# Photoluminescence study of template-synthesized silver microstructures

N. K. VERMA*, SUNIL KUMAR, G. S. SEKHON
*School of Physics and Materials Science, Thapar Institute of Engineering and Technology, Deemed University, Patiala-147 004, India*
*E-mail: nkv_2000@yahoo.com*

S. K. CHAKARVARTI
*Department of Applied Physics, National Institute of Technology, Deemed University, Kurukshetra-136, 119, India*

Nuclear track filter (NTF) or track-etch membrane has emerged as a spin-off from solid state nuclear track detector (SSNTD). The SSNTD is a solid dielectric material capable of storing tracks of energetic, heavily ionizing ions which can subsequently be chemically amplified for optical observation as pores of well-defined geometry and density. The size of the pores depends upon the factors, like the nature and energy of incident particles, the target material, etch conditions, viz. the temperature, nature of etchant, pre-etch storage conditions, etc. The size of pores range from a few nm to mm. Track-etch membranes have been put to various applications besides their use in the synthesis of nano/microstructures [1–3]. Materials such as polymeric sheets, mica, glass, etc. are used for making as track-etch membranes.

'Template synthesis' is a technique to fabricate nano/microstructures. The technique involves synthesis of desired material within the pores of the track-etch membrane. Materials with nano/microscopic dimensions not only have potential technological applications in various areas but also are of fundamental interest as the properties of a material can change in this regime of transition between the bulk and molecular scales. A keen interest is, therefore, being exhibited towards the fabrication of materials like metals [3–5], semiconductors [6–8], polymers [9], glasses [references in ref. 6], etc. The present work reports an indirect method for estimating qualitative change in the resistivity of thermally annealed silver microstructures.

Polycarbonate (PC) samples (monomer composition: $C_{16}H_{14}O_3$; thickness 60 $\mu$m.) were irradiated at normal incidence with $Pb^{208}$ ions (14.0 MeV/nucleon, flux density $10^5$ ions/cm$^2$) employing heavy ion Universal linear accelerator (UNILAC) facility at Gesellschaft fur Schwerionenforshung (GSI), Darmstadt, Germany. The penetration range of ions was kept larger than the sample thickness.

The irradiated PC samples were then etched in electrolytically controlled etching cell [10] using 6N NaOH solution + 10% ethanol at room temperature (27 °C). A sine wave of 1 kHz, 5V peak-to-peak was applied to the electrodes of the etching cell using Audio Frequency Oscillator. The addition of ethanol significantly enhances both the sensitivity of the sample and the rate of etching. The etching was terminated the moment current started flowing in the circuit indicating formation of pores. Fig. 1 shows the scanning electron microscope (SEM) photograph of the developed track-etch membrane.

Silver microstructures are fabricated using an acrylic cell comprising of two hollow compartments provided with O-rings on their inner sides to hold the sample between them when the compartments are screwed together. To fabricate AgI microstructures, $AgNO_3$ solution (0.1 M/l) was taken in one compartment and NaI (1.0 M/l) in the other. The solutions were then allowed to permeate through the NTF pores where the following chemical reaction took place:

$$AgNO_3 + NaI \rightarrow AgI + NaNO_3$$

The silver iodide precipitates into the NTF pores. After the completion of the reaction, the solutions were drained out and the NTF sample, with AgI precipitated into its pores, was taken out, dried in air and gently pasted on an adhesive copper tape. To dissolve the polycarbonate NTF sample, dichloromethane was used drop by drop leaving behind AgI microstructures having dimensions of the size of the NTF pores.

Similar steps were taken to fabricate silver bromide (AgBr) microstructures. In this case, NaBr solution was used and the AgBr microstructures were formed as per the following chemical reaction:

$$AgNO_3 + NaBr \rightarrow AgBr + NaNO_3$$

Fig. 2 shows SEM of silver iodide microstructures. Then the silver iodide and silver bromide microstructures were irradiated separately with ultraviolet (UV) laser light from a Nitrogen laser (power = 10 kW, wavelength = 337.1 nm, pulse width = 7 ns). The emission from the microstructures was collected by a fast (2

*Author to whom all correspondence should be addressed.

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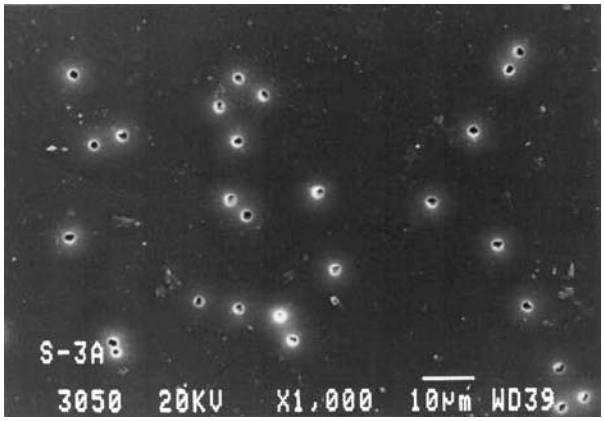


*Figure 1* SEM showing pores of the Nuclear track filter.

