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Fabrication and Characterization of Copper Nanowires

Hardev Singh Virk

*Director Research, Nanotechnology Laboratory,
DAV Institute of Engineering and Technology,
India*

1. Introduction

Nanowires are especially attractive for nanoscience studies as well as for nanotechnology applications. Nanowires, compared to other low dimensional systems, have two quantum confined directions, while still leaving one unconfined direction for electrical conduction. This allows nanowires to be used in applications where electrical conduction, rather than tunneling transport, is required. Because of their unique density of electronic states, nanowires in the limit of small diameters are expected to exhibit significantly different optical, electrical and magnetic properties from their bulk 3D crystalline counterparts. The increased surface area, very high density of electronic states, enhanced exciton binding energy, diameter-dependent band gap, and increased surface scattering for electrons and phonons are just some of the ways in which nanowires differ from their corresponding bulk materials. Synthesis, characterization and application of nanowires and nanotubes comprise a significant aspect of today's endeavor in nanotechnology. During recent years, nanowires and nanorods of metallic and semi-conducting materials have drawn a lot of research interest because of their potential applications in diverse fields, for example, nanoelectronics, opto-electronics and sensors (Sarkar et al. 2007; Ratner & Ratner, 2003; Nalwa & Bandhopadhyaya, 2003; Dresselhaus et al. 2003).

Many studies have focused on the fabrication of copper nanowires (Cao & Liu, 2008; Sun Shin et al. 2009; Ingunta et al. 2008; Fang et al. 2007; Motoyama et al. 2005), because of their potential applications in the micro/nanoelectronics industry and, in particular, for interconnection in electronic circuits. Copper is one of the most important metals in modern electronic technology. Many methods have been developed for the fabrication of copper nanowires but template synthesis is considered to be the most suitable and useful for growth of nanowires.

Template synthesis by electrochemical deposition route is easy, low-cost as well as less cumbersome compared to other fabrication techniques (Sarkar et al. 2007), namely, pulsed laser deposition (PLD), vapour-liquid-solid (VLS) method and chemical vapour deposition (CVD). Another advantage of the electrochemical deposition technique is the possibility of fabricating multi-layered structures within nanowires. By varying the cathodic potentials in the electrolyte which contains two different kinds of ions, different metal layers can be controllably deposited. Electrochemical cell used in electrodeposition of copper into pores of

anodic alumina template was fabricated in our laboratory. Morphology of electrodeposited copper nanowires has been studied using Field Emission Scanning Electron Microscopy (FESEM) and crystal structure by XRD analysis. The diameter of nanowires generally depends upon the pore size of template. Anodic alumina discs of 200 nm and polymer membranes of 100 nm pore diameter were selected for this purpose.

2. Template synthesis of nanowires

Template-based growth is a versatile method of synthesis of metallic and semiconductor nanowires. In template-assisted synthesis of nanostructures, the chemical stability and mechanical properties of the template, as well as the diameter, uniformity and density of the pores are important characteristics to consider. Templates frequently used for nanowire synthesis include anodic alumina (Al_2O_3), nano-channel glass, ion track-etched polymers and mica films. Porous anodic alumina templates are produced by anodizing pure Al films in various acids, for example, oxalic acid is most commonly used. Under carefully chosen anodization conditions, the resulting oxide film possesses a regular hexagonal array of parallel and nearly cylindrical channels, as shown in Fig. 2(a). The self-organization of the pore structure in an anodic alumina template involves two coupled processes: pore formation with uniform diameters and pore ordering. Depending on the anodization conditions, the pore diameter can be systematically varied from $< 10\text{nm}$ up to 200nm with a pore density in the range of 10^9 to 10^{11} pores/ cm^2 (Dingle et al., 1969).

Template materials must meet certain requirements (Cao & Liu, 2008). First, the template materials must be compatible with the processing conditions. For example, an electrical insulator is required for a template to be used in electrochemical deposition. Template materials should be chemically and thermally inert during the synthesis. Secondly, depositing materials or solution must wet the internal pore walls. Thirdly, for synthesis of nanowires, the deposition should start from the bottom of the template and proceed upwards to the other side. This is known as bottom up technique in nanotechnology.

Template-based synthesis offers many advantages over other methods of synthesis (Lai & Riley, 2008): (1) It is performed under mild conditions rather than requiring high temperatures, high vacuum or expensive instrumentation; (2) templated electrodeposition has a relatively high growth rate; (3) the morphology of deposited materials depends on the shape of template pores; (4) the dimensions of the materials obtained can be tuned by tuning of the template pore size; (5) two or more components can be easily deposited into the membrane sequentially to form multi-segmented materials or hetero-junctions.

3. Materials and methods

The electrodeposition technique used in our experiment (Virk et al., 2010) is similar in principle to that used for the electroplating process. Commercial anodic alumina membranes (AAM) (anodisc 25 made by Whatman) having an average pore diameter of 200 nm, a nominal thickness of $60\text{ }\mu\text{m}$ and a pore density of 10^9 pores/ cm^2 , were used as templates. A second set of polymer membrane (Sterlitech USA) of 100 nm pore diameter was selected for the sake of comparison. To achieve uniform deposition of nanowires, templates were cleaned in the ultrasonic bath for 10 minutes. The electrochemical cell, fabricated in our laboratory using Perspex sheets, was washed in double distilled water. A copper rod of 0.8 cm diameter

was used as a sacrificial electrode (anode). The cathode consists of copper foil attached to alumina disc by an adhesive tape of good conductivity. Prior to the electro-deposition process, a thin film of copper (0.5 μm) was sputtered onto one side of alumina disc. This metal layer along with adhesive copper tape provides a stable substrate (cathode) for the growth of nanowires. Figure 1(a) illustrates the scheme of this process.

Polymer membranes can be prepared by irradiation of polycarbonate foils using heavy ion beams (Toimil Molaes, 2001). Author has prepared polymer templates, called Ion Track Filters (Virk & Kaur, 1998), using Makrofol N and Kapton after irradiation at the UNILAC (Universal Linear Accelerator) of GSI, Darmstadt, with highly charged heavy ions having kinetic energies in the GeV range and fluences between 10^6 and 10^{10} ions/ cm^2 . Due to energy loss through interaction with the target electrons, each ion creates along its trajectory a cylindrical damage zone, a few nanometers in diameter. The damaged material can selectively be removed by chemical etching, resulting in pores of cylindrical geometry. Composition, concentration, and temperature of the etching solution determine the size and geometry of the resulting pores, the pore diameter increasing linearly with the etching time. A 6 N NaOH solution containing 10% methanol at $T = 50^\circ\text{C}$ was used for etching to produce pore diameters between 50 and 200 nm by varying time of etching. A thin gold film was sputtered onto one side of the membrane using Jeol sputter and reinforced by copper foil attached by an adhesive tape of good conductivity to obtain a stable substrate. This serves as a cathode suitable for the growth of the nanowires in polymer template in our two-electrode electrochemical cell. A schematic diagram of the polymer template synthesis process is illustrated in Figure 1(b).

The electrolyte used had a composition of 20 gm/100ml $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ + 25% of dilute H_2SO_4 at room temperature. A high concentration of CuSO_4 was used to supply a sufficiently large number of ions inside the pores during the deposition. Sulfuric acid was added to increase the conductivity of the solution and to lower the cathode over-voltage. The electrodeposition was performed at room temperature of 30°C . The low overvoltages avoided side reactions such as hydrogen evolution. The inter-electrode distance was kept 0.5 cm and a current of 2mA was applied for 10 minutes using a regulated power supply. Electrodeposition of copper nanowires depends on many factors, namely, inter-electrode spacing, electrolyte composition, temperature and pH value, current density and time of deposition. The influence of current density, temperature and type of electrolyte on the crystallinity of copper nanowires has been reported elsewhere (Toimil Molaes et al., 2001). We studied the effect of current density on electrodeposition of copper nanowires in our experiment.

