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SEM morphology and XRD characterization of Ni microstructure arrays synthesized by dc electrodeposition in porous polycarbonate templates

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Research on magnetic micro/nanostructures has led to the exploration of novel physics and material properties at reduced physical dimensions [1–3]. There is considerable technological interest in the fabrication of arrays of high aspect ratio structures (e.g., magnetic nanowires) synthesized by electrodeposition for use as sensors and ultra high density information storage [4–6]. Magnetic properties are strongly influenced by the dimension and crystal properties, which again depend on the physical structure of the templates and the growth mechanism of the wires. The morphological study of such structures produced through electrochemical methods and of replicas of etched tracks in NTFs used as templates, has two-fold purpose. One, it provides the finest and critical details of the geometry and dimensions of microstructural constituent elements and the second, as a by product, it enables the study of the various aspects of interaction of a nuclear particle with given material leading to formation of tracks in NTF. It is well known that parameters, which control the size and shape of tracks in NTFs, include the nature of the material; the ion beam and energy deposition rate; pre, post-irradiation storage and environment and the etching conditions. Due to their parallel porous structures ion track polymeric membranes have become good templates for the electrochemical deposition of the highly aligned nano/microstructures [4–10].

With the trend towards the miniaturization of material in the microelectronics industry and the demand for low expenses, high performance electrodeposition has become the potential candidate in many manufacturing technologies [9, 10]. One drawback associated with electrodeposition is, however, the requirement of conducting substrate and the limited number of metals, which can be deposited from aqueous solutions. The properties of electrodeposition are determined by many factors including the electrolyte composition, pH, temperature and agitation, the applied electrodes potential and or the current density. A number of promising results have already been reported using the electrodeposition method as an alternative to the above mentioned time and cost consuming vacuum-based deposition processes. However there is now an increasing interest in the use of lithography and related techniques to fabricate nanotemplate structures as the dimensions, pore location and interpore spacing can be carefully controlled, but the aspect ratio of the nano/microstructures generated using this technique is very small as compared to the nano/microstructures generated using template synthesis technique.

In this paper we present a dc electrodeposition process to produce Ni microstructure arrays in porous polycarbonate templates from a nickel sulphate solution containing boric acid. SEM scanning was employed to investigate the morphology of microstructures. Because the membranes used contain cylindrical pores of uniform diameter, crops of monodisperse microstructures of the desired material, whose dimensions can be carefully controlled, are obtained.

Track etch membranes in the form of nuclear track filters have emerged as a spin-off from solid state nuclear track detectors (SSNTDs)-solid dielectric materials capable of storing tracks of energetic, heavily ionizing ions which can subsequently chemically amplified for optical observations as pores or channels of well defined geometry and pore density [3–6]. The pore size which is controllable, may range from few nm to mm.

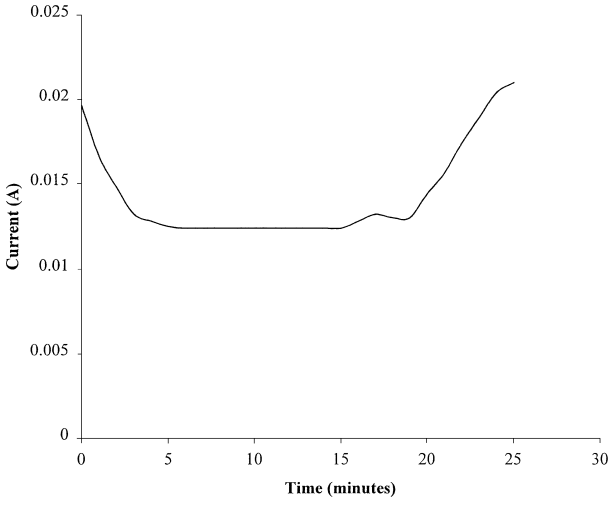

Figure 1 Variation of current with time during Ni electrodeposition through pores in polycarbonate.

