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Large-scale synthesis of uniform nickel nanowires and their characterisation

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Nickel nanowires were synthesised using template synthesis technique. Commercial polycarbonate membranes having pore diameter 100 nm were used as templates. Scanning electron microscopy depicted that the diameter and length of nanowires are same as that of pore diameter and thickness of the template membrane. X-ray diffraction and energy dispersive X-ray fluorescence recommended that the nanowires are of pure nickel metal rather than a nickel compound. Room temperature magnetic characterisation was performed using a vibrating sample magnetometer. The value of saturation magnetisation was observed as different for nickel nanowires with different aspect ratio.

Keywords: template synthesis; magnetic nanowires; hysteresis curve; electrodeposition

PACS Codes: 62.23.Hj; 68.37.Hk; 75.75.+a

1. Introduction

With regard to the growing needs for high-performance data storage devices, at present enormous efforts are being put into the development and study of novel magnetic materials [1–5]. Therefore, various kinds of investigations on magnetic nanowires are nowadays attracting keen interest [6–8]. Nanowires are always measured as single-domain magnets where shape anisotropy of the entire wire governs the magnetism [9–11]. The high ordering, collectively with the inherent character of nanowires may give rise to wonderful cooperative properties of fundamental and technological importance in a broad range of relevant areas [12,13].

Template synthesis is an elegant technique for the fabrication of nanostructured materials. This is an extremely constructive approach for the preparation of arrays of nanowires with controlled aspect ratio, because the organisation of desired material is adaptable through selecting different host template membrane with different pore shape (cylindrical, conical and double-conical) and pore diameter [14–16]. It may be considered as a substitute for usual lithography techniques. At present, anodic alumina

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and polycarbonate membranes are mainly used as templates. Polycarbonate template is better than anodic alumina due to different reasons. First, after template synthesis polycarbonate template can easily be dissolved using dicholoromethane compared to anodic alumina using sodium hydroxide. In the case of polycarbonate, membrane pore size can be made as small as 10 nm by controlling the etching conditions. Polycarbonate membrane is flexible but anodised alumina membrane is very brittle. After depositing, the magnetic material in polycarbonate may be used as flexible interconnects as well as flexible data storage device according to space available [17]. In this work, porous polycarbonate membrane, which contains a large number of cylindrical pores with a narrow size distribution, has been used to fabricate arrays of nickel nanowires. The filling of nickel inside the pores is carried out by electrochemical deposition which is attracting large interest, because it works under normal environmental conditions, does not need costly equipment and may be used in geometries where conventional deposition techniques would be unsuccessful [6].

2. Experimental details

Arrays of nickel nanowires were fabricated by the electrodeposition of Ni in the pores of commercially available high-quality track-etched polycarbonate membranes having pore size 100 nm and pore density $10^7/cm^2$ (Whatman Co.). The growth of nanowires was carried out by electrodeposition at room temperature under constant potential (1.2 V) using a conventional two-electrode cell. The electrolyte solution consisted of 80 g/L $NiSO_4$ and 30 g/L H_3BO_3. De-ionised water with resistivity $\sim$18 MΩ was used to prepare the solution. Pure nickel rod was used as anode. Boric acid was mixed in the plating solution so as to decrease the local increase of pH in the surrounding area of the cathode, owing to its buffering action [2]. The filling of the pores was scrutinised by evaluating the plating current. The deposition process was blocked as soon as over-deposited caps began to form on top of the emerging nanowires, which was exposed by a rapid rise in plating current. Using this development system, well-defined straight wires were fabricated of length equal to the membrane thickness (6 and 10 μm). These template-synthesised Ni nanowires have

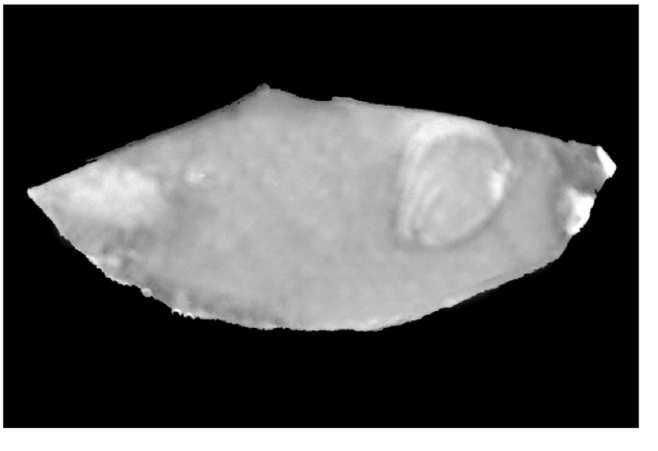

Figure 1. Photograph of polycarbonate membrane with embedded nickel.

moderately little surface roughness, nearly homogeneous circular cross-section all along the wire length [4].