After the electrodeposition was over, copper foil with template-grown nanowires was divided into two parts. One part was kept for study of I-V characteristics in-situ using Dual Source Meter (Keithley Model 4200 SCS) with platinum probes for contacts. The other part was kept immersed in 1 M NaOH for 1 hour in a beaker to dissolve alumina template. The copper nanowires were liberated from the host matrix, washed in distilled water and dried in an oven at 50°C for 30 minutes. The cleaned and dried nanowires were mounted on aluminium stubs with the help of double adhesive tape. Field Emission Scanning Electron Microscope (FESEM, Hitachi S-4300) was used to record cross-sectional and lateral views of grown nanowires at an accelerating voltage of 15kV using different magnifications. X-ray Diffraction studies were carried out at Sophisticated Analytical Instruments Facility (SAIF) set up by Punjab University, Chandigarh using X' Pert PRO (PANalytical, Netherlands) using $\text{Cu K}\alpha$ radiation.

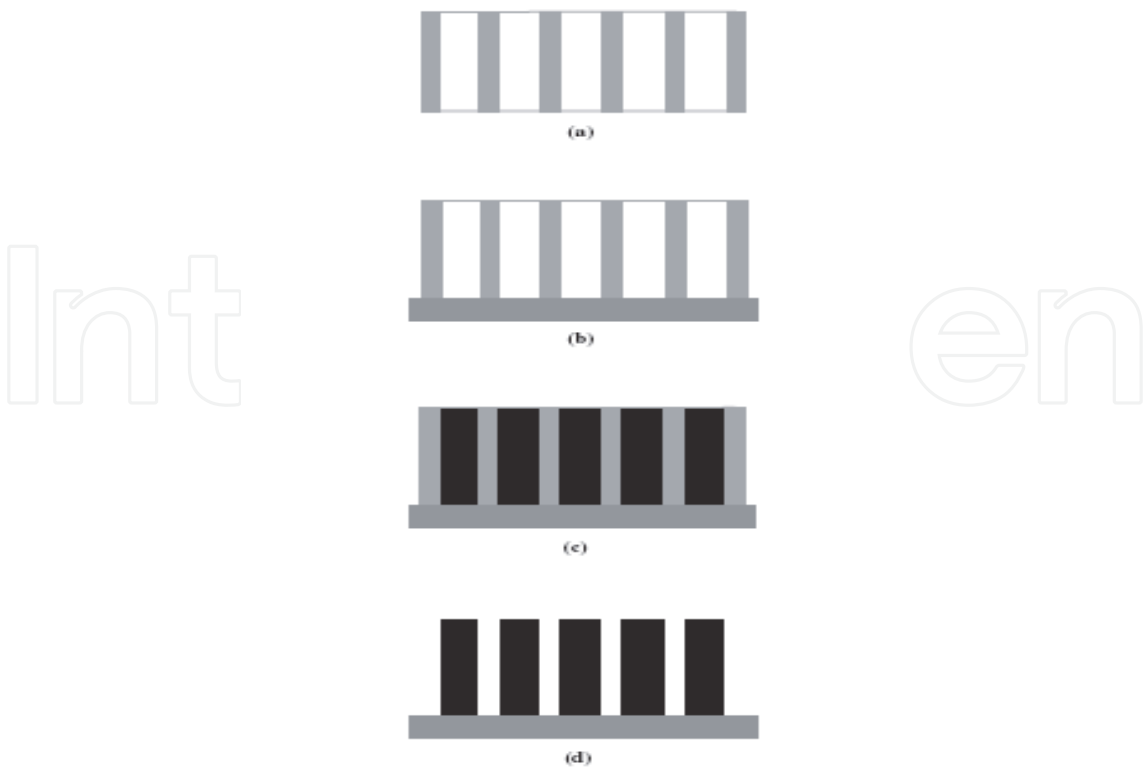


Fig. 1. (a). A schematic diagram of the template synthesis process (Gao et al., 2002): (a) Anodic alumina template, (b) copper sputtered alumina template, (c) electrodeposited copper nanowires, and (d) copper nanowires after removal of anodic alumina template.

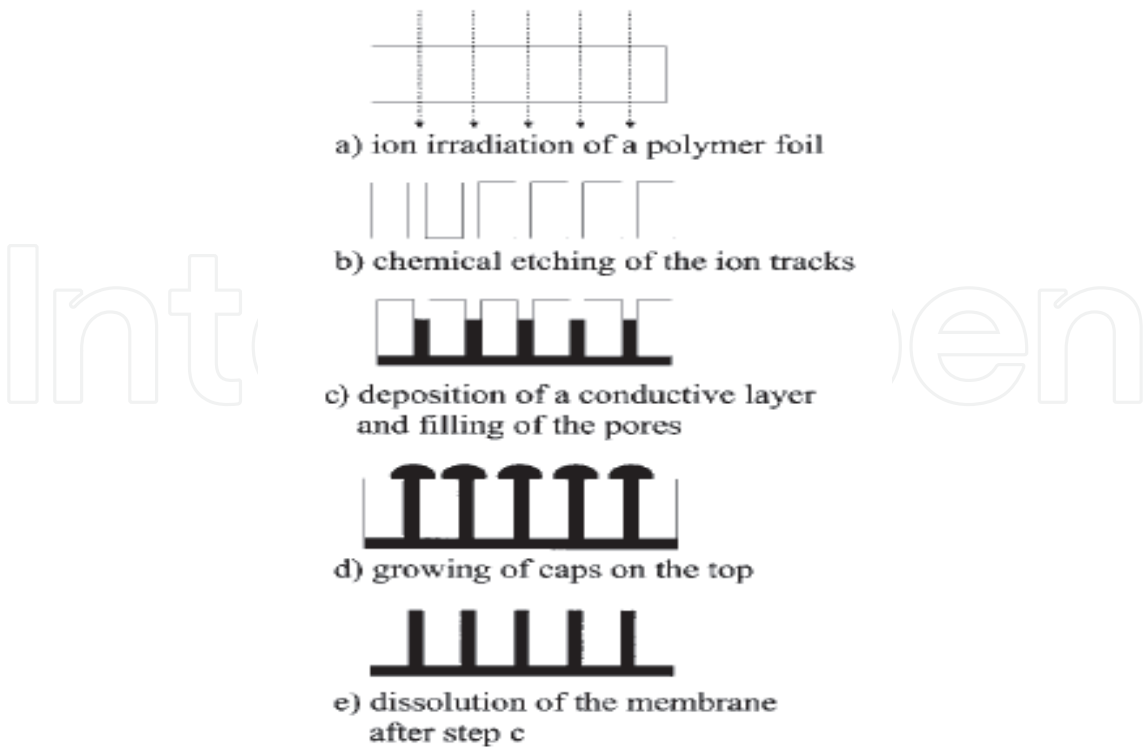


Fig. 1. (b). Scheme of the polymer template synthesis (Toimil Molares et al., 2001).

4. Characterization of copper nanowires

4.1 AFM, SEM and FESEM analysis

Commercial available templates were examined before their use using Atomic Force Microscope (NT-MDT PR 400 Model) installed in our laboratory and Scanning Electron Microscope (Jeol, JSM 6100) facility of Punjab University, Chandigarh. Atomic force microscopic technique (Menon, 2003) shows the two dimensional surface topology of the anodic alumina template with pores regularly arranged on the surface (Fig. 2a). The pores appear nearly at the centre of each hexagonal cell. After gold sputtering, using Jeol sputter JFC 1100, SEM micrograph (Fig. 2b) shows the geometrical pattern of pores on the alumina surface of anodisc.