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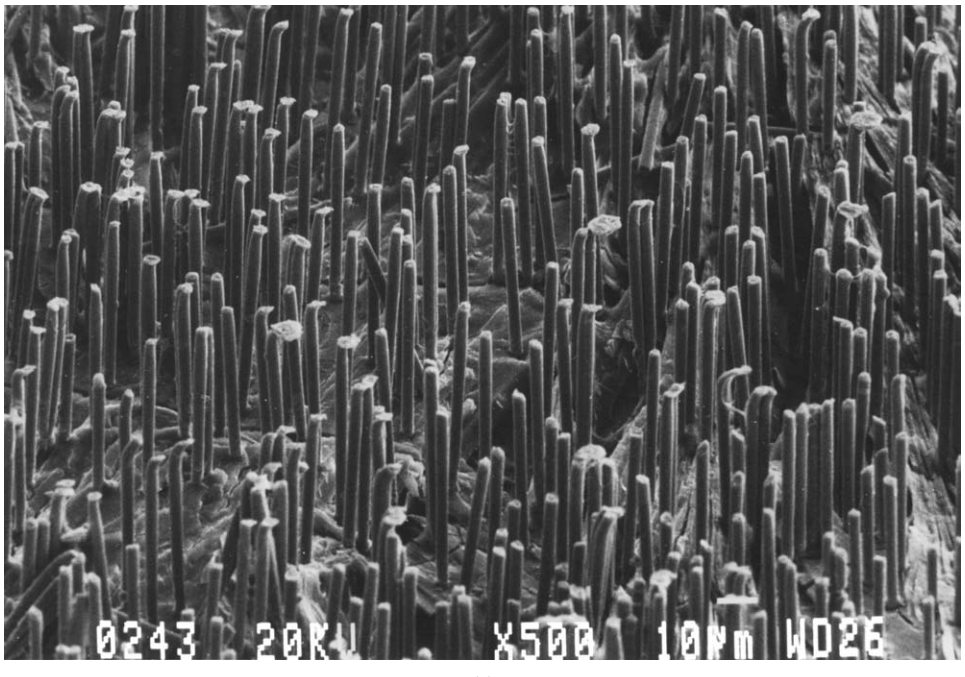
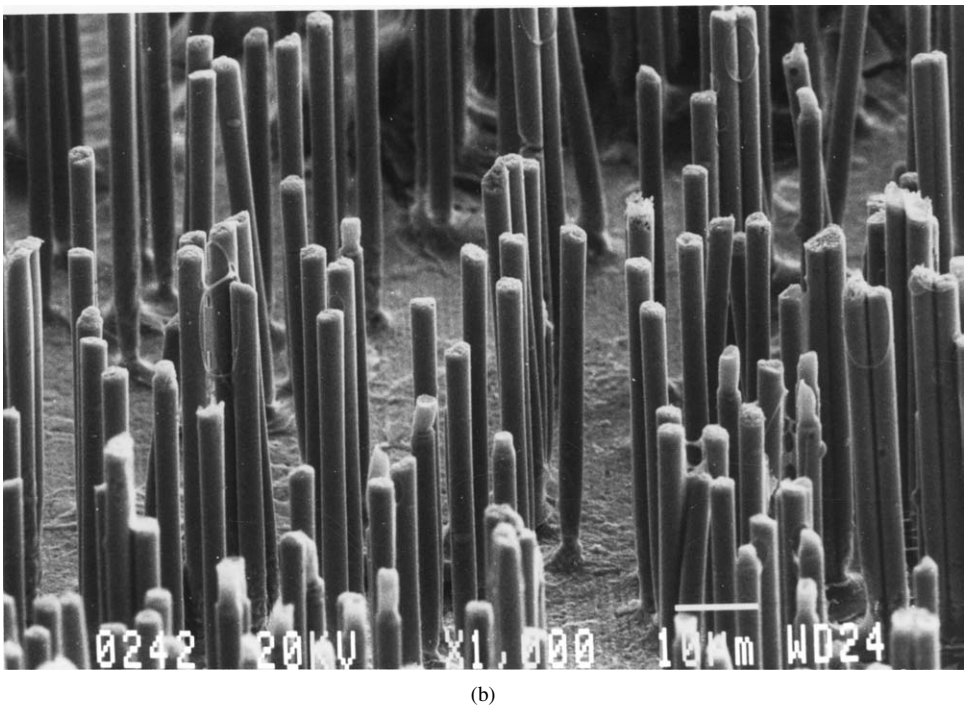
NTFs have been put to numerous applications besides their use in the synthesis of nano/microstructures.

The underlying principle of the technique (template synthesis) used is well known; it is an electrochemical process in which metallic ions in a supporting solution are reduced to the metallic state at the cathode which, if closely covered by an NTF, would lead to the growth of the electroplated film: an embodiment of micro/nanostructures depending upon the size of the templates [4–6]. In general, a suitable cell design is required and the lay-out design of such a cell along with other relevant details of the technique has been used previously [4]. Two electrode electrochemical cell was used for copper deposition in the pores of the template membrane.