Four samples were cut out from the nickel filled membrane for performing different characterisations. One of the samples is shown in Figure 1. For the morphological characterisation of the nickel nanowires by means of scanning electron microscopy (SEM), nickel nanowires, were observed by dissolving the polycarbonate matrix in di-choloromethane. The cleaned and dried samples were mounted on the specially designed aluminium stubs with the help of double adhesive tape and viewed under Jeol 6100 Scanning Electron Microscope available at CIL, Panjab University, Chandigarh, at an accelerating voltage of 30 kV.

X-ray diffraction (XRD) measurements were carried out using 'Panalytical X'pert Diffractometer' equipped with Cu-Kα in $\theta/2\theta$ mode available at CIL, Panjab

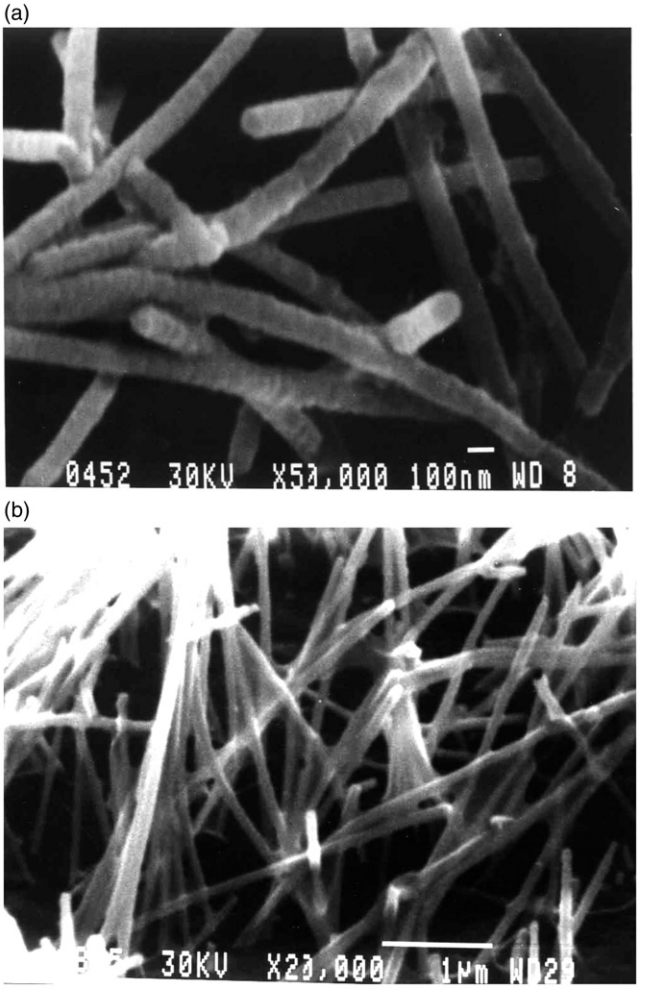

Figure 2. SEM Micrograph of nickel nanowires having aspect ratio (a) 60 and (b) 100.

University, Chandigarh. Elemental composition of nanowires was examined using energy dispersive X-ray fluorescence (EDXRF). Magnetic properties of nickel nanowires were studied at room temperature using vibrating sample magnetometer available at Institute Instrumentation Centre, Indian Institute of Technology, Roorkee.

3. Results and discussion

Template synthesis of nickel nanowires with a high aspect ratio (~60 and 100) has been carried out into the nanochannels of porous polycarbonate template. The achieved array has been observed as the replication of the channels of the template as shown in SEM