Copper nanowires liberated from AAM were examined under SEM and FESEM under different magnifications. Two sets of templates were used for growth of copper nanowires. In one set, current density was changed intermittently which resulted in non-uniform growth of nanowires. Figure 3 represents the cross-sectional view of copper nanowires of 200nm diameter grown in alumina template. Figure 4(a) shows the SEM image of copper nanowires array in lateral view, grown under constant current conditions. Figure 4(b) represents the FESEM image of copper nanowires fabricated under transient current conditions. Overdeposition of copper is clearly visible towards the tip of nanowires resulting in capping effect. Nanowires are quite uniform with diameter in the range of 200 nm but they are not perfect cylinders. It has been reported (Schonenberger et al., 1997) that pore diameters of commercially available templates vary over a large range. The aspect ratio, that is, the ratio of length to diameter, is on the order of 300.

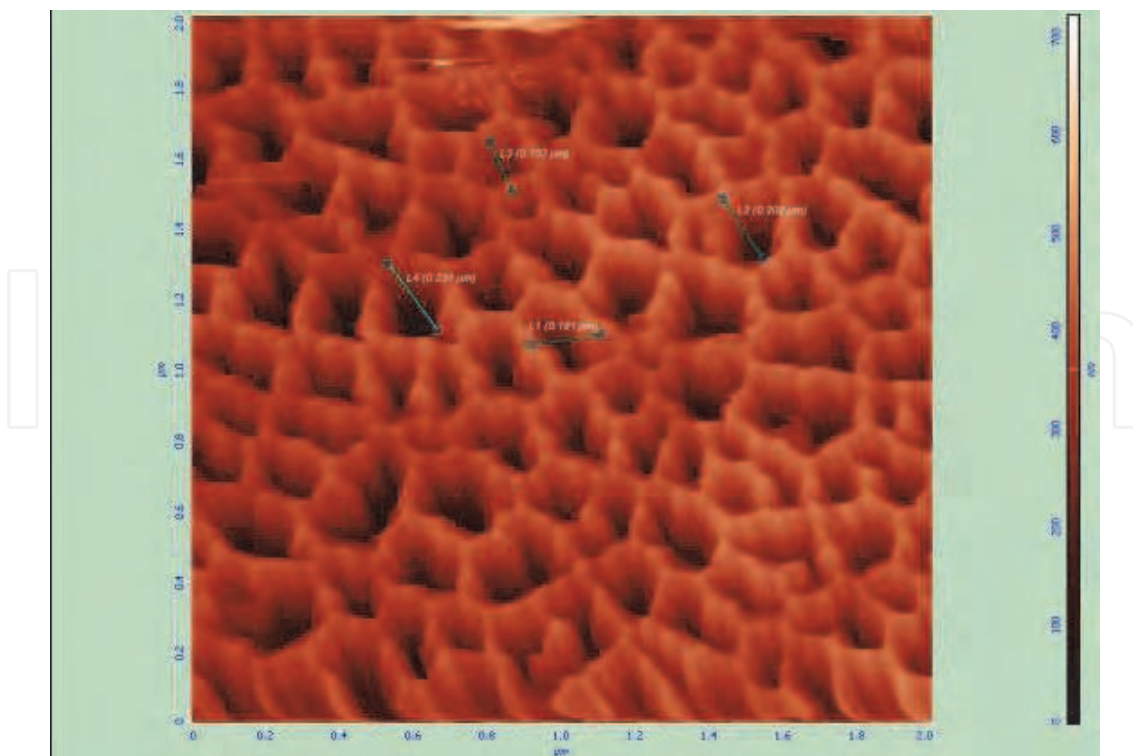


Fig. 2. (a) AFM image of hexagonal pores of anodic alumina template

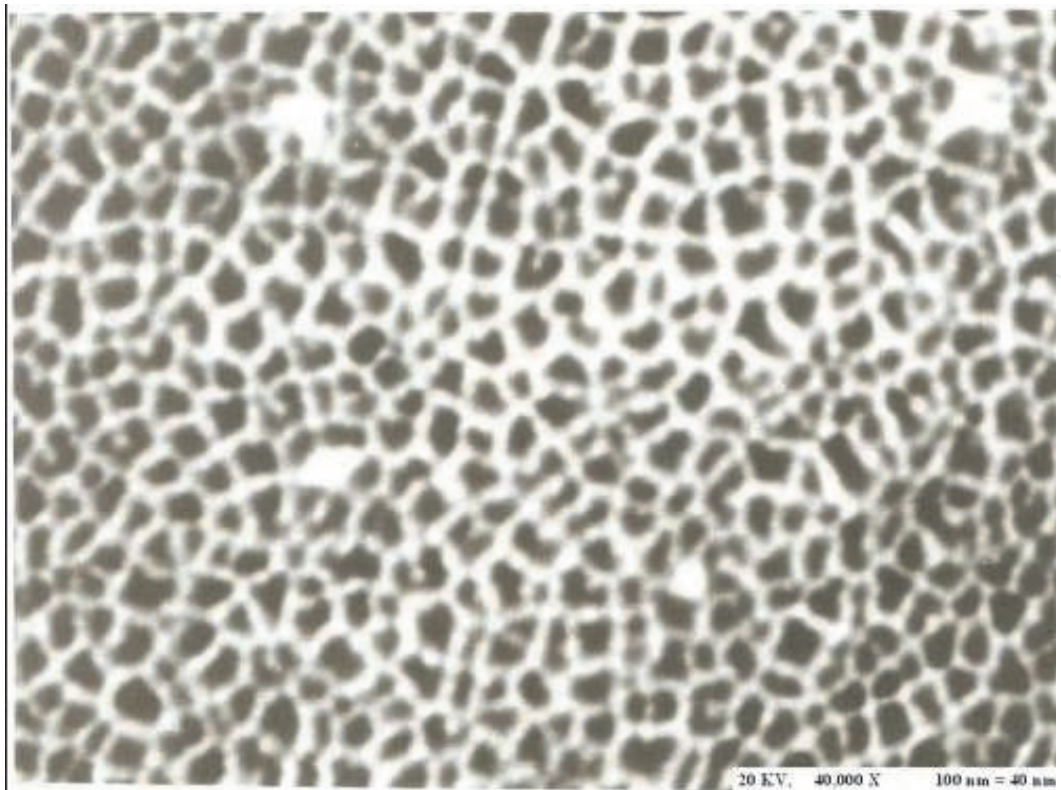


Fig. 2. (b) SEM image of anodic alumina template pores

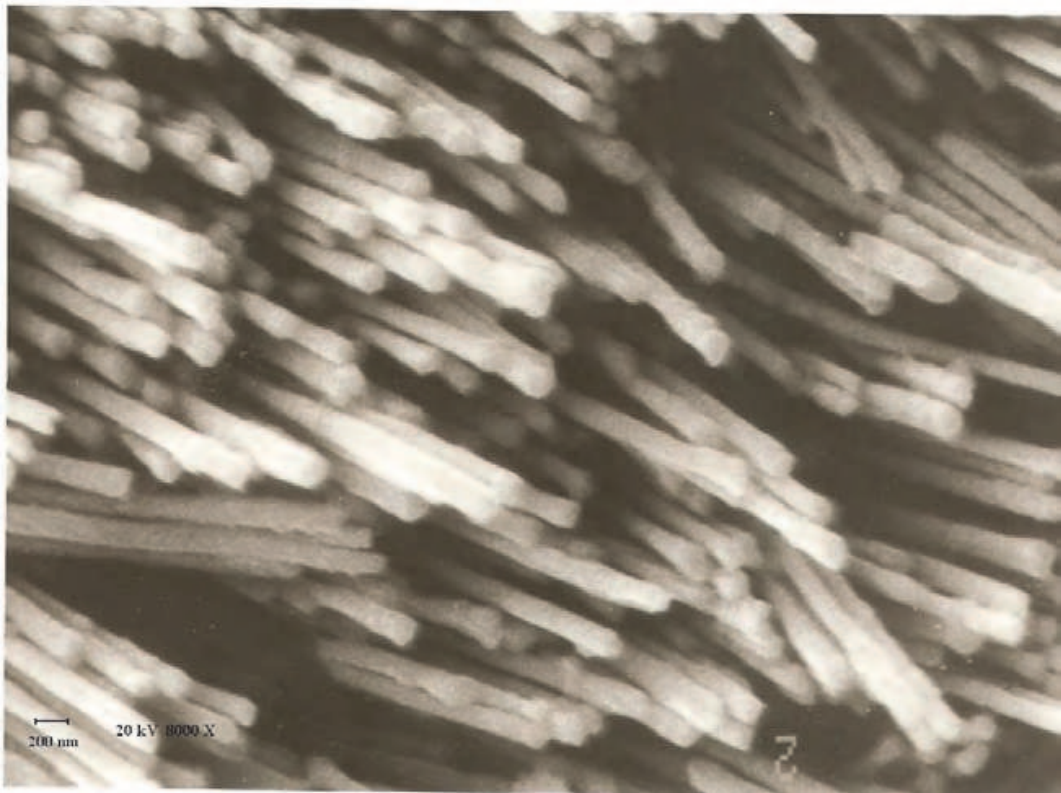


Fig. 3. SEM image of copper nanowires (cross-sectional view, 200 nm dia.)

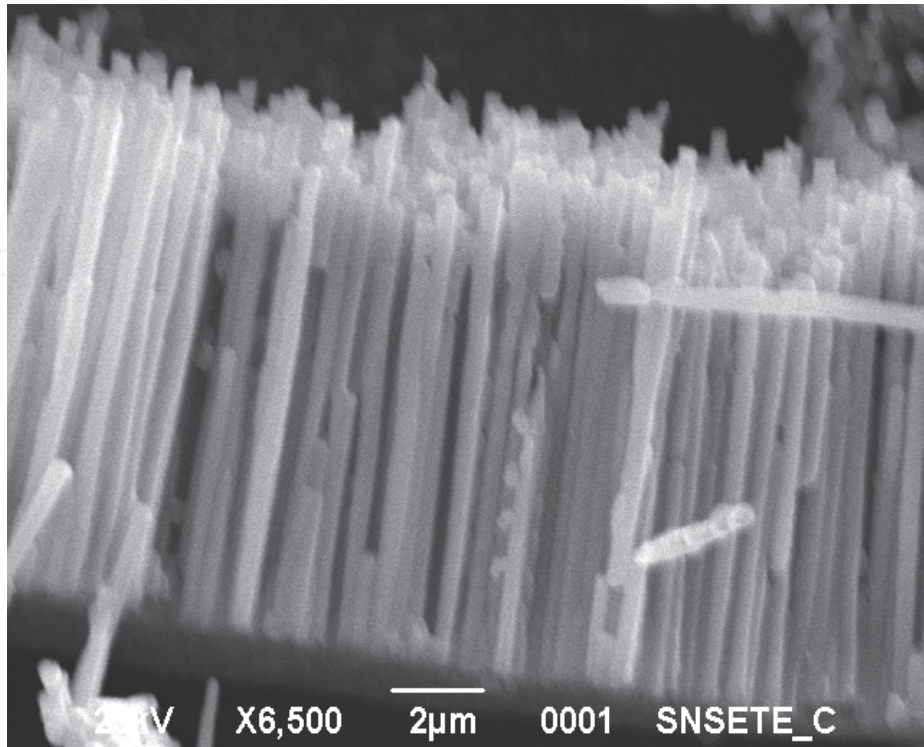


Fig. 4. (a) SEM image of copper nanowires fabricated under constant current

