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Magnetic investigations of post-annealed metallic Fe nanowires via electrodeposition method

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ABSTRACT

Smooth and long Fe nanowire (NW) arrays were successfully fabricated inside nano-channels of anodic aluminum oxide (AAO) template with DC electrodeposition method at room temperature. As-synthesized Fe NWs were investigated by scanning electron microscopy (SEM), energy-dispersive X-ray (EDX) methods and X-Ray diffraction (XRD) for morphology, elemental analysis and structural information, respectively. It has been observed that embedded Fe NWs have bcc crystal structure and polycrystalline nature. Shape anisotropy played the dominant role to orient the magnetic moments along the nanowires long axes. Low squarness (M_r/M_s) values for in-plane magnetization loops demonstrate the presence of dipole interaction field between the nanowires due to high pore density or small inter-pore distance (~125 nm). Annealing treatment improved the magnetic properties of Fe nanowires. The possible magnetic discussions in nano-geometry have been presented in detail.

1. Introduction

Reducing the dimension towards the nanoscale especially in nanowires suggests an additional degree of freedom associated with their inherent shape anisotropy and dominant surface effects. Nowadays magnetic nanowire arrays as a popular group of the nanostructures received considerable attention due to potential applications in the various fields including sensors, data storage media [1–4], as well as for fundamental scientific studies of nanomagnetics [5–8].

In present work, we describe smooth and reproducible Fe nanowires in nano-porous templates. Magnetic behavior has been reported of Fe nanowires at different annealing temperature designed as (F1-As-synthesized), (F2-200 °C), (F3-400 °C), (F4-600 °C). Angular dependence of coercivity and remanence squarness has been discussed. The easy magnetization direction of Fe nanowires was found to orient along the nanowire axis due to the high aspect ratio and the magnetization reversal mechanism takes place by nucleation mode indicated by H_c (θ) curve.

2. Experimental details

Fe nanowires were fabricated by electrodeposition using AAO templates with the pore size 80 nm. We used an electrochemical cell designed to improve current efficiency and to precisely control a deposition area. Direct connection between the electrolyte and the cathode via AAO pores was secured by mounting the AAO template to the cell. Before electrodeposition, one side of AAO membrane was coated with a gold layer as the working electrode for reduction of metallic ions from the electrolyte. A piece of 0.2 mm thick Pt sheet was used as a counter electrode. The bath consists of 1 g/50 ml $FeSO_4$, 1 g/50H_3BO_3 and 0.1 g/50 ml L-ascorbic acid in deionized water. The electrodeposition was carried out under constant stirring, and at a bias of −1.1 V (versus SCE). The wires grew in the usual bottom-up fashion starting from the Au electrode at the pore bottoms.

The samples were micro-structurally analyzed by X-ray diffraction (XRD: RIGAKU-D/MAX-2400, Cu Kα, λ=0.154056 nm). The morphology of nanostructures was collected by field emission scanning electron microscopy (FE-SEM: Hitachi S-4800) and compositional analysis were performed with energy dispersive X-ray spectroscopy (EDS) integrated with FE-SEM. Room temperature magnetic properties were measured by magnetic sample magnetometer (VSM: Microsense EV-9).

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3. Results and discussion

Fig. 1 depicts the XRD pattern of Fe nanowire arrays after completely etching the AAO-template in a 1 M solution of NaOH. From the XRD spectrum it can be inferred that Fe grows in bcc crystal structures and is clearly polycrystalline. The diffraction peaks of (110), (200) and (211) are clearly distinguishable and match perfectly with the bcc Fe structure with a lattice constant of $a=0.28664$ nm (JCPDS 06-0696). It is noteworthy that Fe is often found to grow strongly textured inside AAOs [9–12]. The fact that the wires are polycrystalline may be related to the wider diameter of the pores, the amorphous nature of the AAOs, or the electrodeposition process.

From Fig. 2(a)–(d), a mean nanowires diameter of 100 nm, and a length of 8–10 μm has been analyzed. Images also show that nearly all the pores were filled. Fig. 2(a) and (c) shows some nanowires bent out of the pores. Such bending was produced during the breaking of the AAO template to obtain cross-section SEM images and it demonstrates the malleability and metallic character of the nanowires. Fig. 2(d) displays a representative energy dispersive X-ray microanalysis spectrum (EDS). Because of the AAO template presence, Al is the most intense peak, while O is also detected. Gold peaks are due to the electrically conductive layer sputtered at the bottom of the template, and finally, Fe peaks correspond to the magnetic nanowires.