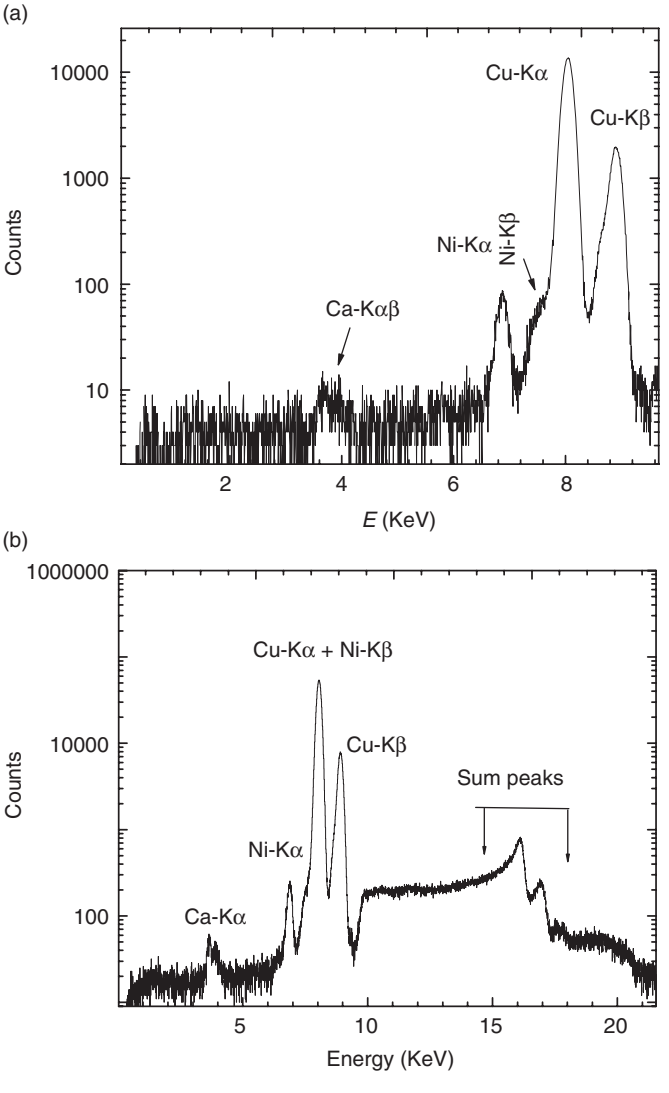

Figure 3. EDXRF graph for nickel nanowires up to energy (a) 10 KeV and (b) 21.5 KeV.

micrographs (see Figure 2a for sample 1 and Figure 2b for sample 2), and the diameter and length of the nanowires are the same as that of the pore diameter and thickness of the template membrane respectively. In the SEM micrograph, nanowires are appearing as randomly aligned. This randomness is due to the dissolution of the host membrane in dicholoromethane.

Figure 3(a) shows an EDXRF spectrum of nickel nanowires embedded in the polycarbonate taken using X-ray tube (up to energy 10 KeV), and Figure 3(b) represents the same up to energy 21.5 KeV taken using an Am source. An EDXRF spectrum of nickel nanowires authenticate the presence of nickel with no indication corresponding to oxygen. The large peaks due to copper are also seen in an EDXRF plot. These peaks are due to the copper that was used as target material. The presence of calcium may be due to the handling of sample with naked hands during EDXRF characterisation.

An XRD pattern (Figure 4) of the nanowires still embedded in the membrane was observed using Panalytical X'Pert Diffractometer with Cu-Kα radiation (wavelength = 1.54 Å), and that the nickel (111) peak was found to be most intense. This implies that the nanowires are growing with a favoured orientation of the (111) plane in the direction of growth. X-ray peaks correspond to face-centred cubic nature of template synthesised nickel nanowires. Both the diffraction pattern and elemental analyses recommend that the nanowire is pure nickel metal rather than a nickel compound.

The magnetic properties of nanomaterials have been supposed to be highly dependent on the sample shape, crystallinity, magnetisation direction and so on [1–8]. The magnetic properties of the Ni nanowires were studied by means of magnetisation measurements carried out at room temperature using a vibrating sample magnetometer which permits the

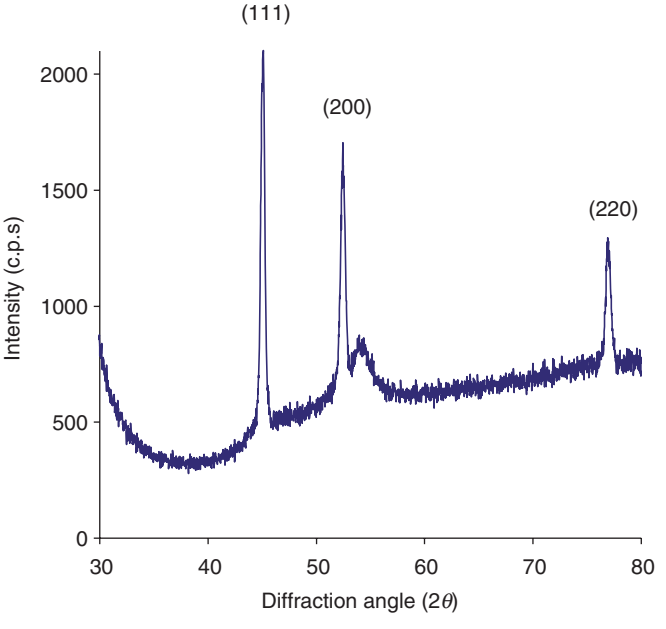

Figure 4. XRD pattern for nickel nanowires.

