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Morphological, structural and optical characterization of nickel nanostructures

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Abstract The electrochemical template synthesis of high aspect ratio nickel nanocylinders in the track-etch membranes of polycarbonate having nominal pore size of the order of 80 nm is considered. The morphological and structural analyses have been carried out through scanning electron microscopy and X-ray diffraction respectively, while optical characteristics have been examined using photoluminescence setup. It has been observed that the fabricated nanostructures are crystalline in nature and their metallic characteristics do not change.

Introduction

During the last few decades, the fast technological development, promoted mainly by the microtechnology industry, has led to a progressive miniaturization of electronic devices. The production techniques employed in this field are well established and comprise of optical or electron-beam lithography, metallization, implantation and etching. While nowadays, the resolution limit is in the sub-micrometer range, new structuring techniques have to be developed when nanometric dimensions are encountered [1, 2]. Such new methods must also be capable of creating micro and nanostructures of higher aspect ratios, which are required, for example, for devices combining mechanical and microelectronic components (MEMS) [3, 4].

The large interest in nanowires [5–9] is based on their promising applications, for instance, as interconnects in future generations of nanometer-scale electronics [10], as emitters in field emission arrays [11, 12], or as constituents of magnetic storage devices [13]. At the same time, they are considered to be excellent objects for studying the fundamental physical phenomena such as electron transport, superconductivity [14], magnetoresistance [15], electron emission [12], or quantum-size effects [16]. Common approaches to Ni metallization include chemical vapour deposition (CVD), selective electroless deposition, sputtering and electroplating. Electrochemical deposition of Ni is the leading technology, since it has low costs, is fast, and is suitable for deposition in trenches of small dimensions and/or high aspect ratios [4, 17, 18].

Available techniques which are suitable for filling the pores include electrochemical and electroless deposition, polymerization reactions, sol–gel-template synthesis and high-pressure injection of a molten material. Compared to the other techniques, electrochemical deposition is more suitable for creating wires of high aspect ratios (length to diameter).

In this paper we present an electrodeposition process for producing Ni microstructure arrays in porous polycarbonate templates from a nickel sulfate solution containing boric acid. SEM was employed for investigating the morphology of fabricated microstructures. Because the membranes used had identical cylindrical pores of uniform diameter, the template synthesis resulted in crops of monodisperse microstructures of the electrodeposited material. Structural analyses have been carried out through X-ray diffraction, while optical characteristics have been carried out using photoluminescence setup.

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Fabrication and morphological characterization

For nickel deposition, 11 μm thick polycarbonate membrane with 80 nm diameter pores at a density of 10^8 pores cm^{-2} has been used. 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 [19]. The electrolyte solution consisted of 60 g/L $NiSO_4 \cdot 6H_2O$ and 30 g/L H_3BO_3. Deionized distilled water was used for preparing the solution. The pH of solution was kept 2.14 using H_2SO_4. After the deposition, the polycarbonate templates with nickel nanostructures were immediately removed from the electrolyte, first rinsed with double-distilled water and ethanol and then dried in air at room temperature. 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 coated with a layer of gold-palladium alloy in Jeol, Fine Sputter JFC 1100 sputter and subsequently mounted on aluminium stubs using double-sided adhesive tape. The samples thus mounted were then viewed under Joel, JSM 6100 scanning microscope at an accelerating voltage of 20 kV. Figure 1 shows the micrographs of the nickel nanostructures.

Structural characterization

In order to confirm the crystalline quality of the deposits, the membrane containing nascent nanostructures was peeled-off from the copper strip and X-ray diffraction of the deposited nanostructures was carried out using D/Max Rint 2000 Rigaku (Tokyo) X-ray diffraction machine using the copper characteristic wavelength of 1.5418 Å.

Three peaks were observed in the span ranging from 40° to 80°, as shown in Fig. 2. Lattice planes corresponding to these peaks have been identified by applying extinction rules, as given in Table 1. The ratios correspond to FCC lattice structure. Further, the large difference in peak intensities may be attributed to a high degree of texturing in the deposited nanostructures.

