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RESEARCH ARTICLE

HEAVY ION TRACKS IN POLYMERS AND THEIR APPLICATIONS

Amrita Kaur. S*, Jyot Amrita and Virk H. S

[1]Guru Ram Das Medical College and Research, Amritsar 2360 Sector 71, mohali (Pb.) India

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ABSTRACT

Heavy ion tracks in polymers offers a tremendous number of interesting applications in various fields of science and technology. The ion track filters produced by these irradiated polymers have advantage over conventional filters due to its simplicity, small geometry, permanent maintenance of the nuclear records and well-defined pore size. We report here some applications of ion track filters, in micro hydro dynamical flow studies, conduction of bacteria and blood cells, development of metal and metal–semiconductor microstructures and nanostructures. These micro/ nano wires have huge potential for their use as biosensors, field emission studies etc. Nano wires based sensors can detect diseases in blood samples.

INTRODUCTION

Now a days, ion track filters have emerged the main spin-off from nuclear track in solids. Ion tracks are created when high energetic heavy ions with energy of about 1 MeV/nucleon pass through matter (Fleischer *et al.* 1975). The technique that led to the development of etched track membranes was first discovered by (Price and Walker 1967).They found that the damage trails in insulating materials caused by the ionization as a result of the passage of travelling charged particles can be revealed by the chemical etching to form the cylindrical pores. They observed the fine pores due to fission fragments in 12 µm thick layer of synthetic mica. Since 1972, fission fragments from various radioactive sources have been used for the commercial manufacture of nuclear track filters up to 10 µm track length (Price and Walker 1967).Heavy ion accelerators are a promising alternative for generating these filters. These microscopic pores provide convenient confining media in various domains of research for e.g.superconductivity (Possin, 1971), super fluidity (Gamota, 1973) ultrafilteration (Quinn *et al.* 1972) etc. Various new micro porous membranes and filters have been developed for use in the fields of science and technology, viz. health, medicine, air pollution, beverage industries, development of microtubules, material science characterization (Spohr,1990; Fleischer, 1997; Virk *et al.*1998) etc. These filters are generally made from polymeric materials, ceramics and minerals. Ion track filters are mainly divided into two categories single pore filters and multipore filters. Commonly used materials for the production these filters are Makrofol-KG, Kapton-H, PVDF, mica films, cellulose nitrate, CR-39, Lexan polycarbonate, etc. Conventional filters normally depends upon several mutually interconnected processing parameters, where as ion track filters are independent parameters, viz. thickness of film, pore diameter and areal density of pores.

For the present study, the samples of polymer films have been irradiated by different heavy ion beams from the UNILAC accelerator at GSI, Darmstadt, Germany.

Separation of circulating cancerous cells from blood , making use of the fact that cancer cells are larger and more rigid than normal blood cells (Seal, 1964). Water contamination removed from suspended particles (Khan *et al.*1989).

Purification of industrial oil from solid components (Ganz *et al.* 1989).Filtration of unwanted micro particles from liquid and gaseous media (Porter *et al.* 1973; Pakard *al.*1986). Purification of aerosol particles in the atmosphere of industrial plants (Tress *et al.* 1982; Vater *et al.* 1993) etc. Fabrication of microstructures is being considered a potential technology not only for use in micromechanics and microelectronics but also in the studies pertaining to behaviour of materials at micro and nano levels. Microstructures comprising micro dimensional devices, dots, fibrils, wires, cones, tubules and whiskers have invited attention for use in multidisciplinary areas (Spohr, 1990). There are different techniques for the development of microstructures (Wu and Bein, 1994) but template growth through the etched pores considered to be very simple and microstructures with extraordinary low dimensions have been reported (Chakarvarti and Vetter, 1996).

In the present investigation, Escherichia colli and colon bacilli bacteria were grown by using Luria broth technique. The malignant blood cells of size 7-15 µm were collected from shri guru ram das hospital and research institute, Amritsar,

***Corresponding author:* Amrita Kaur. S**
Department of Physics, Khalsa College, Amritsar (Pb.), India

microhydrodynamical flow studies on various fluids and development of metal and metal-semiconductor microstructures using ion track filters. Plastic replication was also done using these filters.