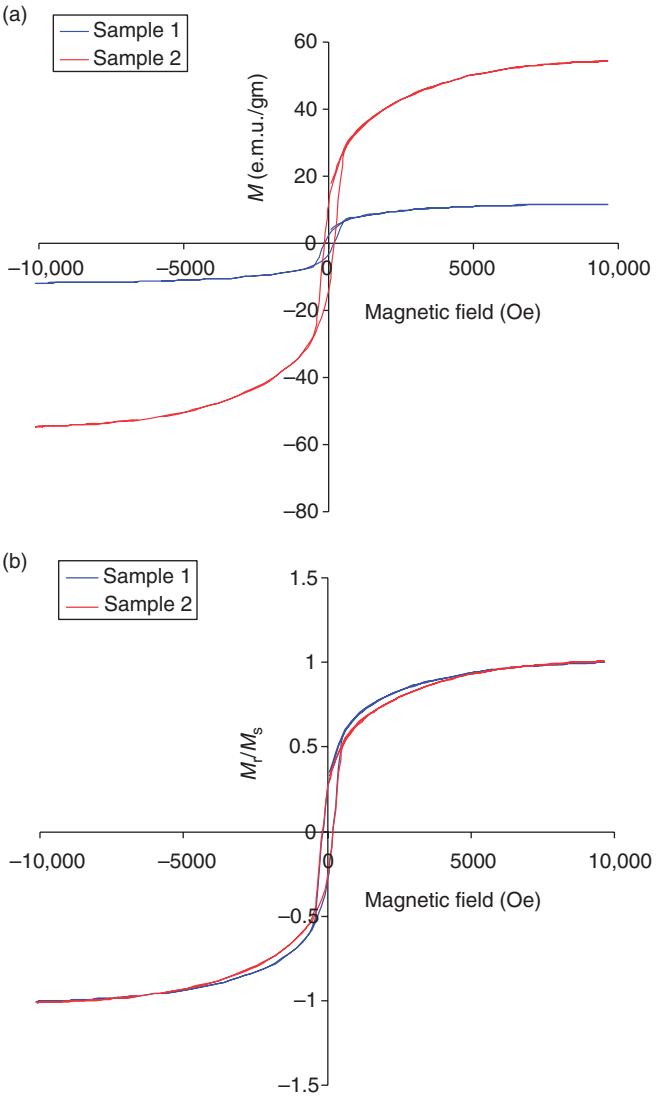

Figure 5. Hysteresis loops for nickel nanowires (a) between magnetisation and applied field and (b) between M_r/M_s and applied field.

determination of magnetic parameters of the arrays as remanence, coercivity or field to reach saturation. The hysteresis loop (Figure 5a) of the Ni samples measured at room temperature (field is applied parallel to the nanowires) shows a ferromagnetic behaviour with saturation magnetisation (M_s), remanent magnetisation (M_r) and coercivity (H_c) values of *ca* 11.73 emu/g, 2.45 emu/g and 180 Oe, respectively, for sample 1 (nanowires having aspect ratio 60) and 54 emu/g, 12 emu/g and 182 Oe, respectively, for sample 2 (nanowires having aspect ratio 100). Figure 5(b) represents hysteresis loops for both samples between M_r/M_s and magnetic field.

Nickel nanowire arrays may exhibit an easy axis which is parallel to the nanowire, and the coercive field is lowest in that direction [18–20]. In our samples, the number of parallel magnetic nanowires is enormously high $\sim 10^7/cm^2$, and wires have analogous value of magnetisation and coercive field. The small value of coercivity along the easy axis can be examined using a simple model [21] which considers magnetic dipolar interactions among nanowires. The dipolar interactions among magnetic nanowires is enhanced when the magnetic field is applied in the direction of the easy axis of the nanowires, and the field originating from every wire supports the reversal of magnetisation [22,23]. The saturation magnetisation for sample 2 is higher compared to sample 1. This may be due to the reason that the nanowires with higher aspect ratio also have higher value of saturation magnetisation, and consistency with the spin is aligned along the long axis of the magnetic nanowire [24]. The comprehensive studies of the magnetic properties of these magnetic nanowires are in development.

4. Conclusion

Nickel cylindrical nanowires with lengths of 6 and 10 μm and a diameter of 100 nm were grown by electrodeposition in the pores of track-etched polycarbonate membranes. Template synthesis proves to be an ideal technique for the fabrication of magnetic nanowires. It can be concluded from structural studies that nickel (111) planes run parallel to the axis of the nickel nanowire. Ni nanowires have easy axes along the wire axis due to the shape anisotropy. Dipolar interactions among dense parallel magnetic nanowires modify the magnetic behaviour resulting in the decline in coercive field in the easy axis of the nanowire. The value of saturation magnetisation was observed as different for nickel nanowires with a different aspect ratio.

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