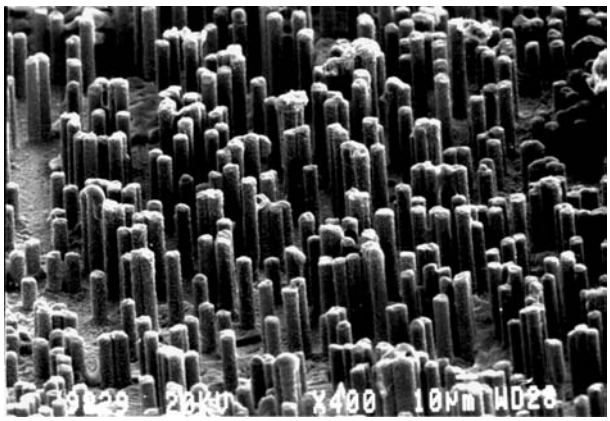


*Figure 2* SEM of silver iodide microstructures.

ns rise time) photomultiplier tube (PMT) through an assembly of monochromator as a wavelength selective element and a glass slab to filter out the UV radiation. The signal from the PMT was fed to a Digital storage oscilloscope (Tektronix make TDS-220) interfaced (RS-232) with a computer to record decay curves. To investigate the effect of annealing on resistivity of the silver microstructures, the above process was repeated after thermally annealing the microstructures first for one hr at 80 °C, and then further for another one hour at 120 °C.

The results are shown in Fig. 3 indicating their decay behavior and, the corresponding peaks giving the emission wavelengths.

Table I shows the emission wavelengths for unannealed AgI and AgBr microstructures; these are respectively 620 and 583 nm. On annealing, the emission wavelengths for both the silver microstructures are found to shift to the lower wavelength side. Corresponding to temperatures 80 ° and 120 °C, the new emission wavelengths become 565 and 454 nm for AgI microstructure whereas 540 and 460 nm for AgBr microstructure (see Table I). The decrease in emission peak wavelengths indicates increase in band gaps of the silver microstructures, i.e. increase in their resistivity.

TABLE I Annealing time vs. emission wavelength

| Name of compound | Annealing time (hr) | Annealing temperature | Emission wavelength (nm) |
|---|---|---|---|
| AgI | Unannealed | 27 °C (Room temperature) | 620 |
| | 1 | 80 °C | 565 |
| | 2 | First for 1 hr at 80 °C and then again for 1 hr at 120 °C | 454 |
| AgBr | Unannealed | 27 °C (Room temperature) | 583 |
| | 1 | 80 °C | 540 |
| | 2 | first for 1 hr at 80 °C and then again for 1 hr at 120 °C | 460 |

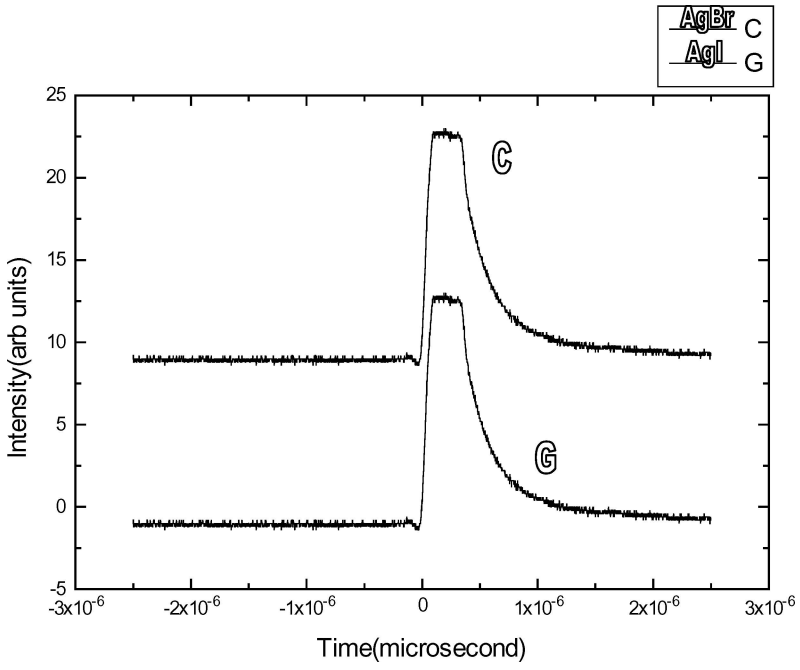


*Figure 3* Decay curves of AgBr and AgI.

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