MATERIAL AND METHODS

Samples of various polymer films, MICA, KAPTON and PVDF have been irradiated by different heavy ion beams from the UNILAC accelerator at GSI, Darmstadt, Germany. Details of ion beams, irradiation and chemical etching parameters are shown in the (Table 1).

Filters in Makrofol-KG

The samples of Makrofol-KG have been irradiated by the ^{132}Xe (14.5 and 5.9 MeV/u), ^{208}Pb (13.6 MeV/u) and ^{238}U (14.0 MeV/u) heavy ions. All the irradiations were made at an angle of 90^o with respect to the surface of the detector. The irradiated samples were cut into small pieces and etched in 6.0 N NaOH solution at various temperatures, viz. 40, 50, 60 and 70°C (Fig.1). The etched samples were dried in the folds of a tissue paper.

The etched and dried samples were scanned under a Carl Zeiss optical microscope. The pore diameters were measured using a calibrated graticule. Multipores of diameter 5 and 10µm and single pores of diameter 2 and 4µm were used for this investigation (Figs. 2&3). The nanopores were also obtain using these samples having thickness 10 µm and fluence 10^8 etched at 27^{o}C for 10 min. in 6N NaOH solution.

Filters in muscovite mica

The samples of mica irradiated by ^{132}Xe ions (14.5 MeV/u and 5.9 MeV/u) were etched in 48 vol% HF at room temperature (29°C). The etched samples were scanned under the optical microscope. As the irradiation was at an angle of 90° with respect to the surface, the etched tracks are rhombic in shape (photomicrograph of the etched pore for ^{132}Xe ion of energy 5.9 MeV/u is shown in (Fig. 4).

Filters of Kapton and PVDF. The pores of Kapton and PVDF were obtained by goinometer having diameters 1.38 µm and 10.12µm As shown in (Figs. 5&6).

RESULTS AND DISCUSSION

Applications of ion track filters

Microhydrodynamical flow studies in various liquids using ion track filters

There exist a variety of techniques for understanding the solute solvent interaction. We have made microhydrodynamical flow studies on various fluids (water, alcohol, acetone) using ion track filters (ITFs) of Makrofol-KG (thickness = 60 µm).

The variation of rate of flow (d*V*/d*t*) with concentration has been studied for two miscible solutions (solute + solvent) at constant pressure. It has been observed that the flow rate decreases with increasing concentration of solutes keeping water as a base in both cases, i.e. propan-2-ol and acetone (Fig. 7).

Conduction of bacteria and blood cells through polycarbonate filters

The conduction of bacteria and malignant cells has been studied through the single and multi-pore track filters. The method used here for the growth of bacteria (*E. coli* and *Colon bacillus*) is Luria broth (LB). For the preparation of culture, the requirement is trypton 5 g, yeast extract 10 g, NaCl 10 g and distilled water 500 ml. The grown cells of *E. coli* (ball shaped) and *C. bacillus* (rod shaped) have diameter of the order of 1 µm. The malignant blood cells of size 7–15 µm were collected from Sri Guru Ramdas Hospital and Medical Research Institute, Amritsar. The conduction effect through the polycarbonate pores (sieves) has been observed by using the conductivity cell (Kaur and Virk, 1995). A membrane partition between the two chambers served as a barrier to bacteria and cell migration and the change in current shows the resistance to the flow of the contaminants through the pores.

The resistance through the pores of different diameters is measured using Ohm's law and the change in resistance (Fleischer, 1975) ΔR, with respect to pore diameter is obtained by using the relation $\Delta R = 4r\, d^3/p\, D^4$ and the corresponding resistivity of the solution is found by $r = RA/l$, where R is the resistance, A the area of cylindrical shaped conductivity cell and l the length of the hole inside the cell. It had been observed that the resistance between the two electrodes depends primarily on the conducting path through the pore. As a particle enters, the resistance increases by an amount proportional to the volume of the particle. It is observed that conduction is reduced progressively with increasing concentration of pollutants if the negative polarity is given to the polluted water and positive polarity to the clean water. Taking different concentrations of the contaminated water and human blood, the conduction through the pores of different diameters has been studied. The diameters of microfilters used here are: multi-pore (2 and 4 µ m) and single pore (5, 7 and 10 µm). The resistance through the pore is highly sensitive to geometry and is proportional to d^3/D^4, where d is the diameter of the particle and D is the diameter of the pore. The variation of change in resistance with concentration and pore diameter for single pore filter is shown in (Figs. 8 &9) respectively. A similar variation is observed in the case of multipore filters. (Fig. 10) shows the variation of current and voltage observed in polluted water using nanopore filters.