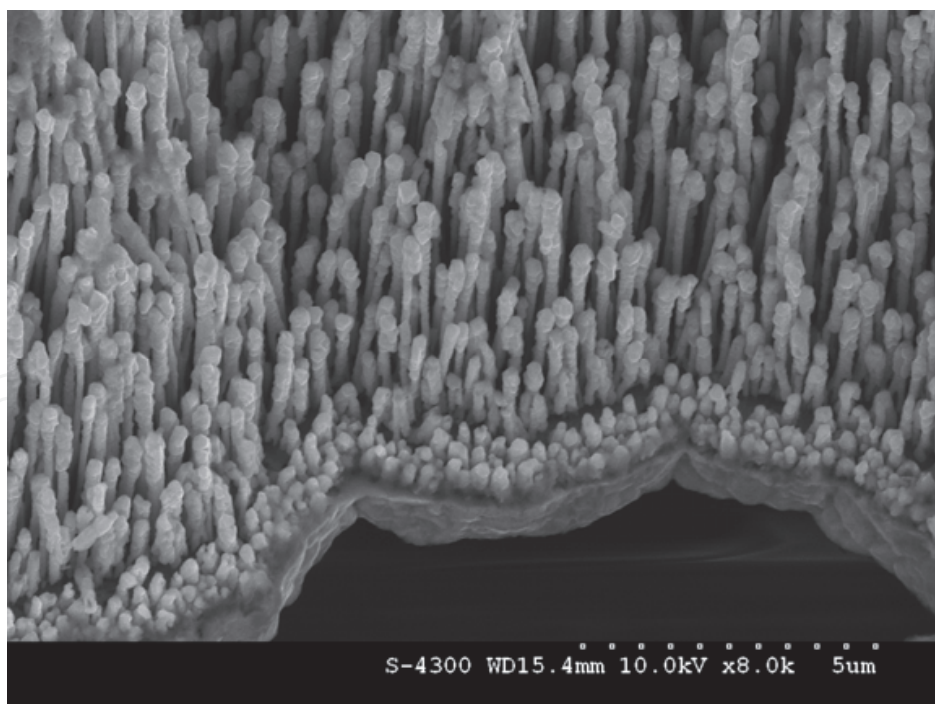


Fig. 4. (b) SEM image of copper nanowires showing capping effect

Experiment was repeated using polycarbonate membrane with pore diameter of 100 nm as a template and keeping the other conditions identical. The polymer template was dissolved in dichloromethane to liberate copper nanowires from the host matrix. The rest of the

procedure is same. Instead of nanowires, we observed under FESEM the exotic patterns in the form of microflowers (Fig. 5) having their petals in nanometer dimension and copper buds (Fig. 6) leading to mushroom effect. Similar results with exotic patterns were reported in our earlier experiment (Virk et al. 2010).

There is as yet no specific theory to explain exotic patterns developed during electrodeposition of copper in anodic alumina or polymer templates. A speculative explanation (Gao et al., 2002) is provided on the basis of overdeposition. During the growth of copper nanowires in the template pores, the current remains nearly stable until the wires arrive at the template surface. If the electro-deposition process is not stopped at this stage, the current keeps on rising very gradually leading to overdeposition of copper. Flower like morphologies of metal overdeposits have been attributed to the changes in hydrodynamic conditions due to excessive hydrogen evolution during electrodeposition process (Kumar et al., 2008).



