

# Feature Article

## An Overview of Nanotechnology Research at DAV Institute of Engineering & Technology, Jalandhar

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### Abstract

The DAVIET Research Centre was established in January 2008 to promote research in interdisciplinary area of Nanotechnology. Universal Scanning Probe Microscope (Model NT-MDT Solver PRO M4) was imported from Russia and installed in August 2008. Hind High vac Thin Film coating unit was installed in October 2008. Thus DAVIET became the first engineering institute in North India to establish a well furnished nanotechnology laboratory. Our thrust area of research is fabrication of Quantum Dots and Nano wires for use in nanoelectronics and Opto-electronics. Template synthesis of Quantum Dots and Nanowires has been achieved by using Anodic Alumina membranes and Polymer membranes of pore diameter varying from 20-200 nm. The electrochemical cell fabricated in our laboratory has been used to grow copper nanowires of 200 nm using an electrolyte with a composition of 20gm/100 ml  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 25% of dilute  $\text{H}_2\text{SO}_4$  at room temperature. Using Reverse Micelle technique, nanorods of Barium carbonate and barium oxalate were synthesized along with nanocrystals of Iron oxalate. Cadmium sulphide nanocrystals and needles have been grown using micro-emulsion technique using different co-surfactants. Characterization of grown using micro-emulsion technique using different co-surfactants. Characterization of grown nanostructures has been done by using SEM, TEM and XRD.

**Keywords:** Nanotechnology, Quantum Dots, Nanowires, Anodic Alumina Templates.

### 1. Introduction

In recent years, one of the most active areas of research has been nanotechnology [1]. Besides the general drive towards miniaturization (as in the microelectronics industry), nanosized materials are interesting for the new range of physics that they allow one to study. For example, quan-

tum dots of CdSe show size dependence in several of their properties, such as photoluminescence. They also have the potential for many interesting applications. For example, the high surface area of  $\text{TiO}_2$  nanorods has been demonstrated to yield far better photocatalytic behavior than bulk  $\text{TiO}_2$  films.

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Quantum dots (0D), also known as nanocrystals, are a special class of materials known as semiconductors, which are crystals composed of periodic groups of II-VI, III-V, or IV-VI materials. Semiconductors are a cornerstone of the modern electronics industry and make possible applications such as the Light Emitting Diode (LED) and personal computer. Semiconductors derive their great importance from the fact that their electrical conductivity can be greatly altered via an external stimulus (voltage, photon flux, etc), making semiconductors critical parts of many different kinds of electrical circuits and optical applications. Quantum dots are unique class of semiconductors because they are so small, ranging from 2-10 nanometers (10-50 atoms) in diameter. At these small sizes materials behave differently, giving quantum dots unprecedented tunability and enabling never before seen applications to science and technology.

Nanowires/nanorods (1D) have attracted considerable attention in recent years [2-3] because of their novel physical properties and potential applications as interconnects in nanometre-scale electronics. The progress in this field has been accelerated by advances in both synthetic methods of preparing the nanoporous templates, and development of techniques capable of filling the pores of such membranes. Examples of long aspect nanoporous membranes include nanochannel glass membranes, anodized aluminium substrates, and various polymeric membranes. Filling of the pores of such membranes with long aspect ratio nanowires/nanorods has been accomplished by electro-deposition, high-pressure metal melt injection, and photochemicals methods.

Heavy ion tracks in dielectric films offer unique possibilities for the realization of nanometer-sized structures at low cost and high throughputs. In combination with lithography they open up new ways for biofluidic, electric, magnetic and optic device fabrication. Heavy ions produce along their path a nanometer channel of modified material with track diameter between 1 and 10 nm, adjustable by the chosen ion and its kinetic energy. The latent tracks created in irradiated

materials may be used directly, e.g., creating conducting and magnetic nanowires in insulating matrices or they may be selectively etched into pores and then used for nanobiofluidic applications or as templates for growing micro/nanostructures. Commercial irradiation can produce ion track membranes with pore density ranging from  $10^4$  to  $10^8$  pores per  $\text{cm}^2$  per second.