It is observed that the conduction is reduced progressively with increasing concentration of bacteria and blood cells. The change in resistance encountered by the bacteria and blood cells through a 10 µm pore is found to be lower than that through a 5 µm pore in case of a single pore filter, and in case of multipore filters, 4 µm pore is found to conduct better than a 2 µm pore. But since the blood cells have the property of deforming their shape while traversing through the pores, the conduction in case of blood sample is better than in case of *E. coli* and *C. bacillus* in water. Current through nanopores increases exponentially with the increase in voltage.

Development of metal and metal–semiconductor microstructures through ion track filters

The methodology of the development microstructures is based upon the earlier work of (Possin, 1970) and (Penner and Martin, 1987) producing thin metal wires, etc. This simple and well known underlying concept of electrode position of metals is described as an electrochemical process in which metallic ions in supporting solution are reduced to the metallic state at the cathode if it is covered by an ion track membrane, and thus would lead to the formation of growth of plated film as embodiment of micro and nano-structures (Molares *et al* 2004;

Kumar and Chakarvarti, 2006; Bandyopadhyay *et al* 2003; Ganguli and Tokeer, 2007) . After chemical dissolution or peeling off the polymer film from the metal substrate, the free metallic whiskers are obtained. In the present work, we describe the simple method of electrodeposition of copper into the etched pores of polymeric ITFs by using an electrochemical cell (Kaur and Virk 1995). The electrolyte used here is $CuSO_{4.}5H_2O$ acidic solution in high resistivity ion-free water + 25% vol. H_2SO_4. A current of 0.005 A/cm^2 was applied for 20 min. After the electrodeposition was over, the electrolyte was drained out and the copper electrode was detached from the copper substrate. After drying, the Makrofol-KG thin film was removed from the microstructure substrate by dissolving in chloroform ($CHCl_3$) followed by rinsing with water and ethanol. The developed microstructures were coated with gold by the sputtering method and then scanned under scanning electron microscope (SEM) (Jeol, JSM-6100) for morphological and structural studies. The microstructures of copper (Cu) grown through the single and multipore ITFs of Makrofol-KG re shown in (Fig.11) respectively. The metallic needles developed by this technique have various possible applications, e.g. field ion emitters, as a stylus in STM, cantilever of AFM, etc. The 3-dimensional heterostructure of Cu-Se was grown using electrolytes of $CuSO_4.5H_2O$ + 25% of dilute H_2SO_4 and $Na_2SeO_3.5H_2O$ by template synthesis technique (Fig.12). Virk has developed copper nanowires grown in AAM of 200nm diameter and length of few micron as shown in Fig.13 (Virk, 2009). We have developed 250nm diameter nanorods using makrofol KG observed under Carl Zeiss Supra 55 Scanning Electron Microscope at an accelerating voltage of 10 KV as shown in (Fig, 14).

Track (Plastic) replication

Track replication is considered to be one of the most appropriate methods for evaluating the significant track parameters viz. length, diameter, bulk track etch rates, dip angle and shape etc. (Vetter *et al.* 1990). A simple method which can be used is to dissolve the given polymer in an organic solvent and permit to settle down this viscous fluid through the membrane. After it get dried up, the microstructure is pulled up with adhesive tape. We have developed plastic replication of the etched pores of mica and PVDF ion track filters after scanning under SEM (JEOL, JSM-6100) (Fig. 15&16)

Filtration applications

Filtration is the process of removing physically-suspended matter from a given volume of liquid or gas by forcing the material through a porous, mechanical barrier or a filter. This facilitates the extraction and analysis of the material separated from the fluid or gas. The filtration efficiency is defined as the ability of the media to distinguish between particles of different specific sizes. Ion track filters have advantages over the other conventional filters due to their well-defined pore size which makes it possible to remove all particles bigger than its pore size. In this investigation, the contaminated water was filtered using a filter apparatus.. The photomicrograph (Fig.17) shows the filtered *E. coli* and *C. bacillus* by ITF. The removal efficiency depends upon the pore size of the microfilter and also on the diameter of the pollutant.