Fig. 5. FESEM micrographs showing flower patterns grown in polymer templates

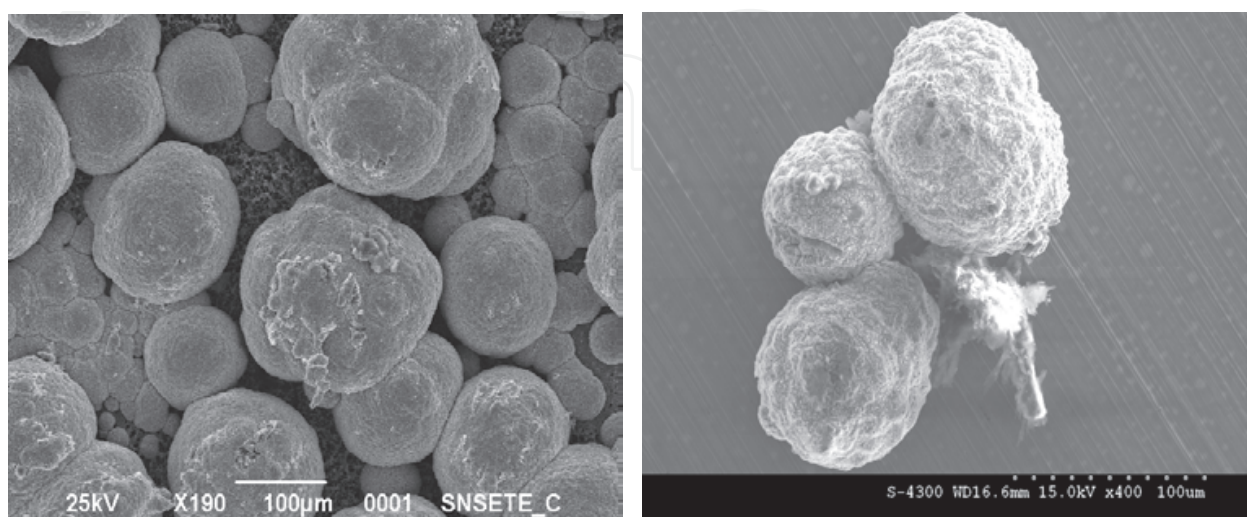


Fig. 6. FESEM micrographs showing copper buds grown in polymer templates

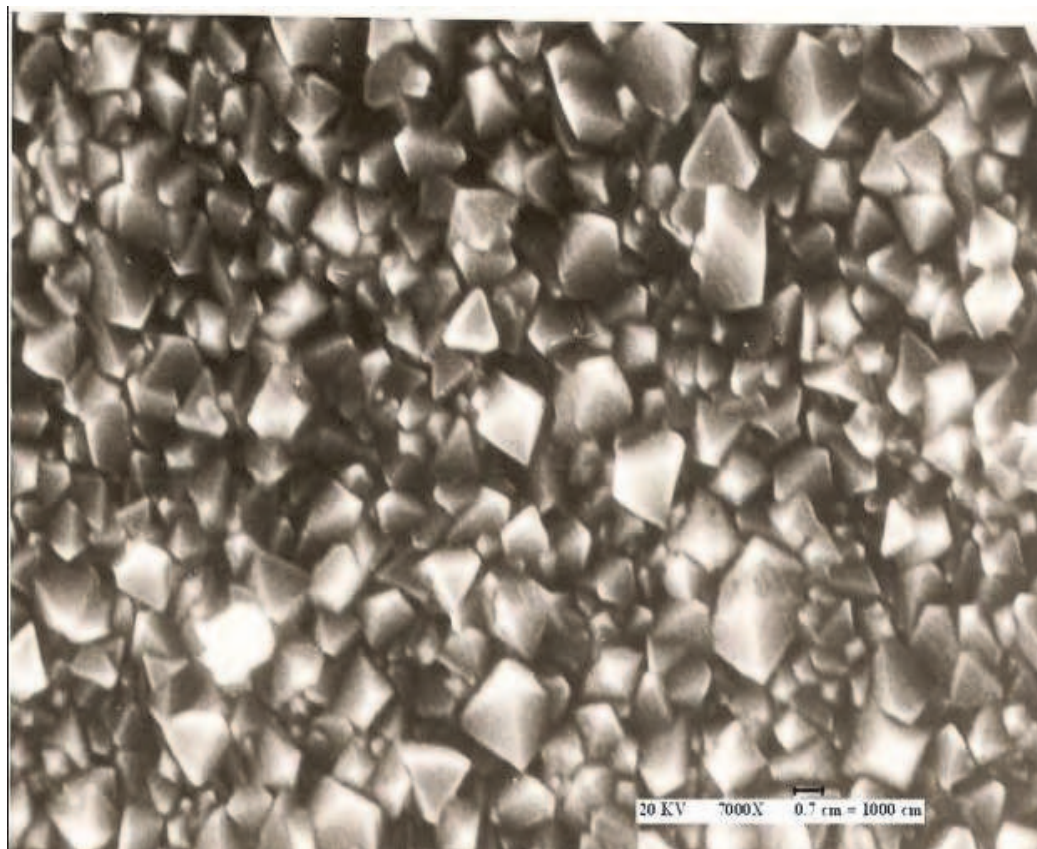


Fig. 7. SEM micrograph of pyramid shaped polycrystalline copper crystals

We repeated the experiment for 20 nm pore diameter polycarbonate template. The template was not coated with a conducting layer during electrodeposition. It resulted in failure to grow nanowires but the failure of experiment proved to be a blessing in disguise. Instead of copper nanowires, we observed growth of double pyramid shaped copper crystals (Fig. 7). We could not find evidence for this phenomenon in literature. It is anticipated that copper ions from the electrolyte do not enter template pores due to poor conductivity but get deposited on the cathode surface in the form of polycrystalline crystals.

4.2 X-ray and EDAX analysis

The characterization techniques that are commonly used to study the crystal structure and chemical composition of nanowires include X-ray diffraction and X-ray energy dispersion analysis (EDAX). Both these techniques have been employed in our analysis. The crystal structure of the double pyramid shaped copper crystals has been determined using X-ray diffraction analysis. XRD spectrum (Fig. 8) shows two prominent peaks corresponding to $2\theta = 43.4610$ and 50.5803 , with d spacing = 2.082 and 1.804, respectively. These peaks reveal the polycrystalline nature of copper crystals, indicating that preferred growth direction of crystals is the (200) plane. Template based synthesis of single crystal copper nanowires has been reported in literature (Toimil Molares, 2001; Gao et al., 2002; Mingliang et al., 2003) with preferred growth direction along (111) plane, but to the best of our knowledge, there is hardly any report for copper nanowire arrays or copper crystals with a (200) preferred orientation.

