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Structural and magnetic properties of Fe_3Ga alloy nanowires: Effect of post annealing treatment

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A B S T R A C T

We report on the influence of the annealing temperature on the crystal structure phase change, effective anisotropy energy and magnetization reversal of electrodeposited Fe_3Ga nanowires (NWs) with diameter of 80 nm. Scanning transmission electron microscopy (STEM) energy-dispersive X-ray (EDX) spectroscopy technique for the elemental mapping of Fe_3Ga NWs showed clearly the homogeneous distribution of Fe and Ga. These NWs are highly aligned to each other and exhibits high geometrical aspect ratio. The crystal structure of these nanowires shows strong dependence on the sintering temperature. X-ray diffraction reflects the highly textured A2-disorderd phase with precipitates of DO_3 and $L1_2$ phase for as-synthesized Fe_3Ga NWs. As the annealing temperature increases, the intensity of strong disordered phase A2 or short range order phase DO_3 suddenly decreases and grains are textured along the stable $L1_2$ phase with small precipitates of DO_3 phase. As a result of these structural changes, magnetization reversal mode switches from transverse to a combination of transverse and vortex modes. Angular magnetic response at room temperature is employed to identify the magnetization reversal modes for Fe_3Ga NWs. Temperature dependent coercivity response can be understood in terms of thermal activation over an energy barrier with a $\frac{3}{2}-$ power dependence on the field.

1. Introduction

It is well known that nowadays magnetic NW arrays have received considerable attention due to potential applications in the field of sensors, data storage media, sensors, actuators, etc [1–7]. The accurate measurement of the magnetic properties of nanostructures is highly important. The magnetic response in magnetic nanostructures is highly dependent upon their dimensional configuration like as shape, size, aspect ratio and spacing between adjacent elements [8]. It has been reported in many research articles that the materials possessing magnetic property of magnetostriction are of interest in energy harvesters, actuators, and sensors [9]. Large magnetic field which induces strains approximately 2k-ppm has been achieved in Terfenol-D which is considered a material with large magnetostrictive property [10]. Alloys based on FeGa (Galfenol) are promising candidates due to their several advantages like low saturation magnetic field, large tetragonal magnetostrictive coefficient, low cost and excellent ductility [11–14]. It is well-known that composition, annealing history and synthesis routes have been accused for magnetic response of Fe based alloys and compounds [15]. Furthermore, magnetic nanowires, due to their low dimensionality becomes of particular interest as compared with their bulk properties in the field of electronics, optics, semi-conductors, and magnetism [16]. The aim of present work is to report fabrication with their structural and magnetic characterizations of Fe_3Ga nanowires that are grown by the electrodeposition technique.

2. Experimental details

Fe_3Ga nanowires were fabricated by electrodeposition using AAO templates with pore size of 80 nm. Before electrodeposition process, the shining side of AAO template was coated with a copper layer thickness of 200 nm as the working electrode for reduction of Fe and Ga ions from the electrolyte. A small strip of 0.2 mm thick Pt sheet was used to serve as a counter electrode. The electrolyte was consisted of 0.1 g/50 ml $FeSO_4$, 0.5 g/50 ml $Ga_2(SO_4)_3$, 0.3 g/50 sodium citrate and 0.01 g/50 ml L-ascorbic acid in deionized water. The electrodeposition was carried out under constant magnetic stirring,

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and at a biasing voltage of −1.2 V (versus SCE). The NWs of Fe_3Ga grew in the usual bottom-up way starting from the Cu electrode at the pore bottoms of AAO template. After deposition, the magnetic NWs were annealed at temperatures 550 °C, 600 °C and 650 °C. The samples were designated as T1-As-synthesized, T2-annealed at 550 °C, T3-annealed at 600 °C and T4-annealed at 650 °C.

The samples were micro-structurally analyzed by X-ray diffraction (XRD: RIGAKU-D/MAX-2400, Cu Kα, λ = 0.154056 nm). The detailed morphology of NWs was collected by Field Emission Scanning Electron Microscopy (FE-SEM: Hitachi S-4800) and compositional analysis were characterized with Energy Dispersive X-ray spectroscopy (EDS) integrated with FE-SEM. (TEM) transmission electron microscopy (TEM: TecnaiF20). Magnetic measurements were performed by magnetic sample magnetometer (VSM: Microsense EV-9) and physical property measurement system (PPMS-model, 9 T).

3. Results and discussion

Unique anodic aluminum oxide (AAO) templates with self-assembled hexagonally arranged nanopores are ideal for synthesis of nanoscale materials due to easy preparation and controllable pore size. Here, porous alumina membranes with pore diameter of 80 nm is used to fabricate the Fe_3Ga NWs. Fig. 1 depicts the schematic diagram of the synthesis process of Fe_3Ga nanowires within the pores of AAO-template via electrodeposition method in detail. First, the shining side of AAO membrane is sputtered with Cu (200 nm), then NWs were grown through electrochemical rout. Afterwards, the template filled with NWs were chemically etched with NaOH etchant (partially) for 30 min at 60 °C and (complete) 24 h at 60 °C for FE-SEM and TEM measurements respectively, (Fig. 1). Fig. 2 shows the morphology of Fe_3Ga NWs characterized by using a field emission scanning electron microscope (FE-SEM) and a transmission electron microscope (TEM). The diameters for all of the NWs are consistent with the diameters of the AAO channel. It is apparent from Fig. 2 (a–b) that Fe_3Ga NWs were grown in highly uniform fashion. The length of these crack free and smooth NWs extends to several μm that can be seen in Fig. 2 (c) and inset of Fig. 2 (d). The elemental contribution of Fe and Ga in FeGa-alloy system of NWs is shown in Fig. 2 (d). This confirms the existence of Fe and Ga in the required ratio of (3:1) respectively. It should be pointed out that some other elements are also present besides Fe, and Ga in the spectrum due to incomplete etching and Au sputtering before SEM measurement. Furthermore, STEM-EDX elemental mapping of Fe_3Ga nanowire showing clearly the homogeneous distribution of Fe and Ga in Fig. 3.

