to the stability of a given magnetic state against low and even zero external fields (remanence). Then, the magnetization reversal processes operating in the individual nanowires become of central interest.

Recently, one dimensional bimetallic Fe-rich ($Fe_{90}Rh_{10}$) – nanowires were successfully synthesized by electrodeposition into alumina templates [2], and the resulting microstructures characterized. Nanowires morphology, grain size and phase composition were determined and the magnetic hysteresis properties explored. Special attention was paid to the magnetization reversal

mechanism operating in these polycrystalline Fe₉₀Rh₁₀ biphasic nanowires by measuring the temperature dependence of the coercive field and the angular variations of coercivity and remanence. The observed behaviors [2] were consistent with a magnetic polarization reversal mechanism controlled by the nucleation of inverse domains by local curling [3]. The apparent activation energy values obtained were comparable to those reported by Paulus et al. (2.4–5.1 eV) [4] for a mechanism of inverse domain nucleation in Fe nanowires. This activation barrier is a measure of the excess in free energy necessary to locally create a domain wall in the nucleation site, determined by factors such as the magnetocrystalline and shape anisotropies, the magnitude of local magnetostatic stray fields and in particular structural defects.

It is then important to provide independent evidence of such localized nucleation to further confirm the mechanism previously proposed, and to estimate the characteristic nucleus size.

In this short article, we report the room temperature values of two magnitudes closely related to the thermally activated polarization reversal mechanism in $Fe_{90}Rh_{10}$ 'as synthesized'



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nanowires: the fluctuations field and the activation volume. The resulting values for these magnitudes strongly support the idea of a magnetization process controlled by the nucleation of inverse domains in quite small regions in the nanowire.

2. Experimental procedures

Porous anodized aluminum oxide (AAO) membranes are used as hard templates [5]. The complete procedure to obtain these porous membranes from a high-purity aluminum foil (99.995%, Alfa AEsar) is described elsewhere [2]. Arrays of ordered pores 20 nm in diameter and about 3 μ m in length are obtained.

Fe-Rh nanowires are prepared by AC electrodeposition from an aqueous electrolytic bath containing 0.10 M FeSO₄·7H₂O, 0.0067 M RhCl₃, 0.75 g/L ascorbic acid and 30 g/L H₃BO₃ in ultrapure water (MilliQ RiOs 16, Millipore), keeping pH = 4.0. Electrodeposition is carried out at room temperature with an AC voltage of 15 V_{rms} and 60 Hz during a few minutes.

XRD profiles are recorded in a PANalytical Empyrean diffractometer, using Cu K α radiation ($\lambda = 1.5418$ Å), in the 2 θ range from 30° to 90°. Samples are measured after dissolving the remaining Al substrate in a CuSO₄ and HCl solution to eliminate intense Al x-ray peaks. Samples for SEM observations are further immersed in a 1.0 M NaOH solution to dissolve the AAO template and separate the nanowires. After sonication and rinsing in distilled water, dispersed nanowires are obtained, as illustrated in Fig. 1 a.

The nanowire morphology is inspected in a scanning electron microscope FE-SEM Zeiss and the mean array composition determined by energy-dispersive X-ray spectroscopy (EDS), in the SEM. This mean composition is nominal, because nanowires are multiphase, as will be seen below. The individual wire nanostructure and composition are investigated by transmission electron microscopy (TEM) in a Philips CM200UT microscope, operating at 200 kV, with an EDS analysis device. Samples for TEM observations are prepared by dispersing the liberated nanowires in ethanol and further depositing a drop of this emulsion on a holey carbon-coated copper grid.

Room temperature hysteresis loops and polarization relaxation are measured in a vibrating sample magnetometer (VSM) Lakeshore 7300, with a maximum field up to 1.5 T, with the external field applied parallel (PA) and perpendicular (PE) to nanowires major axis. The total magnetic moment of the assembly has contributions from the Al support (paramagnetic), the alumina template (diamagnetic) and the metallic wires filling the pores (ferromagnetic), so the ferromagnetic component must be estimated after subtracting the other contributions.

The hysteresis loops are analyzed considering a linear contribution arising from paramagnetic (Al) and diamagnetic (alumina template) contributions superimposed to a ferromagnetic one, given by Ref. [6]:

$$J_{FM} = \frac{2J_{S,FM}}{\pi} \arctan\left[\frac{(H_i \pm H_{iC})}{H_{iC}} \tan\left(\frac{\pi J_{R,FM}}{2J_{S,FM}}\right)\right],\tag{1}$$

where, $J_{S,FM}$, $\mu_0 H_{iC}$ and $J_{R,FM}$ are the saturation polarization, the coercive field and the remanent polarization, respectively, associated with the ferromagnetic contribution.

The effective fluctuations field $\mu_0 H_{FR}$ [7,8] is determined from the field time rate dependence of the coercive field at constant temperature [9]:

$$H_{FR}(H_i, J) = -\frac{\partial H_i}{\partial \ln R}\Big|_I$$
(2)

where R is the speed at which the field changes during loop tracing and the internal field: $\mu_0 H_i = \mu_0 H - NJ$, with H the applied field and N the wire demagnetizating factor. Near the coercive field, J~0



Fig. 1. (a) SEM micrograph showing nanowires after partial dissolution of the alumina template. (b) TEM bright field (BF) image of polycrystalline Fe₉₀Rh₁₀ nanowires, composed by large α -Fe grains (white arrows) and ensembles of small grains of a Rh-rich ClCs-type phase (black arrows). (c) BF image of small grains of the α' - Fe(Rh) ordered phase at the tip of an α -Fe phase grain (large circle). *Inset*: HRTEM image of the ordered phase, showing very small grains with a mean diameter of (2.0 ± 0.5) nm. (d) Detail of an α -Fe grain: BF micrograph and corresponding 110 dark field (DF) image. A ~4 nm thick magnetite layer is observed at the wire surface.

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