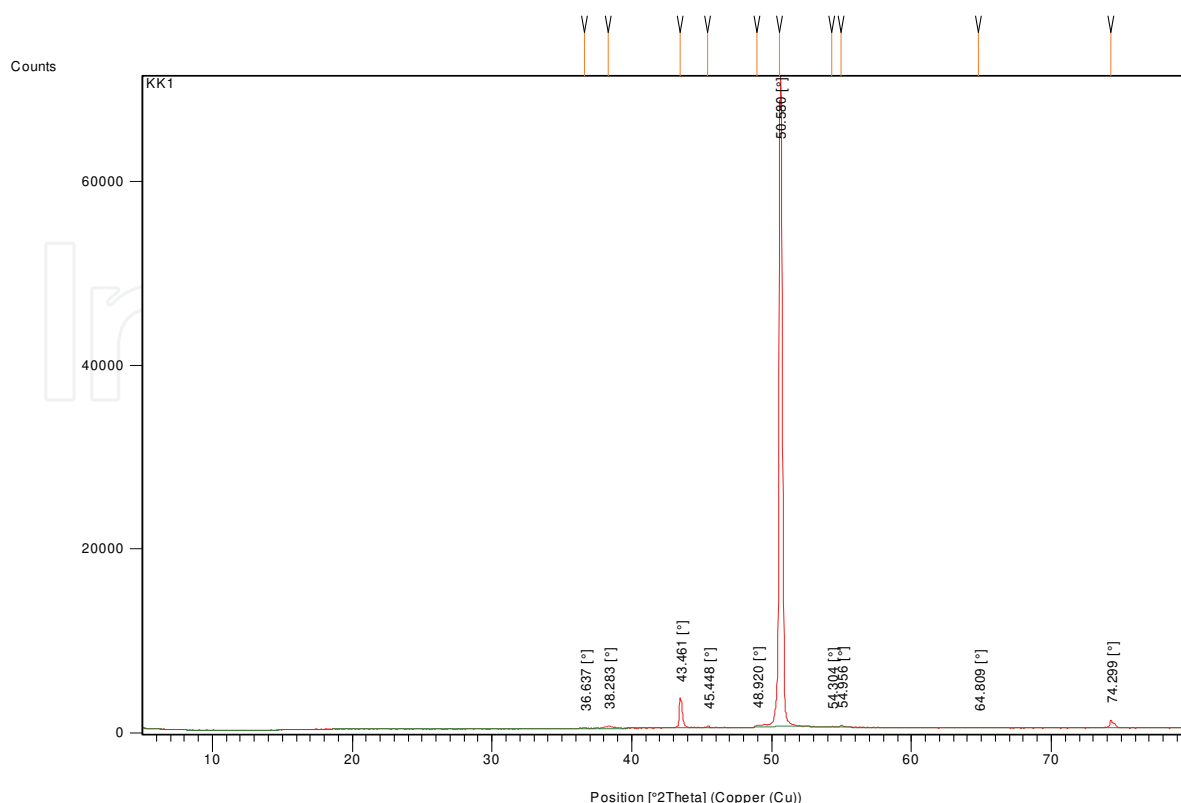


Fig. 8. XRD spectrum of pyramid shaped polycrystalline copper crystals

The crystallographic structure of copper nanowire arrays was investigated by X-ray diffraction analysis (XRD). For sake of comparison, XRD spectrum of Cu foil used as a substrate was also recorded (Fig. 9). XRD diffractograms were obtained in the 2θ range from 10° to 80° with a step of 0.02° , using the Cu K α radiation source of $\lambda = 1.5406 \text{ \AA}$. XRD spectrum (Table 1) shows three prominent peaks corresponding to $2\theta = 43.5966$, 50.8127 and 74.4331 , with d spacing = 2.074, 1.80 and 1.27, and corresponding Miller indices, (111), (200) and (220), respectively. All peaks can be attributed to the cubic form of metallic copper (Ingunta et al., 2008). XRD spectrum of copper nanowires (Fig. 10) shows some interesting results. There are in all 8 peaks in the spectrum; with 2 additional peaks at $2\theta = 37.0062$ and 54.9761 , which are of negligible intensity and may be ignored. Three main peaks are also there as in Fig. 9 but two of them split into double and triple peaks (Table 2), which may be attributed to X-ray scattering at the substrate. These peaks reveal the polycrystalline nature of copper nanowires, the most prominent peak at $2\theta = 50.9870$, indicating that the preferred growth direction of nanowires is the (200) plane. Due to polycrystalline nature of copper nanowires, the most prominent peak at $2\theta = 43.5966$ (Fig. 9) shifts to $2\theta = 50.9870$ (Fig. 10). Template based synthesis of single crystal copper nanowires have also been reported in literature (Gao et al., 2002; Mingliang et al., 2003) with preferred growth direction along (111) plane.

The average size D of the crystalline grains in the Cu nanowires is calculated using the Debye Scherrer's formula (Cullity, 1956): $D = 0.9 \lambda / \beta \cos \theta$, where $\lambda = 1.5406 \text{ \AA}$ is the wavelength of the X-ray radiation used, β is the full width at half maximum (FWHM) of the diffraction peak (0.1224), K , shape factor is assumed to be 0.9 and θ is the Bragg diffraction angle of the most prominent XRD peak. Substituting appropriate values in the formula, the crystallite size value of Cu nanowires comes out to be 1.22 nm. However, the value of crystallite size calculated for Cu foil is exact double, of the order of 2.44 nm.

Pos. [°2Th.]	WHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Area [cts*°2Th.]
43.5966	0.0612	2.07438	100.00	847.65
50.8127	0.0816	1.79542	48.53	548.43
74.4331	0.1428	1.27358	11.94	236.07

Table 1. XRD spectrum peaks data of copper film

Pos. [°2Th.]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Area [cts*°2Th.]
37.0062	0.3346	2.42724	2.29	76.37
43.4706	0.0669	2.08010	33.59	223.69
43.8561	0.2509	2.06270	93.99	2347.28
50.5881	0.0816	1.80286	74.62	819.15
50.9870	0.1224	1.78969	100.00	1646.70
51.1172	0.1224	1.78544	87.56	1441.89
54.9761	0.4080	1.66889	0.75	41.24
74.4238	0.4080	1.27372	5.90	324.08

Table 2. XRD spectrum peaks data of copper nanowires

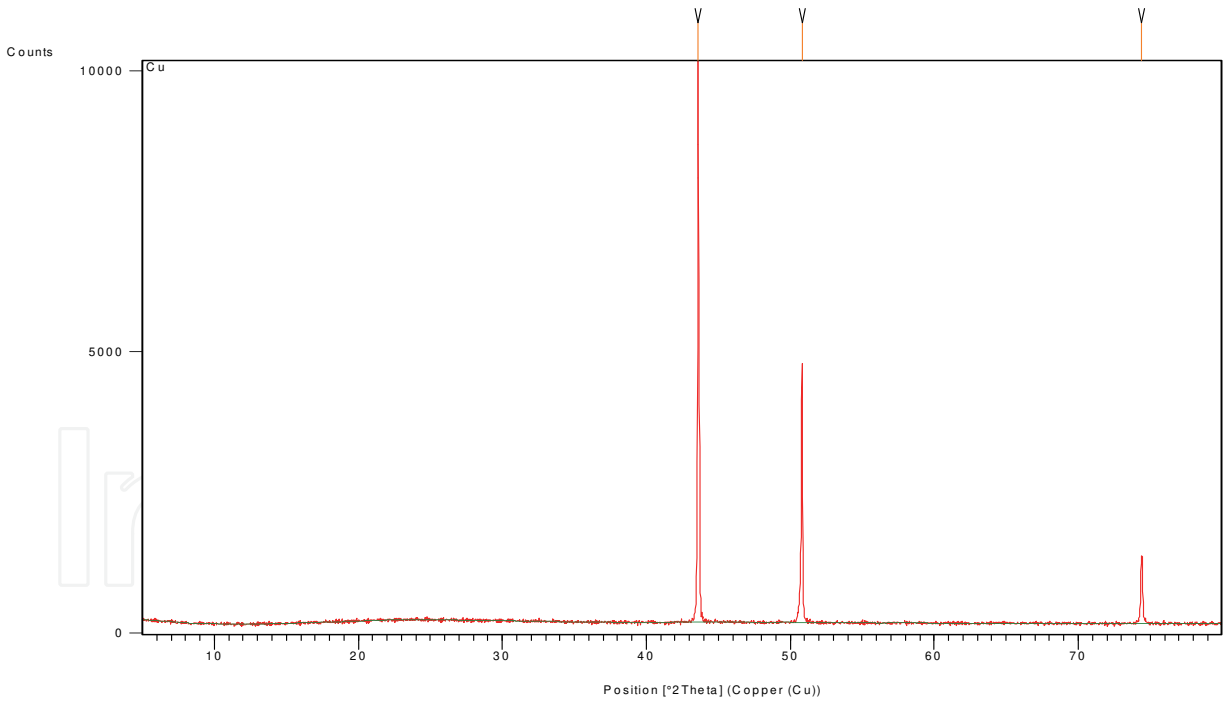


Fig. 9. XRD spectrum of Copper film serving as a substrate

Energy dispersive X-ray analysis (EDAX) of Cu nanowires was carried out at FESEM facility of CSIO, Chandigarh to determine chemical composition of nanowires. The spectrum (Fig. 11) reveals 3 peaks of copper with 100% pure copper content and no traces of any impurity in Cu nanowires. It also establishes that multiple XRD peaks are not due to any impurity but due to polycrystalline nature of Cu nanowires.