The hysteresis loops under applied magnetic field parallel to the nanowires axis have been measured at room temperature for as-prepared samples and after annealing at F2-200 °C, F3-400 °C, F4-600 °C (Fig. 3). For a better description of the influence of thermal treatments, the evolution of coercivity and squarness are tabulated in Table 1. As observed, the squarness takes a value of 0.12 in the as-prepared state, and it increases gradually by increasing the annealing temperature and reaches a maximum of 0.18 for the sample annealed at 600 °C. Squarness takes very small values (less than 0.04) in the case of hysteresis loops under applied

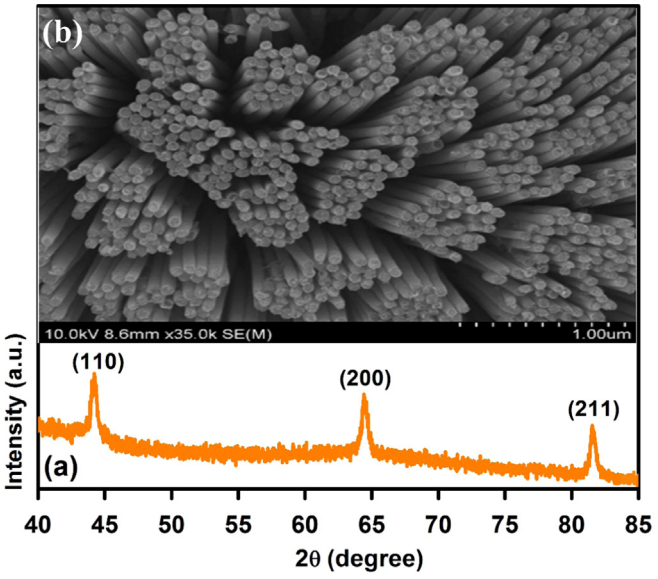

Fig. 1. XRD-Analysis and cross-sectional view of Fe nanowires.

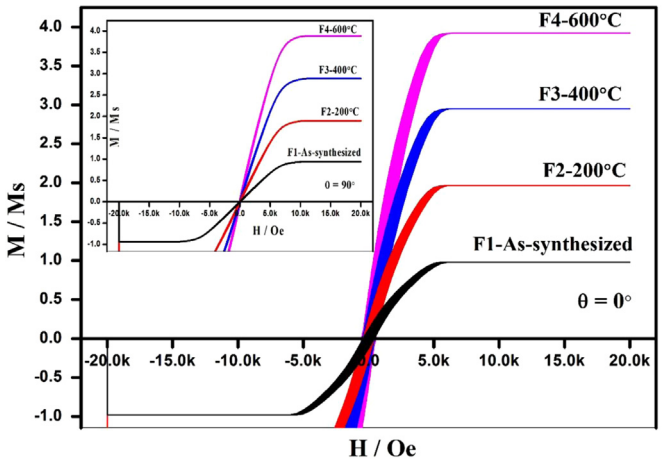

Fig. 3. Magnetic responses of Fe nanowires; an external magnetic field is aligned parallel to the long axis of nanowires.

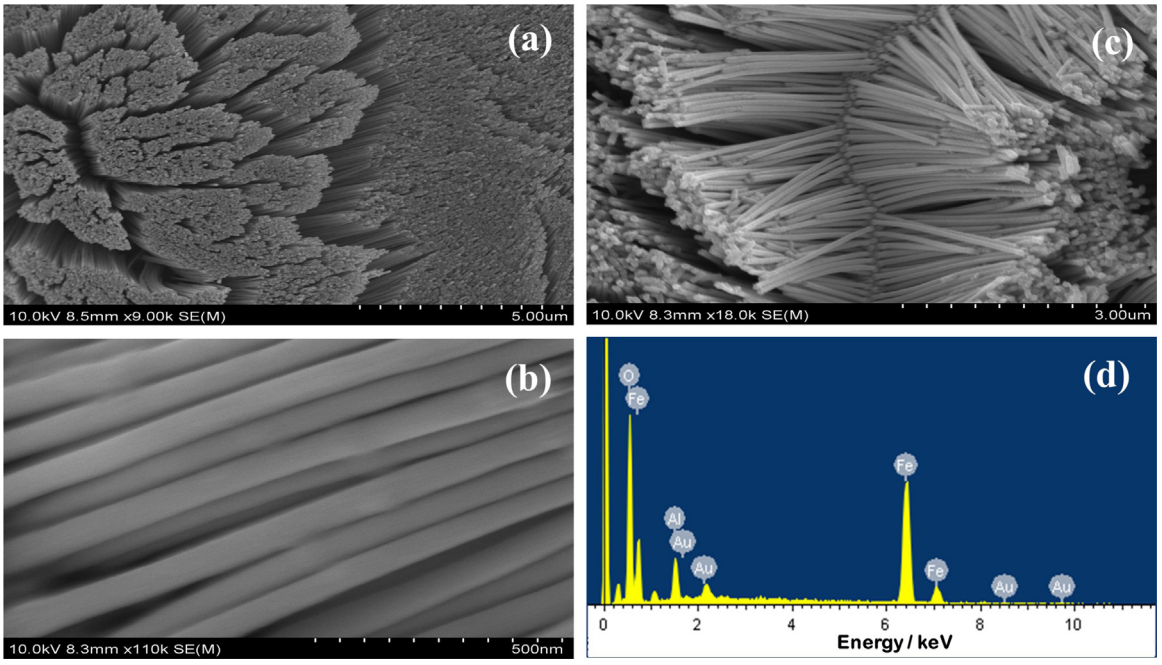

Fig. 2. FE-SEM and EDX analysis of Fe nanowires.