Porous polycarbonate templates with pore size 3.5 μm were grown by irradiating 60 μm thick polycarbonate foils with ^{238}U ions (13.64 MeV/n) having fluence of the order of 10^6 ions/cm^2 from GSI, Darmstadt, Germany and followed by chemical etching in 6N NaOH solution at 60 °C for 25 min. Ni was cathodically deposited at a constant voltage of 1.2 V for 15 min in a two electrode cell having pure nickel as anode and copper tape having conductive adhesive as cathode at 40 °C. The electrolyte solution consisted of 60 g/l $NiSO_4$ and 30 g/l H_3BO_3. Deionized water with resistivity ∼18 MΩ was used to prepare the solution. The pH of solution was kept 2.90 using H_2SO_4. Fig. 1 shows variation of current with time during Ni electrodeposition through pores in polycarbonate. After



(a)



(b)

Figure 2 (a) and (b) SEM micrographs of Ni microstructures having diamater 3.5 μm and length 40 μm from different angles.

the deposition the polycarbonate templates with Ni microstructures were immediately removed from the electrolyte, first rinsed with double-distilled water and ethanol, finally dried in dry air at room temperature and subjected to further analysis. The porous polycarbonate membrane was removed by dissolving it in dichloromethane for 10 min and washing several times with double-distilled water.

The cleaned and dried samples were mounted on specially designed aluminum stubs with the help of double adhesive tape, coated with a layer of gold palladium alloy in Jeol, Fine Sputter JFC 1100 sputter, coater and viewed under Jeol, JSM 6100 scanning microscope at an accelerating voltage of 20 kV. The SEM image of the Ni microstructures prepared by dc electrodeposition in the porous polycarbonate templates with pore size of about 3.5 μm are shown in Fig. 2a, b. X-ray diffraction measurements were carried out a Philips PW 1710 diffractometer with Cu-K_α in $\theta/2\theta$ mode. Three peaks were observed corresponding to three planes (111), (200), (220). The XRD analysis of the samples has revealed that the nickel microstructures are polycrystalline. The wires deposited at 40 °C showed a strong (220) preferred orientation. Fig. 3 shows XRD diffractogram for the electrodeposited Ni microstructures having diameter 3.5 μm. XRD diffractogram confirms the polycrystalline nature of Ni microstructures.

In conclusion template synthesis is an elegant technique to prepare metallic (ferromagnetic) nano/microstructures. XRD diffractogram depicts the polycrystalline nature of Ni microstructures. It must be noted that the metal gets deposited layer by layer on the pore walls. If the process is interrupted before the pore gets completely filled, then hollow tubules may be produced. The NTFs were etched so as to produce micropores of suitable dimensions so as to reveal the morphological details viz. diameter, length, shape (conical, cylindrical etc.) which are of interest for track profiling studies. This also facilitates the deposition of metals quite easier. These microstructures can be used as field ion emitters, micromagnets etc.

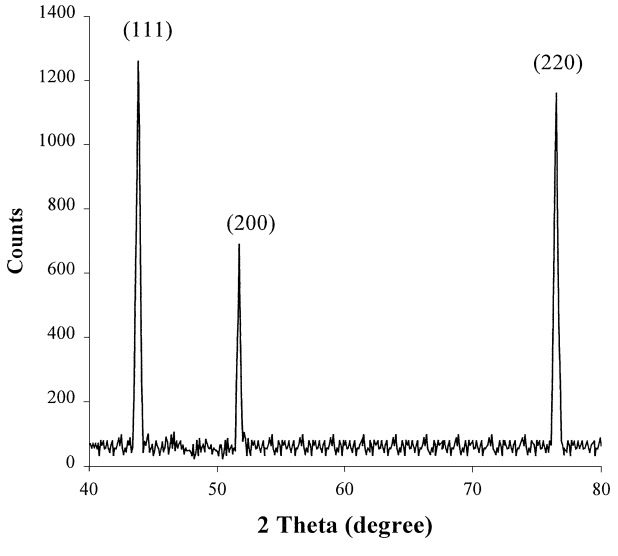


Figure 3 XRD diffractogram for the electrodeposited Ni microstructures having diameter 3.5 μm.

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