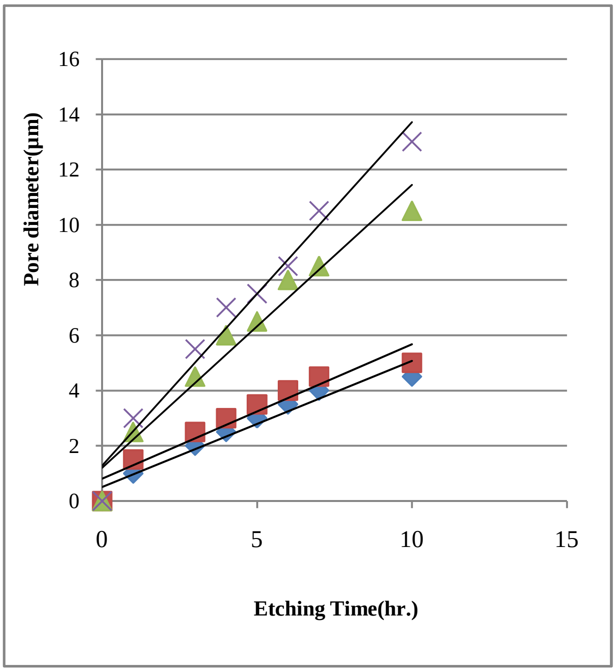

Figure 1 Variation of pore diametr with etching time for ^{132}Xe (14.5 MeV/u) at various temperatures of the etching solution in Makrofol-KG

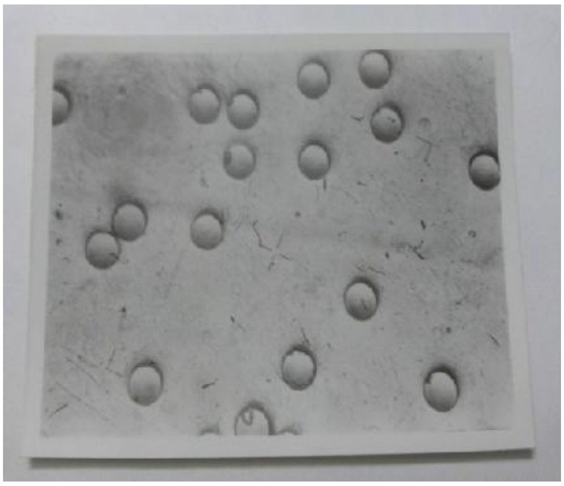

Figure 2 Photomicrograph of ^{132}Xe (14.5 MeV/u) ion track multipores (~21.68 µm)

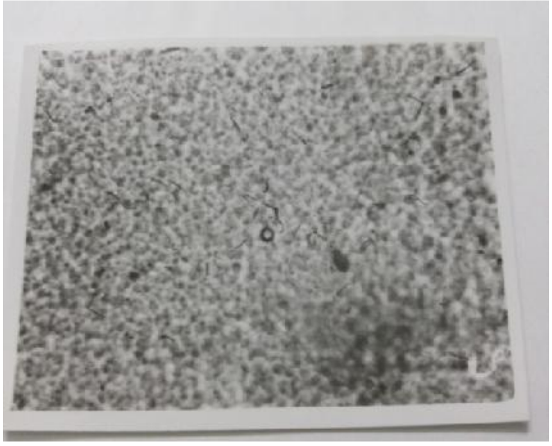

Figure 3 Photomicrograph of ^{132}Xe (14.5 MeV/u) ion track singlepore (2µm) in Makrofol-KG

