Journal of Magnetism and Magnetic Materials 321 (2009) 1142–1147

Contents lists available at ScienceDirect

Journal of Magnetism and Magnetic Materials

journal homepage: www.elsevier.com/locate/jmmm

Preparation and FMR analysis of Co nanowires in alumina templates

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ARTICLE INFO

Article history:
Received 7 May 2007
Received in revised form
11 July 2008
Available online 8 November 2008

PACS:
75.60.−d
75.30.Gw
76.50.+g
75.60.−d

Keywords:
Co Nanowires
Template synthesis
Magnetostatic interactions
Magnetic anisotropy
Ferromagnetic resonance (FMR)

ABSTRACT

Magnetic and structural properties of a high aspect ratio Co nanowire (NW) array electrodeposited in free-standing porous alumina template with a pore diameter of ~200 nm are studied. Considered collectively, X-ray diffraction analysis, magnetometer and ferromagnetic resonance (FMR) measurements indicate that both the *c*-axis of crystal structure and the easy axis of magnetization are aligned preferentially perpendicular to the NW axis. The FMR spectra are characterized with very broad (a few kG) breadths and exhibit asymmetric shape in low field region due to under-saturation effects. Surprisingly, FMR spectra also revealed the presence of a spin-wave mode (SWM) as the applied field direction approached parallel to the film plane, i.e. perpendicular to the NWs. A brief discussion on this observation is provided. Further, characteristic magnetic parameters of the studied NW array were obtained by fitting the field angle-dependent FMR spectra and resonance field by using an analytical model that considers various factors affecting the total anisotropy.

1. Introduction

Ferromagnetic nanowire (NW) arrays have attracted considerable interest in recent years owing to their potential applications in magneto-electronic, thermoelectric, and field-emitting devices, (perpendicular) magnetic data storage and biological applications [1–4]. Various approaches ranging from *top-down* electron beam, X-ray, and laser-interference lithographies to *bottom-up* template and self-assembly (chemical) methods have been used to prepare metal NWs [5–11]. Among these, the template method stands out as it can deliver high-density NW arrays with controllable parameters (e.g., length, diameter, crystallinity, etc.) and is highly cost-efficient. Template synthesis of metal NWs have usually been carried out by electrodeposition into porous alumina and polycarbonate membranes.

The magnetic properties of NWs have been investigated by using a variety of techniques such as the magneto-optical Kerr effect, vibrating sample magnetometer (VSM), magnetic force microscopy, torque magnetometers, Brillioun light scattering, scanning Hall microscopy, neutron diffraction, superconducting quantum interference device, alternating gradient magnetometer, scanning magnetoresistance, spin-polarised scanning tunnelling microscopy and ferromagnetic resonance (FMR) techniques [1,12–18]. Of particular importance is that, the FMR technique can provide valuable information on the magnetization, magnetic anisotropy, the Landé splitting factor, *g*-value and spin–spin relaxation times, as well as the damping in magnetization dynamics, the structural quality and magnetic (in)homogeneity of the NW films through observation of the line-width characteristics of resonance signals [19,20]. FMR experiments have been performed for some NW arrays with wire diameters ranging from 12 to 500 nm [1,21–24]. Spin-wave modes (SWMs) in magnetic NWs have been investigated by different researchers [25–30]. The FMR and magnetization show that magnetostatic/dipolar interactions are important in determining the final anisotropy field for different diameter NWs [22–25,28,31–38].

In hexagonal close packed (hcp) Co energy density of magnetocrystalline anisotropy (5×10^6 erg/cm^3) is comparable to that of shape anisotropy (6×10^6 erg/cm^3) [40], and since the easy axis of magnetocrystalline anisotropy is parallel to the *c*-axis in this structure, it has recently become fashionable to try to control the *c*-axis and ultimately the effective anisotropy in (hcp) Co NWs via modifying the sample preparation conditions (see e.g., Refs. [41,42]).

In the present work, we fabricated high aspect ratio (length/diameter) Co NW arrays with an average NW diameter of 200 nm

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doi:10.1016/j.jmmm.2008.10.039

and density of $\sim 10^9$ per cm^2, and studied their magnetic properties by VSM and FMR techniques at room temperature and as a function of external field direction. In order to have an unaltered microstructure, a single sample with 20-μm-long NWs is used in X-ray diffraction (XRD) and in all magnetic measurements. Effective anisotropy, filling factor, inter-wire dipole–dipole interactions as well as a resonant SWM are reported.

2. Experimental details

Cobalt NWs were prepared by dc electrodeposition into the pore channels of 45-μm-thick commercial (Whatman Anodisc) porous alumina films with ~200 nm pores. The deposition procedure (illustrated in Fig. 1) followed the method described in Ref. [43]. Briefly, first a conformal metal film is coated on one side of the membrane (to serve as the working electrode) via combination of Au sputter-deposition and Cu electrodeposition (steps 1 and 2). The pores were then thoroughly wetted with deionized water using ultrasonic agitation. These approaches ensured homogeneous filling of the pores with Co NWs (*vide infra*). The NWs were electrodeposited (step 3) in a conventional three-electrode cell at −1.2 V (vs. Ag/AgCl) and using an electrolyte containing 100 g/l cobalt sulphate and 50 g/l boric acid at pH = 3. A Philips XL30 scanning electron microscope (SEM) coupled with an energy-dispersive X-ray (EDX) spectroscopy equipment was used for microstructural investigations. Cross-sectional SEM images were taken simply by cleaving of alumina templates containing Co NWs. Template filling efficiency was checked by collecting topographic images of surface-polished alumina/Co NWs composite samples. For this purpose, templates containing NWs of >20 μm length were selected. At first mechanical grinding was employed to remove the unfilled (20–30 μm thick) portion of alumina template, i.e. till the NWs were exposed, and then the sample surface was fine-polished using 1 μm-sized diamond particle suspension. Such surfaces also proved very useful in the assessment of inner pore structure of the template. Crystallinity and phase of the NWs were checked with θ–2θ XRD (Cu Kα).