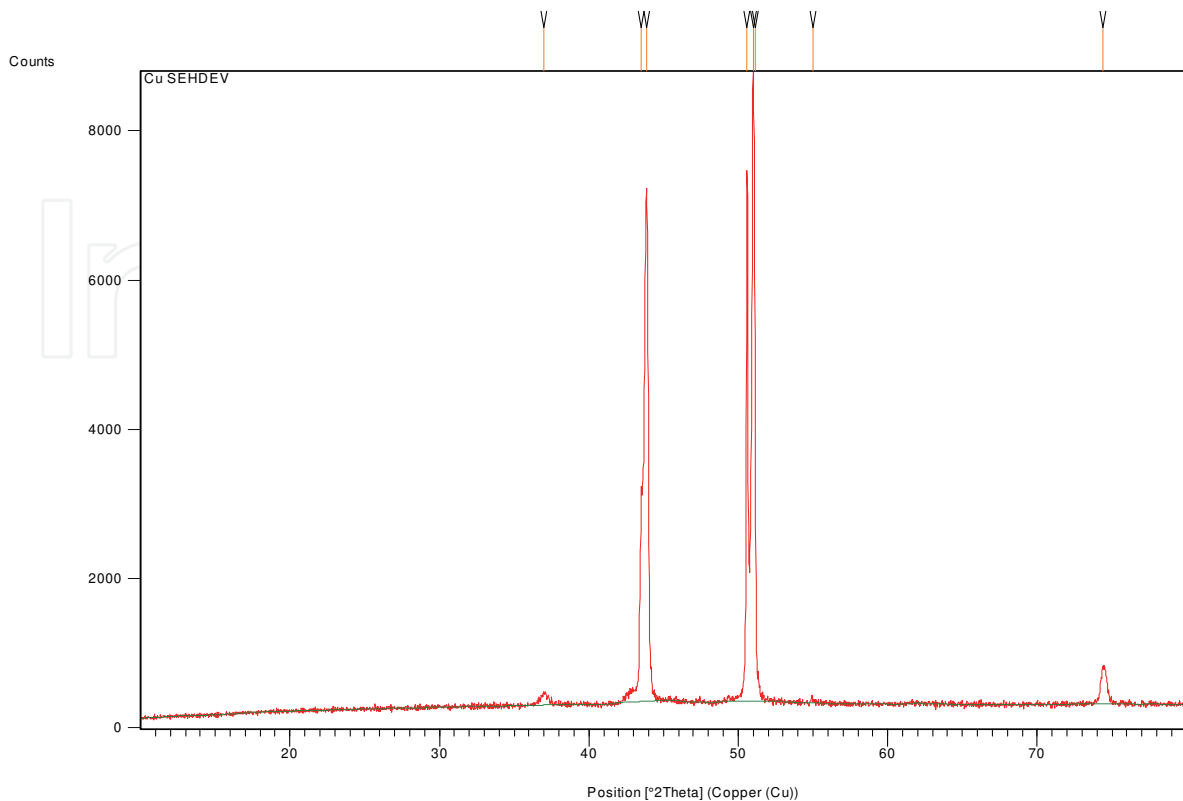


Fig. 10. XRD spectrum of Copper nanowires of 200 nm diameter

4.3 I-V Characteristics of copper nanowires

I-V properties of aligned copper nanowires have been studied using a current- sensing AFM (Cao et al., 2006). Electronic transport through nanocontacts has been an active research area. The ultimate aim for nanowires is to find applications in the nanoelectronic devices. How can a copper nanowire produce a nonlinear I-V curve? The simplest possibility for observing such a phenomenon is generation of a tunnelling barrier at the wire-lead junction whose effect gradually collapses as a function of increasing bias voltage (Mehrez & Guo, 2004). The nonlinear curves of Cu nanowire arrays may be caused by the existence of impurities (such as oxide) near the wire-lead contact region. Nonlinear phenomena of silver wire and gold wire have also been observed in air (Mehrez & Guo, 2004; Wildoer et al., 1998). It has been demonstrated that the nonlinear I-V characteristic is the basis of functional electronic devices (Itakura et al., 1999).

I-V characteristics of copper nanowires were recorded in-situ, as grown in pores of anodic alumina template, using Dual Source Meter (Keithley Model 4200 SCS) with platinum probes for contacts. The combination of copper nanowires on alumina, an insulator, results in the formation of a strange device. I-V plot (Fig. 12) shows some interesting features of a resonating tunneling diode in the forward bias mode but nothing special in the reverse bias mode. The offset in I-V plot around zero voltage may be due to slight non-ohmic characteristic of the contact, or due to quantum confinement behaviour of electrons traversing through copper nanowires.