## 2. Experimental Techniques

Electrochemical cell used for electrodeposition of metals into etched pores was fabricated in our laboratory. The metallic ions in a supporting solution are reduced to the metallic state at the cathode which is covered by an ITM. The etched pores of ITM used would act as a template. The rate of deposition of metallic film depends, upon many factors, viz. current density, interelectrode distance, cell voltage, temperature and concentration of electrolyte used in the cell. Anodic Alumina Membranes (AAM) manufactured by Whatman Company has been used to grow quantum dots and nanowires by electrodeposition technique. The pore size selected varies from 20 nm to 200 nm. The electrochemical cell fabricated in our laboratory was used to grow copper nanowires of 200nm using an electrolyte with a composition of 20gm/100ml  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  + 25% of dilute  $\text{H}_2\text{SO}_4$  at room temperature. The copper nanowires were liberated from their host AAM matrix by dissolving it in 1M NaOH at room temperature ( $22^\circ\text{C}$ ) for 2 hours. The copper nanowires grown on copper foils were dried in an oven at  $50^\circ\text{C}$  for 30 minutes. The cleaned and dried nanowires were mounted on aluminium stubs with the help of double adhesive tape, coated with a layer of gold palladium alloy in Jeol Sputter JFC 1100 and viewed under Scanning Electron Microscope (Jeol, JSM-6100) at an accelerating voltage of 20 kV.

We also followed chemical route to nanotechnology using Reverse Micelle technique of microemulsions [4, 5] to grow nanorods/needles and quantum dots of some metals and hexaferrites of Barium. A microemulsions system consists of an oil phase, a surfactant, and



an aqueous phase. It is a thermodynamically stable isotropic dispersion of the aqueous phase in the continuous oil phase. The size of the aqueous droplets is the range of 5-20 nm rendering the microemulsion system optically transparent. Chemical reactions, such as precipitation will take place when droplets containing the desirable reactants collide with each other. These nanodroplets can be used as nanoreactors to carry out the chemical reactions. The mechanism for the formation of nanocrystalline material using reverse micelles may be understood qualitatively by considering the synthesis of a simple compound like metal oxalate and carbonate. Microemulsion A should be metal ions and B should be oxalate in case of metal oxalate, and in case of metal carbonate, B should be carbonate. These two microemulsion are mixed by constant stirring and the droplets continuously collide, which results in the interchange of reactant. During this process, the reaction takes place inside the nanoreactor. In microemulsion system, the reverse micelles of metal and oxalate/carbonate instead of ammonium carbonate.

ate forms the fused dimmer which finally breaks down into two stable smaller droplets because surface tension becomes high due to large surface area and dimmer is unable to sustain its geometry.

In a typical experiment, the barium oxalate and carbonate were synthesized by using the reverse micellar route with CTAB as a surfactant, n-butanol as the co-surfactant and iso-octane as the non-polar solvent. The composition of various constituents is given in Table 1. For the synthesis of barium carbonate two different microemulsions, one containing 0.1 M aqueous solution of barium nitrate and other containing the 0.1M aqueous solution of ammonium carbonate were slowly mixed and stirred overnight. The product was separated from microemulsions by centrifugation and washed with 1:1 mixture of chloroform and methanol and dried at room temperature. The synthetic procedure of barium oxalate is similar as discussed above for barium carbonate except that the second microemulsion contains 0.1 M aqueous solution of ammonium ox-

**Table 1.** Composition of the microemulsion system used in all nanoparticle preparations

S.No.	Microemulsion System	Quantity
1.	Aqueous Phase (0.1M)	2.50 ml
2.	Iso-Octane	15.0 ml
3.	n-Butanol	3.50 ml
4.	Cetyl trimethylammonium bromide (CTAB)	4.20 gm

