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Synthesis and low temperature magnetic measurements of polycrystalline Gadolinium nanowires

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ABSTRACT

Highly ordered and reproducible Gd nanowires have been reported via DC electrodeposition method. In this regard, templates with different diameters 100 nm, 50 nm and 20 nm were utilized. Shape morphology, reproducibly and homogeneity of nanowires were investigated with field emission scanning electron microscopy (FE-SEM). Morphological analyses confirm as required diameter, length and aspect ratio. Structurally, body centered cubic (bcc) phase was observed with Rietveld refinement of X-ray diffraction (XRD) patterns for all diameters. Magnetic investigations are including zero field cooled (ZFC) and field cooled (FC) and hysteresis measurement by using physical property measurement system (PPMS). These magnetic analyses of nanowires embedded in AAO templates predict mix phase with superparamagnetic and ferromagnetic clusters.

1. Introduction

The 4f rare earth elements with their notable characteristics associated with magnetic and structural properties have been studied extensively for more than 4 decades [1–8]. Gadolinium (Gd) is such a rare earth element that together with Fe, Co and Ni is one of the element which reveals ferromagnetic nature at room temperature. Gd has also been known as promising candidate for various applications including thermomagnetic generation at cryogenic temperature [9,10], magnetocaloric refrigeration [11] and in high magnetic moment applications [12].

In the past, scientists have worked to study magnetic properties of bulk, thin films and nanoparticles counterparts but still none of the research article published related to 1D Gd nanowires, according to best of our knowledge. The primary objective behind this research is to introduce, reliable, reproducible, relatively simple and less expensive approach to synthesise scalable growth of low temperature ferromagnetic Gd nanowires with different diameters by using chemical electrodeposition method. Second object is focused, particularly, to test diameter dependent magnetic measurements of Gd nanowires.

2. Experimental methods

In the beginning of the experiments, pure Aluminium foils (99.999%) with thickness of 5 cm × 3 cm × 0.5 mm were utilized as starting materials and subsequently washed with deionized water to remove dust particles on its surface. After cleaning, AAO templates as a function of different diameters were prepared in the presence of oxalic bath to generate cylindrical pores with different diameters by using two step anodization process [13–15]. Later on, to make template conducting, one side of it was coated with 100 nm thick Au by using sputtering machine. Chemical electrodeposition method was employed to fabricate nanowires using three electrode system. Synthesis process was continued with 1 M Gadolinium nitrate [Gd $(NO_3)_3{\cdot}6H_2O$] in aqueous solution at room temperature. In addition, bias voltage of −1.0 V was applied for nanowires with different diameters under continuous stirring. All experiments were performed without the presence of any reducing agents or surfactants.

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Dimension and shapes of Gd nanowires were investigated using Hitachi S-4800 Field Emission Scanning Electron Microscope (FE-SEM). Elemental analyses were employed with energy dispersive X-ray spectroscopy integrated with SEM. The structure of magnetic samples was assessed by D-MAC 2400 Rigaku X-ray diffraction (XRD). 9T Oxford designed physical property measurement system (PPMS) was used for magnetic response measurements.

3. Results and discussion

Since, Gd nanowires are embedded in AAO template via template assisted methods. Therefore, to explore the shape and dimensions of nanowires, sample preparation is required. It is necessary to etch template using wet chemical methods. Etching parameters including, atmospheric temperature, etchants and time are highly imperative. In present case, morphology of Gd nanowires is taken after dissolving AAO template in 1 M NaOH at 70 °C for 1 h. Morphology was analysed with FE-SEM after wet chemical etching of AAO templates. Fig. 1(a–c) represent microscopic images of nanowires which depict that nanowires are clean, highly dense and homogeneous. It can also be observed that nanowires predict smooth, dense, ordered and uniform behaviour. The high magnification images of nanowires are represented in their corresponding insets where it can be depicted that average length of nanowires is 8–10 μm and average diameters are 100 nm, 50 nm and 20 nm (Fig. 1(a–c)) which in turns are related with pore size of AAO templates. The micrographs also show that almost all of the template pores were filled with Gd. It is worth to mention that deposition rate and length of Gd nanowires can easily be controlled as a function of deposition time.