Figure 4 Photomicrograph of ^{132}Xe (5.9 MeV/u) ion track pores (major axis ~26.9 µm and minor axis ~17.71 µm) in muscovite mica

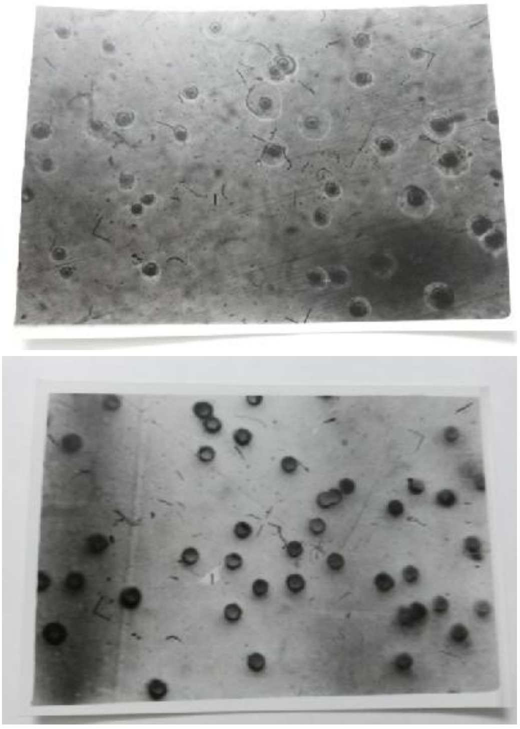

Figure 5&6 Photomicrograph of Kapton (~2µm) and PVDF (10.12µm)

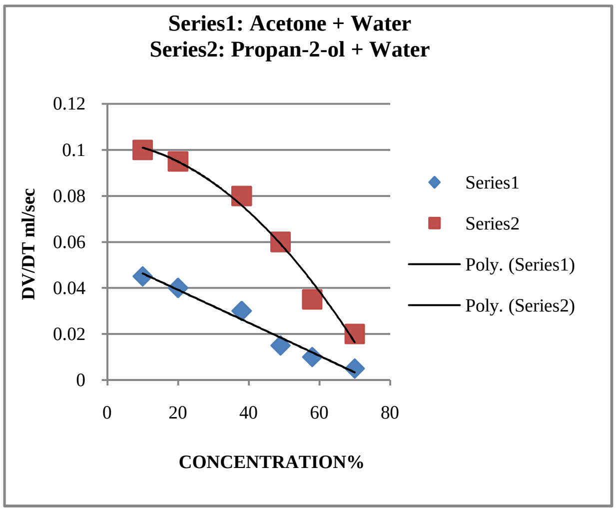

Figure7 Variation of flow rate, d*V*/d*t* with concentration of solutes in water at a constant pressure difference

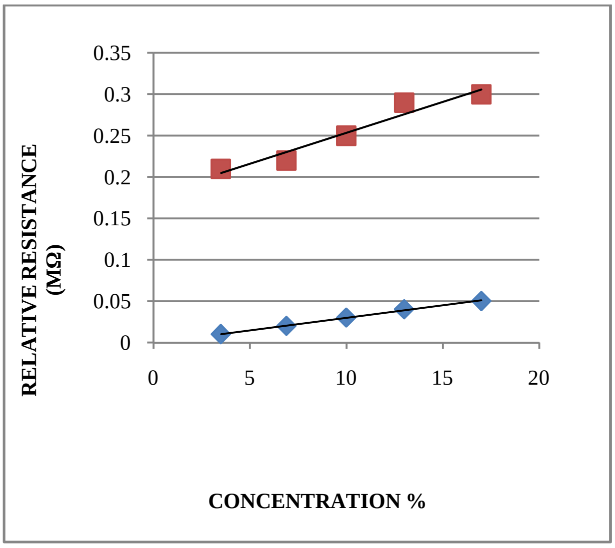

Figure 8 Variation of change in resistance with concentration using single pore filter for *E. coli* and *C. bacillus* in water