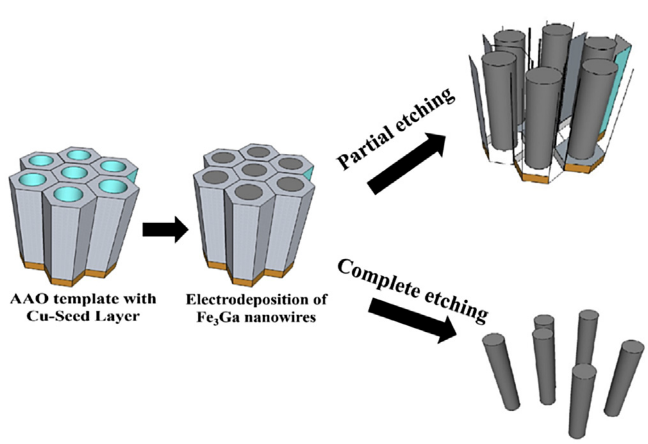

Fig. 1. Complete fabrication procedure of Fe_3Ga nanowires.

The XRD patterns of as-synthesized and post annealed nanowires are shown in Fig. 4. Only three strong peaks were observed from as-synthesized sample at ~42.6°, ~43.6° and ~80.8° corresponding to (111), (110) and (211) reflections originating $L1_2$ and A2 structures. Since Fe_3Ga is most likely crystal structure with possibility for DO_3 crystal phase and have X-ray reflections at (220) and (422) at 43.6° and 80.8°, respectively, since the alloy composition is $Fe_{75}Ga_{25}$. The number of diffraction peaks changed with annealing temperature. At temperature T2, the $L1_2$ structure is partially dominant along with DO_3 phase. As the temperature increases to T3, the intensity of strong disordered phase A2 or order phase DO_3 suddenly decreases and grains are textured along the stable $L1_2$ phase with small precipitates of DO3 phase. At higher temperature T4, the disorder phase along (110) and (211) again start to develop along the stable phase. From Fig. 4, we have the intensity ratio (I_{211}/I_{110} or I_{422}/I_{220}) = 0.247, 0.264 and 0.434 respectively for T1, T2 and T4 which depicts that Fe_3Ga is highly (110) or (220) textured for T1 and T2. For randomly oriented Fe powder, the intensity ratios of highly texture reflections are: $I_{200}/I_{110} = 0.50$ and $I_{211}/I_{110} = 0.80$ [17].

The grain size, D, can be estimated from the broadness of the diffraction peak using Debye–Scherer's relationship given by

$$D = \frac{k\lambda}{LCos\theta} \tag{1}$$

where λ is the wavelength of the Cu-Kα radiation, θ is the diffraction angle of highly textured/intensity reflection, k is the Scherer's constant which takes the value of 0.89 for spherical crystals with cubic symmetry [18,19] and L is the FWHM of the diffraction line corrected for the instrumental broadening. The grain size collected for as synthesized and post annealed nanowires are 31 nm, 27 nm, 40 nm and 44 nm respectively. This behavior is attributed to the recrystallization phenomena of grains and transformation from the disorder to the stable phase with raising of annealing temperature. Smaller crystallites are thermodynamically less stable than larger ones due to their high surface energy. Accordingly, they recrystallize such as in bubble coalescence during the longer delay times to attain more stable bigger crystallites [20].

We observed a non-hysteretic behavior for θ = 90°. This trend attributes that the shape anisotropy of Fe_3Ga NWs may induce a hard axis of magnetization for θ = 90° and is shown in Fig. 5. There are two stable orientations of magnetic moments that are directed parallel and perpendicular to the wire long axis. The slightly hysteretic nature for perpendicular measurement may be the effect of rather weak magneto-crystalline anisotropy. The magnetic spins are easily aligned along the magnetic field direction, so we get the squared hysteresis loop and high values of coercivity in comparison with coercivity and slightly hysteretic nature when field is applied perpendicular to the NWs axis. Fig. 5 reflects that the in-plane coercivity values enhancing with annealing treatment and attains maxima for stable $L1_2$ phase of Fe_3Ga alloy. It also clears that NWs having the mixed highly disorder (A2)/order (DO_3) phase have the low coercivity values as compared to the highly textured $L1_2$ phase. The gradual approach to saturation of the curve measured with the perpendicular field is an indication of magnetization rotation process. The angular hysteresis loops for as-synthesized nanowires shown in Fig. 6 also demonstrate that the value of saturation field increases from θ = 0° to θ = 90°. This behavior is typical of the systems with uniaxial anisotropy.

Fig. 7 elucidates that easy axis is parallel to the NWs long axis (highest values of SQ are at θ = 0°) for all the samples under investigation. Conventionally, the sign of the saturation fields difference $\Delta H_s = (H_s)_{para}$ - $(H_s)_{perp}$ gives the idea about the easy magnetization axis in case of NWs., where $(H_s)_{para}$ and $(H_s)_{perp}$ are