Hysteresis loops were recorded using an ADE VSM. The FMR spectra were collected by an X-band (~9.8 GHz) Bruker EMX spectrometer at different angles of applied field with respect to the film plane, in order to attest sample homogeneity (magnetic or structural) and to deduce magnetic parameters.

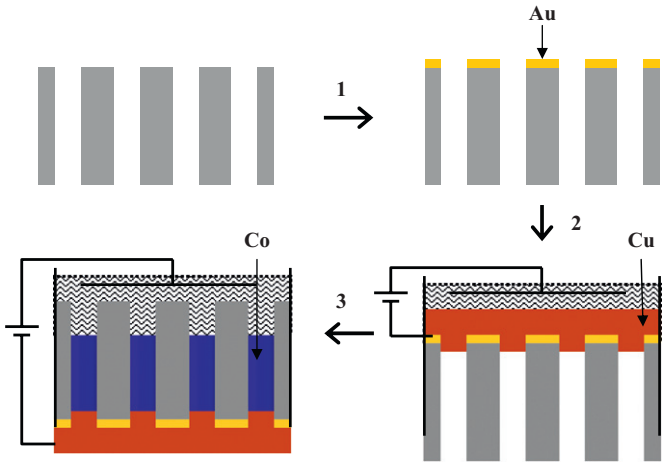

Fig. 1. Nanowire (NW) deposition scheme used in this work: (1) Au thin-film sputter-deposition on alumina template, (2) Cu electrodeposition and sealing of the pores and (3) Co electrodeposition and *bottom-up* growth of Co NWs inside the pore channels.

3. Calculation of the resonance field and modelling of the FMR spectra

The following theoretical considerations were used to calculate the resonance field and to fit the (main) FMR band. The sample geometry, relative orientation of the equilibrium magnetization ***M***, the applied DC magnetic field ***H*** and experimental coordinate systems are shown in Fig. 2. The free energy density equation for homogeneously magnetized films (here the NW array) can be written as

$$E = -MH(\sin\theta\,\sin\theta_H\,\cos(\phi-\phi_H)+\cos\theta\,\cos\theta_H)+K_{eff}\sin^2\theta \quad (1)$$

where (θ,ϕ) and (θ_H,ϕ_H) are the angles for magnetization (***M***) and applied field vector (***H***), respectively, in spherical coordinates. The first term represents the Zeeman energy of the sample in the applied magnetic field (***H***). The second term is the effective anisotropy energy, characterized by the effective uniaxial anisotropy parameter [23,24],

$$K_{eff} = \pi M^2(1-3f)+K_U \quad (2)$$

Here the first term is due to the magnetostatic energy of perpendicularly arrayed NWs [22,23,44] and constant, K_U, takes into account some additional second-order uniaxial anisotropy [21] with the symmetry axis along the wire direction. Parameter f is the filling factor (f = metal filling fraction in the matrix structure), determined for a perfectly (hexagonally) ordered NW array as

$$f = \frac{\pi}{2\sqrt{3}}\frac{d^2}{r^2} \quad (3)$$

where d is the average NW diameter and r is the center-to-center interwire distance (see Fig. 2). Note that as f increases, the magnetization easy axis of the NW array changes direction from the parallel to the perpendicular to wire axis. The equilibrium values of polar angles θ for the magnetization vector ***M*** are obtained from static equilibrium conditions of $E_\theta = \partial E/\partial\theta = 0$

$$E_\theta = -MH(\cos\theta\,\sin\theta_H\,\cos(\phi-\phi_H)-\sin\theta\,\cos\theta_H) + K_{eff}\,\sin 2\theta = 0 \quad (4)$$

Using the Landau–Lifshitz dynamic equation of motion for magnetization with the Bloch–Bloembergen damping term [16,19,20,45,46], the resonance field was obtained from the

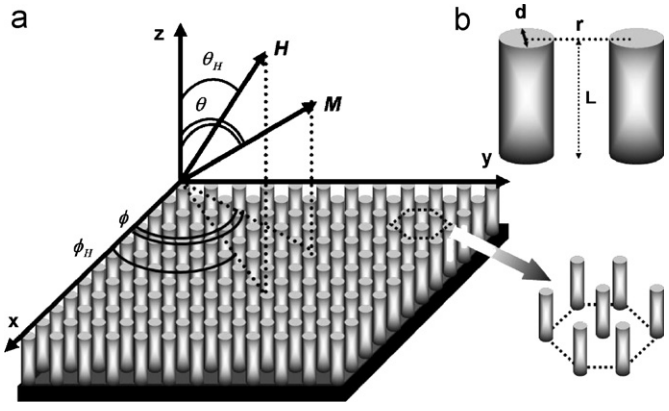

Fig. 2. (a) Sample geometry and relative orientations of equilibrium magnetization ***M*** and the dc components of external magnetic field ***H***. (b) Sample parameters used in calculating the filling factor, f, and interwire magnetostatic interaction field.