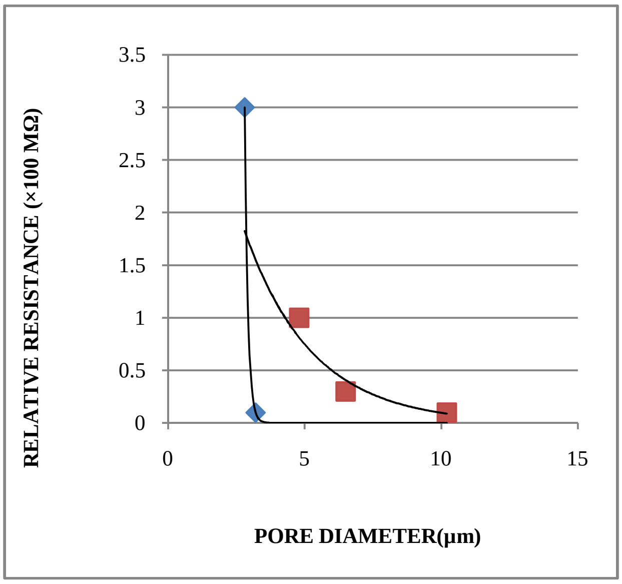

Figure 9 Variation of change in resistance with pore diameter for *E. coli* and *C. bacillus* in water

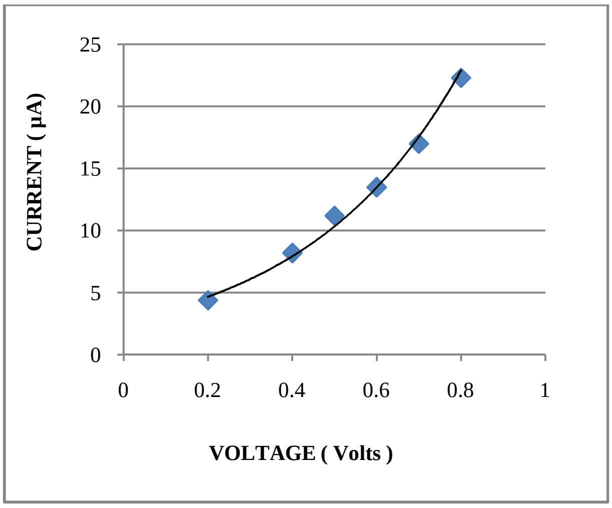

Figure 10 V-I variation observed in nanopores using polluted water

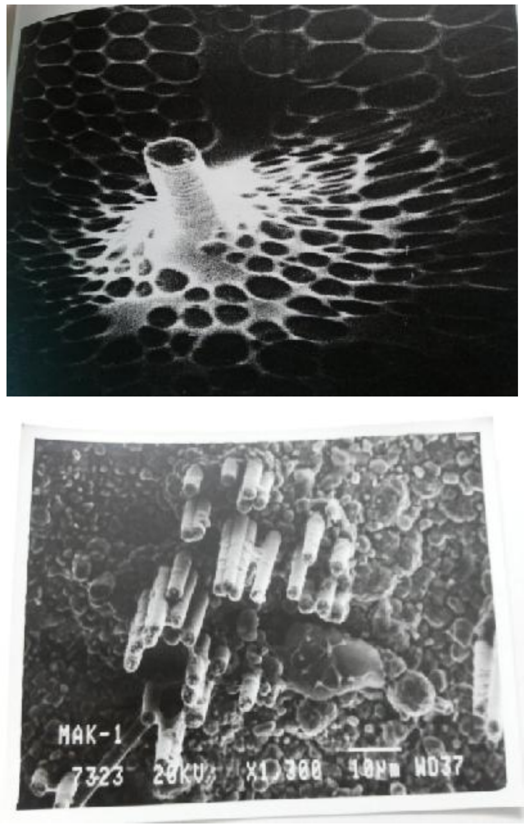

Figure 11 Microstructure (scanned by SEM) ensembles of Cu grown electrochemically through single-pore and multipore filters of Makrofol-KG

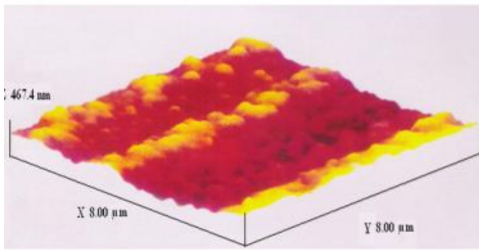

Figure 12 AFM image of 3-dimensional ensemble of Cu-Se grown electrochemically using ITFs of Makrofol-KG

Figure 13 SEM Photograph of copper nanowires of 200nm diameter grown by electrodeposition in AAM[25].