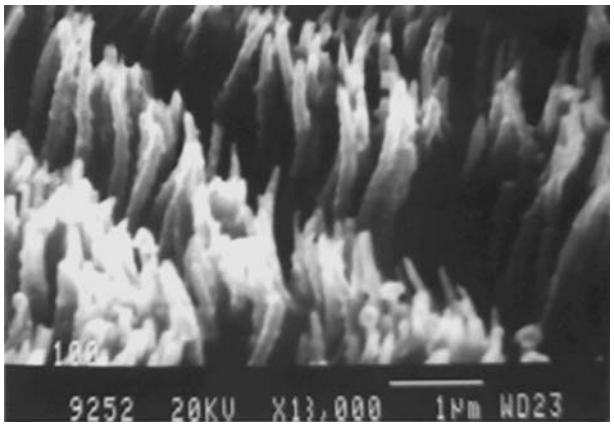

Fig. 1 SEM micrograph of 80 nm Nickel nanostructures

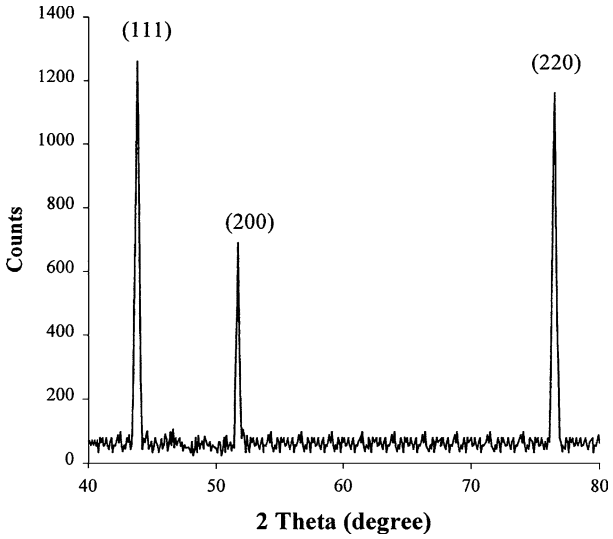

Fig. 2 X-Ray diffractogram for 80 nm Ni microstructures

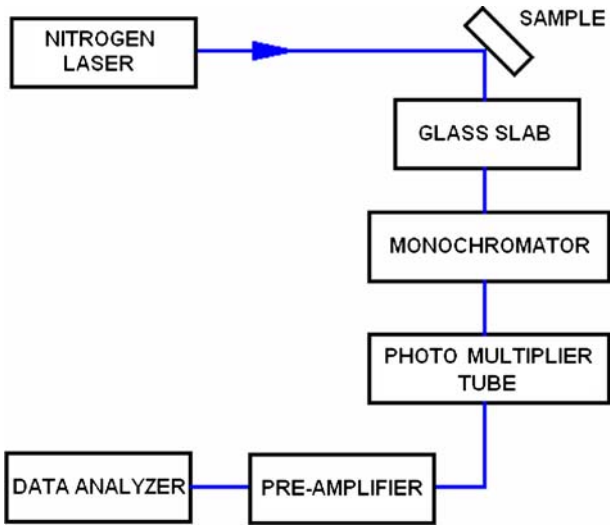

Fig. 3 Block diagram of experimental set-up for recording optical spectra

Table 1 Determination of lattice structure

2θ	Sin θ	$\sin^2 \theta$	Ratios	Normalized ratios	Lattice planes
44.24	0.37	0.14	1.00	3	(111)
51.80	0.44	0.19	1.33	$3.98 \approx 4$	(200)
76.03	0.62	0.38	2.66	$7.99 \approx 8$	(220)

Optical characterization

In the present work, we have used time-resolved laser spectroscopy through pulsed excitation method for investigating the effect of confinement on the fabricated Ni nanostructures in relation to photoluminescence. A nitrogen laser (10 kW, Ultraviolet) was used as the pulse excitation source, while the emission from microstructures was collected by a fast photomultiplier tube having a rise time of a few nanoseconds. An assembly of glass slab was employed in tandem with a monochromator to select the wavelength of the signal being recorded, while also filtering out any UV radiation that might be present in the signal being recorded.

The synthesized nickel nanostructures were placed in front of the laser radiations at 45°; no emission was recorded, which implies that nickel does not exhibit transition from metallic state to semiconducting state at the level (80 nm) under study. The detailed description of the experimental set-up used for recording optical spectra is given in Fig. 3.

Conclusions

In conclusion, template synthesis is an elegant technique for preparing metallic nanostructures having high aspect ratios. The resulting nickel wires had an average diameter of 80 nm. SEM micrograph shows that the wires were tilted by some angle. This is because of hydrodynamic forces during dissolving process of membrane. XRD diffractogram shows a crystalline nature of the deposited nickel nanostructures. Photoluminescence studies reveal that nickel microstructures do not exhibit band gap formation up to 80 nm scale.

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