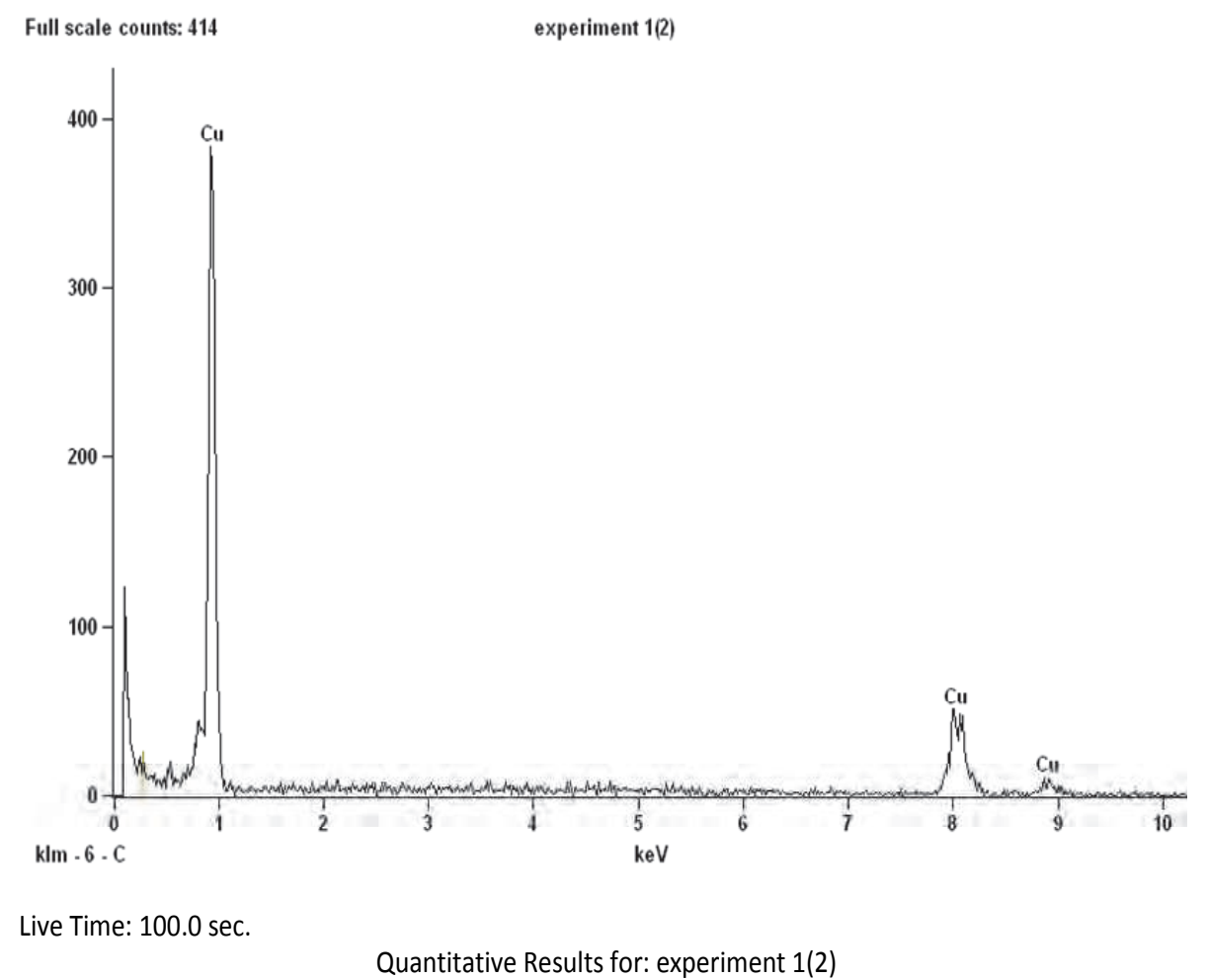


Fig. 11. EDAX spectrum and elemental composition of Copper nanowires

5. Conclusions

Our investigations confirm that electrodeposition of copper nanowires in anodic alumina is the simplest route to nanotechnology. The copper nanowires reveal effect of high current density resulting in overdeposition in the form of capped growth, and not as perfect cylinders. The aspect ratio is very high, of the order of 300. XRD analysis shows polycrystalline nature of nanowires and pyramid shaped copper crystals with preferred growth direction in the (200) plane. The crystallite size of nanocrystals in copper nanowires is determined to be 1.22 nm. Overdeposition results in growth of copper buds and beautiful flower patterns. I-V characteristics do not conform to normal p-n junction behaviour and need further investigation. The nonlinear I-V characteristic of the as-synthesized copper nanowire arrays suggests the presence of a potential barrier. Due to high aspect ratio, copper nanowires may be used as field emitters.

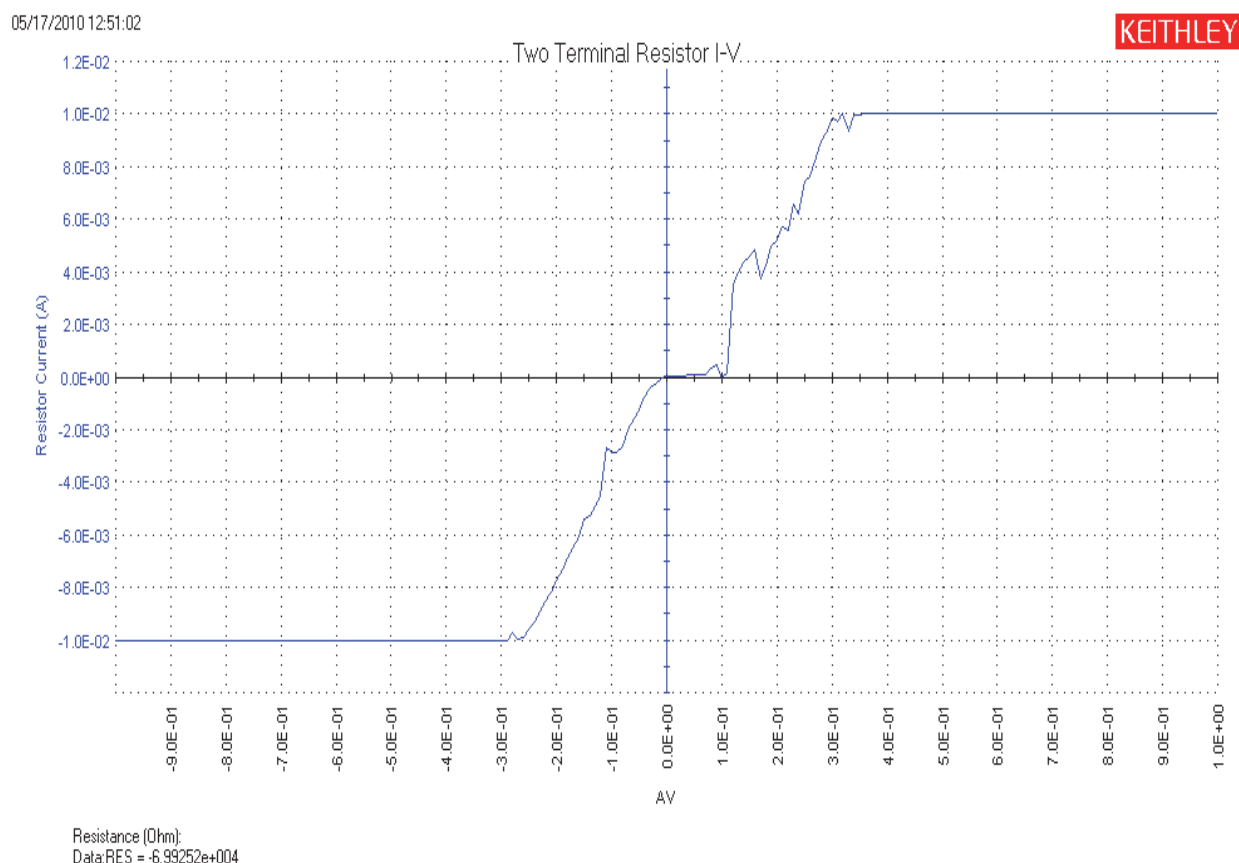


Fig.12. I-V characteristics of copper nanowires grown in-situ in anodic alumina template

Copper oxide nanowire arrays have already found applications in gas sensing, field emission and photovoltaic devices. A recent study (Rathmell et al., 2010) has established that copper nanowires could revolutionize the development and production of low-cost flexible displays, light emitting diodes and thin film solar cells. Copper is 1000 times more abundant than indium or silver, and is 100 times less expensive. As a consequence, films of copper nanowires represent a low-cost alternative to silver nanowires or ITO for use as a transparent electrode.

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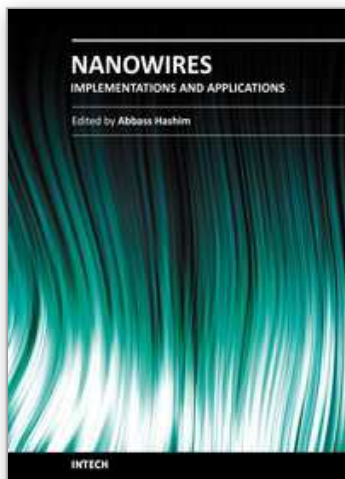
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This potentially unique work offers various approaches on the implementation of nanowires. As it is widely known, nanotechnology presents the control of matter at the nanoscale and nanodimensions within few nanometers, whereas this exclusive phenomenon enables us to determine novel applications. This book presents an overview of recent and current nanowire application and implementation research worldwide. We examine methods of nanowire synthesis, types of materials used, and applications associated with nanowire research. Wide surveys of global activities in nanowire research are presented, as well.

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University Campus STeP Ri
Slavka Krautzeka 83/A
51000 Rijeka, Croatia
Phone: +385 (51) 770 447
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InTech China

Unit 405, Office Block, Hotel Equatorial Shanghai
No.65, Yan An Road (West), Shanghai, 200040, China
中国上海市延安西路65号上海国际贵都大饭店办公楼405单元
Phone: +86-21-62489820
Fax: +86-21-62489821

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