## Discussion of Results

Electrodeposition of copper nanowires depends on many factors, namely, inter-electrode spacing, electrolyte composition and pH value, current density and time of deposition. To achieve uniform deposition of nanowires, templates were cleaned in the ultrasonic bath for 10 minutes. After cleaning, the templates were fixed to the adhesive copper tape to make it a perfect cathode. Copper nanowires were examined under SEM under different magnifications. Fig 1(a) shows a typical cross-sectional view of copper nanowires grown in alumina template. Fig. 1 (b) represents

the lateral view of nanowires. Obviously, the diameter of copper nanowires is identical to the diameter of pores (200nm) of anodisc. Nanowires are quite uniform but they are not perfect cylinders. It has been reported [6] 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.

Electrodeposition of copper nanowires was achieved under identical conditions in polycarbonate template (Sterlitch, USA). The polymer template was dissolved in chloromethane at room



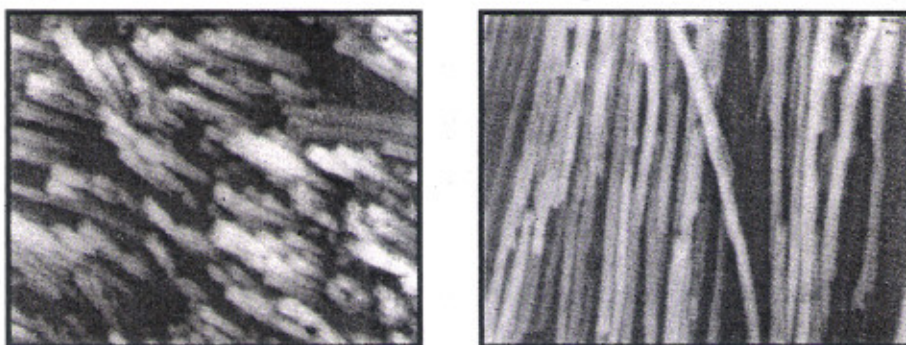


Fig. 1(a,b) SEM images of Copper nanowires (cross-sectional & lateral axial views)

temperature for liberating the copper wires. SEM micrographs (Fig.2) of grown copper nanowires show strange features. The cross-sectional and lateral views are somewhat distorted and not as smooth as in case of porous alumina templates. The diameter of copper nanowires matches with the pore diameter (100 nm) of polycarbonate template.

We also used chemical route of Reverse Micelle technique to fabricate nanorods/needles of various metals. Figure 3 (a) shows the TEM image of  $\text{BaCO}_3$  using reverse micelles (with CTAB). The selected area electron diffraction (SAED) pattern (Figure 3b) shows number of spots arranged in circular manner which indicates the

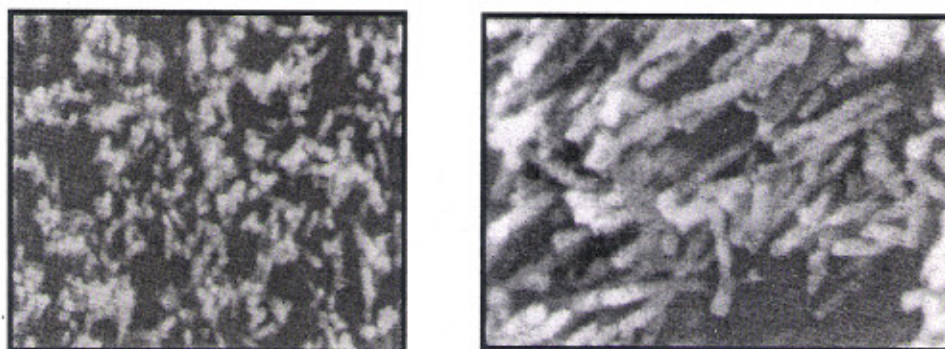


Fig. 2 (a,b) SEM images of Copper nanowires (cross-sectional & lateral axial/surface views)