Diffraction patterns for Gd nanowires recorded at room temperature with variation in wire diameter are shown in Fig. 2. The peaks appearing at $2\theta = 30.9^\circ$ corresponds to strongest intensity peak, representing direction of preferred orientation of nanowires along (0 0 2) plane. It is worth to note that there are no oxide reflections has been observed in the patterns. The obtained diffraction patterns for all samples show nearly same peaks however, it has been observed that with decrease in wire diameter full width at half maxima (FWHM) of peaks becomes broader resulting smaller crystallite size. In addition, Rietveld refined patterns for Gd nanowires are achieved using Full-Proof software. Refinement is mainly performed for determination of unit cell parameters that are 4.061 Å, 4.064 Å, and 4.068 Å for 100 nm, 50 nm and 20 nm diameter nanowires, respectively. The Bragg reflections of XRD

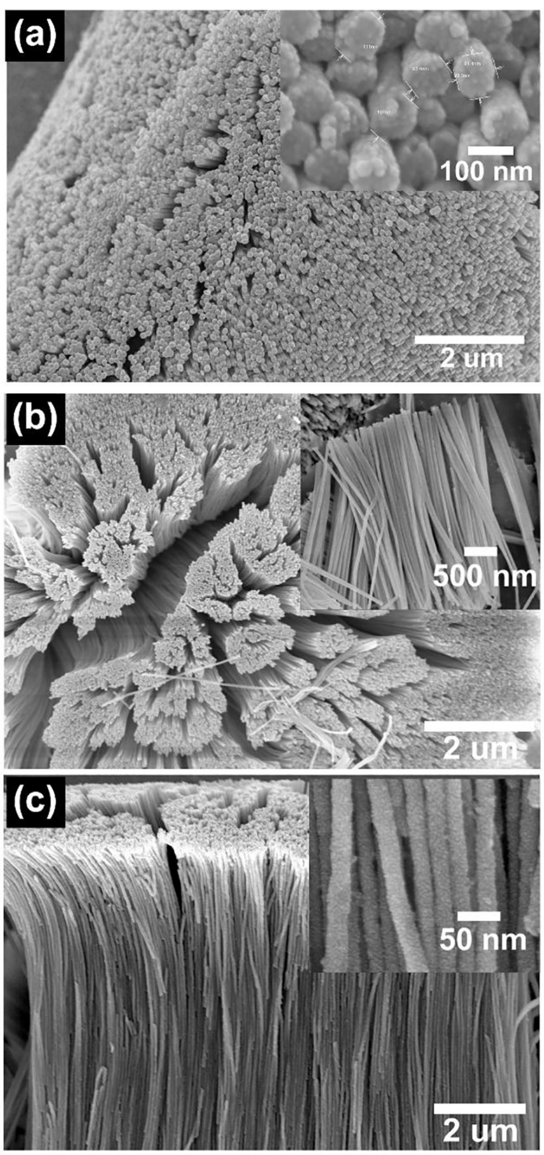

Fig. 1. Field emission scanning electron microscope (FE-SEM) images of Gd nanowires taken after dissolving AAO template in 1 M NaOH at 70 °C: Gd nanowires with diameter of (a) 100 nm, (b) 50 nm and (c) 20 nm. whereas their corresponding insets depict clear overview of nanowires morphology and size.

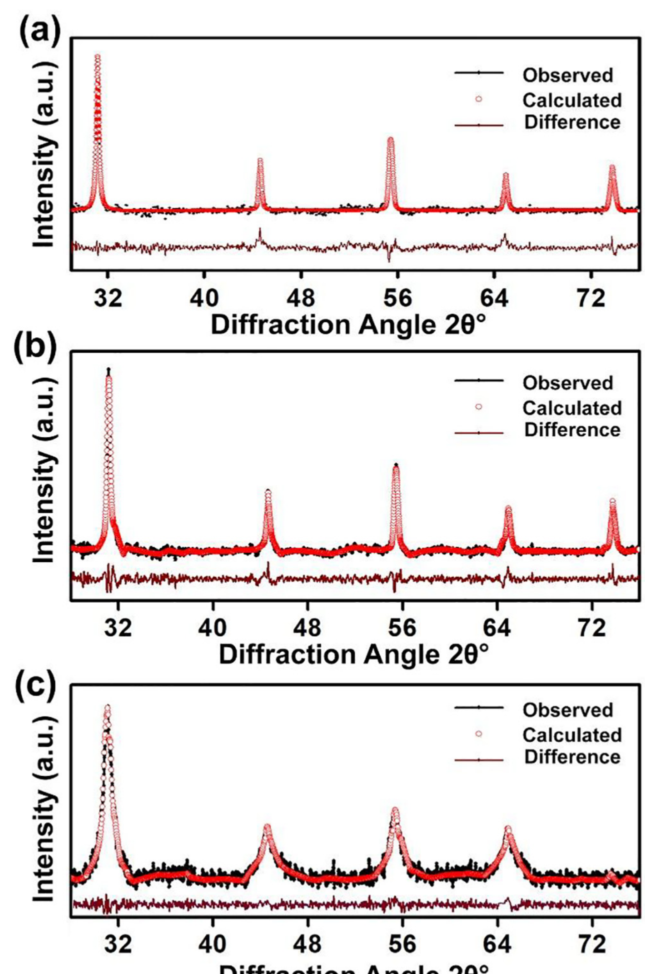

Fig. 2. XRD patterns with Rietveld refinement for Gd nanowires with variation in wire diameter (a) 100 nm, (b) 50 nm and (c) 20 nm.