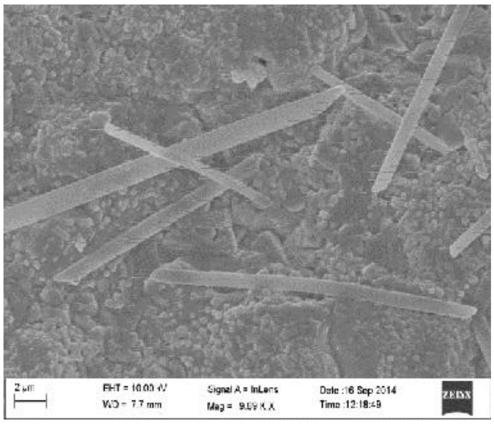

Figure14 SEM Photograph of copper nanowires of 250nm diameter grown by electrodeposition in makrofol- KG.

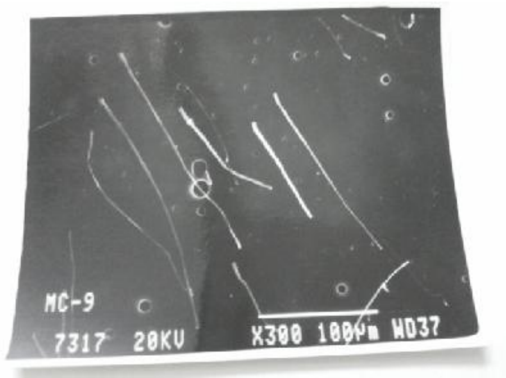

Figure15 Plastic replication of microtubules scanned under SEM of Kapton

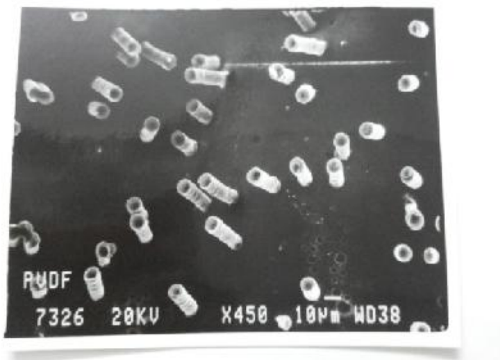

Figure 16 Plastic replication of microtubules scanned under SEM of PVDF

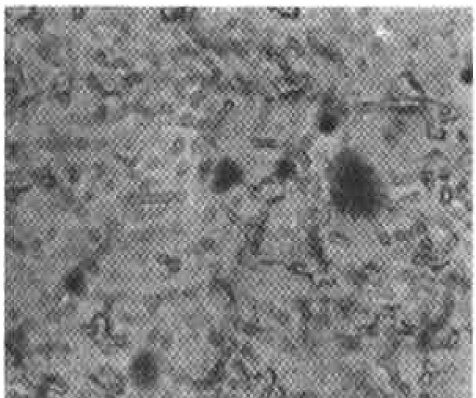

Figure 17 Photomicrograph of filtered *E. coli* and *C. bacillus* bacteria separated by a Makrofol-KG microfilter

CONCLUSION

Heavy ion etched tracks in polymers are most suitable for the tremendous number of interdisciplinary interesting applications in various fields of science and technology. Developments in micro/nanofilters are finding uses in micro/nano-optical, biological (waste water treatment, air purification), energy storage and medical (artificial kidneys) systems. Nanowires based sensors can be detect diseases in blood samples, for this nanowire is first functionalised by attaching nucleic acid molecules to it. If fibrosis gene is present in the blood sample, the conductance of the nanowire changes. The nanowires have huge potential for the use as biosensors field emission studies (Spohr, 2006) etc.

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