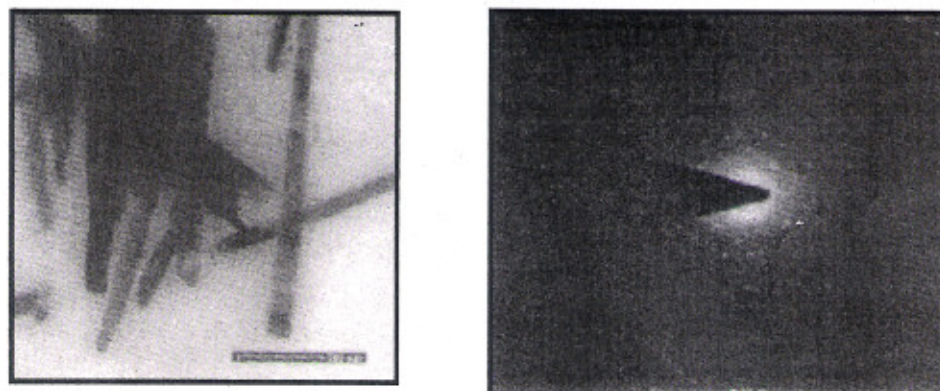


Fig. 3 (a) Transmission electron (b) selected area electron diffraction micrographs of Barium carbonate ( $\text{BaCO}_3$ )



nanocrystalline nature of nanorods formed. Figure 4 shows SEM image of rod-like  $\text{BaCO}_3$  particles which is in good agreement with the image obtained in case of TEM. Therefore rod-like morphology of both SEM and TEM images strongly supports the  $\text{BaCO}_3$  particles as rod-shaped.

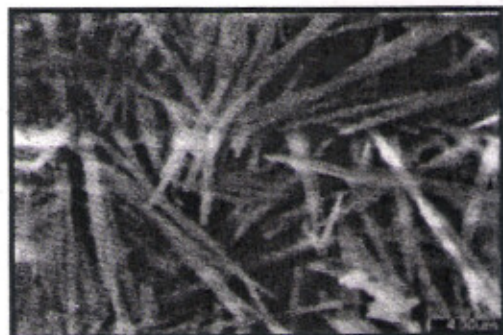


Fig. 4 Scanning electron micrograph of Barium carbonate ( $\text{BaCO}_3$ ) nanorods

#### XRD Spectra

The x-ray diffraction patterns of  $\text{BaCO}_3$  and  $\text{BaC}_2\text{O}_4$  are shown in figure (5a) and (5b). XRD pattern shows that (1,1,1) hkl indices of pure orthorhombic witherite structure of  $\text{BaCO}_3$  is the strongest peak suggesting that  $\text{BaCO}_3$  crystals obtained mainly grow along with (1,1,1) face, whereas in case of  $\text{BaC}_2\text{O}_4$ , (2,0,0) indices of monoclinic structure is the strongest peak. Along with other several strong diffraction peaks, XRD pattern suggest that the crystallinity of  $\text{BaCO}_3$  and  $\text{BaC}_2\text{O}_4$  particles obtained is very good. This crystallinity may be attributed to interfacial homogeneity nucleation in reverse micelles. The size of the particles was also calculated by the width of highest peak using Debye-Scherrer's formula [8] and was found to be in good agreement with size calculated using TEM measurements.

TEM micrographs of Barium Oxalate Nanorod and Iron oxalate nanocrystal are shown in Figure 6 (a,b) using Transmission Electron Microscope (200 kV FEI, Amsterdam). The morphology of these nanostructures reveals their crystalline nature.

Our investigations also reveal some interesting feature in case of Cadmium Sulphide. When we changed the co-surfactant n-butanol by n-

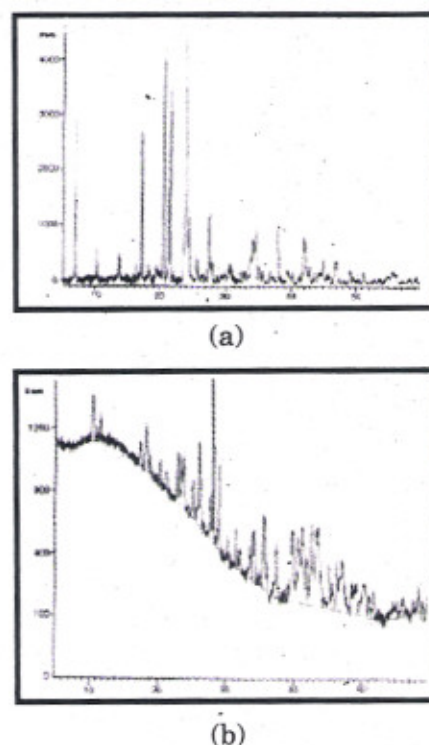


Fig. 5 (a) X-ray diffraction pattern of Barium Carbonate ( $\text{BaCO}_3$ ) and (b) Barium Oxalate ( $\text{BaC}_2\text{O}_4$ )

hexanol, we observed the CdS nanodots convert to nanoneedles as shown in TEM micrographs of Figure 7(a,b).

#### Our Future Outlook

After installation of universal NT-MDT M4 SPM in our laboratory, our thrust area program is focused on fabrication of quantum dots and nanowires using the Anodic Alumina and Ion crafted polymer Templates [9] as well as Reverse Micelle Techniques. Since the dimensions of the dot are in the order of 10 nm or less, one is in a regime where quantum effects become dominant. We propose to investigate single electron effects, as evidenced by Coulomb blockade, observable at even room temperature [10]

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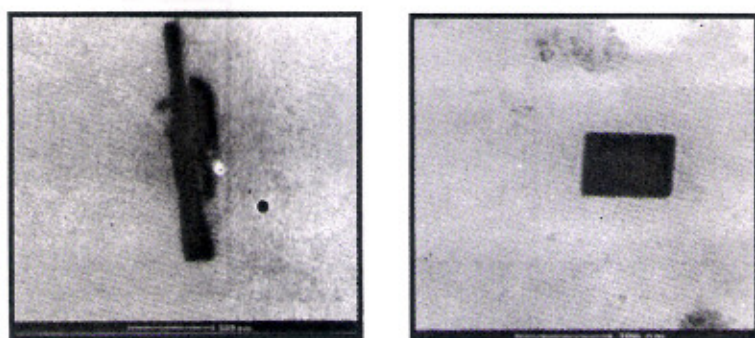


Fig. 6 TEM micrographs: (a) Nanorod of Barium Oxalate (b) Nanocrystal of Iron Oxalate

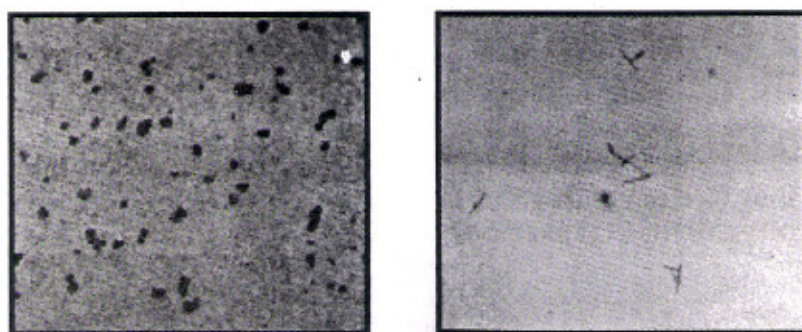


Fig. 7. TEM micrographs: (a) Cadmium Sulphide Nanocrystals, (b) CdS Nan-needles

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