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Enlarged Nuclear Tracks in $FeCl_3$ doped Polymeric Membrane

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ABSTRACT

Polymer membranes are widely used in separation technology. Separation performances of such membranes depend on their track or pore size. Sometimes it is challenging to optimise the size in transparent membrane. Here, attempt has been paid to overcome this issue. In present work, we propose the development of nuclear tracks in transparent polymer membrane. Transparent membrane was doped with FeCl3 at very low concentration. Objective for such low doping is just colour the membrane from transparent to yellowish to visualisation of tracks. Irradiation of heavy nuclear particle was used to create loosely bounded structure. These doped irradiated membranes were allowed to etch chemically. Relatively longer etching makes these tracks visible under digital optical microscope. We have reported the variation in track size with etching time.

Key words: Polycarbonate, nuclear tracks, irradiation, chemical etching, digital microscope

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INTRODUCTION

Nuclear tracks in solids were studied in past few decades. These tracks can be developed in metal, thin film semiconductors and in thin polymer sheets i.e. membranes. Size of track halo depends on type of radiation as well as nature of material [1-4]. Doping of any foreign material in polymer matrix can change its appearance and other physical properties. The concentration of doping material plays important role on its properties. This can also modify the physical properties at certain extent. Size of tracks have been studied and reported that it varies from nm to several micron. Chemical etching is employed to enlarge these tracks. Etching conditions i.e. concentration of the etchant, temperature and duration will affect the ultimate size of tracks. Polymer materials are largely affected by ionizing radiation; however surface properties of polymers are greatly depends on non-ionizing nature of radiation. Visualization of nuclear tracks under optical microscope is challenging job for transparent material. For this, it is required to use dopant in polymer matrix to produce transmission gradient.

Chemical etching involves a chemical reaction between etchant to attack the membrane surface [2]. The passage of ionized or nuclear particles through polymer membrane creates narrow paths of intense damage on an atomic scale. These damage tracks may be revealed and made visible in an ordinary optical microscope by treatment with a properly chosen chemical reagent that rapidly removes the surrounding undamaged matrix in such a manner as to enlarge the etched holes that mark and characterize the sites of the original, individual, damaged regions [5-8]. The polymer materials damaged during chemical etching simultaneously via two different ways: (i) bulk etching and (ii) track etching, the associated physical parameter with these are bulk and track etch rate. Track etching is relatively high compared to bulk etching. Fig. 1 shows the sequential methodology for generation of nuclear tracks in dense polymer membrane. Fig. 1 (a) is a dense membrane of several micron thickness; Fig. 1 (b) shows the passage of nuclear particle through dense membrane; Fig. 1 (c) shows the opening of these tracks while etching is just employed, the loosely dense material dissolve in the etchant to left the track wider. In case of continuous etching a permanent track structure is being developed as shown in Fig. 1 (d). Now these wider tracks can be visualised under optical microscope [9-10].

In present work, we propose to develop the nuclear tracks in transparent polymer membrane. Transparent membrane was doped with FeCl3 at very low concentration. Objective for such low doping is just colour the membrane from transparent to yellowish to visualisation of tracks. Irradiation of heavy nuclear particle was used to create loosely bounded structure. These doped irradiated membranes were allowed to etch chemically. Relatively longer etching makes these tracks visible under digital optical microscope. We report the variation in track size with etching time.

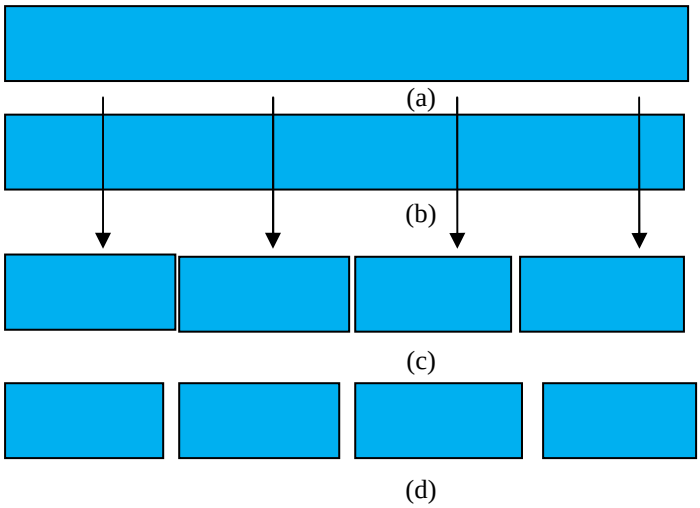

Fig. 1 Nuclear Track Generation in dense polymer membrane: (a) Dense polymer membrane (b) Irradiated Membrane before etching (c) Opening of nuclear tracks in membrane after etching (d) Creation of permanent nuclear tracks

EXPERIMENTAL DETAILS

Materials and Sample Preparation

In present study we have used polycarbonates (PC) as standard material for membrane preparation. PC is a commonly available polymer. It contains carbonate groups (–O–(C=O)–O–) in its backbone structure. The constitutional repeating unit for PC is shown in Fig. 2.

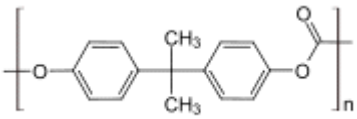

Fig. 2 Constitutional repeating unit for Polycarbonate

$FeCl_3$ is a commercially available material, which was purchased by MERCK. Membrane of few microns was prepared by using solution cast method. Initially, the granules of PC was dissolved in dichloromethane and stirred for 5-6 hours at room temperature. Adding of 0.1 wt% of $FeCl_3$ made this viscous solution yellowish and now it ready for casting. This solution was poured on a flat bottomed pettrie disc and allowed to evaporate the solvent overnight [6]. A 50 micron thick membrane was prepared in this way and used for further experimental process.

Irradiation and Etching

Nuclear particle irradiation was performed at our laboratory. A nuclear source of sufficient half-life was used. These radiations were allowed to fall perpendicularly on doped membrane. A detector was used to confirm the passage of particle through the membrane. Fig. 3 shows the schematic of the experimental setup used for the present study. Initially the set up was calibrated for different time to optimise the dose. It was observed around 6.45 counts/sec for Radium ($^{226}Ra_{88}$) source.

Chemical etching was performed in a constant temperature water bath using 6N NaOH as etchant at 60 °C. Etching time can be increased as and when it required. Increase in etching time will help to increase in size of the track, which will be further characterised under digital optical microscope. The characterization of enlarged tracks was done using commercially available 800x digital microscope.

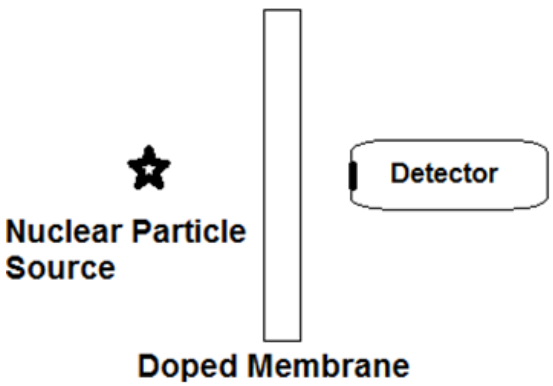

Fig. 3: Irradiation of polymer membrane

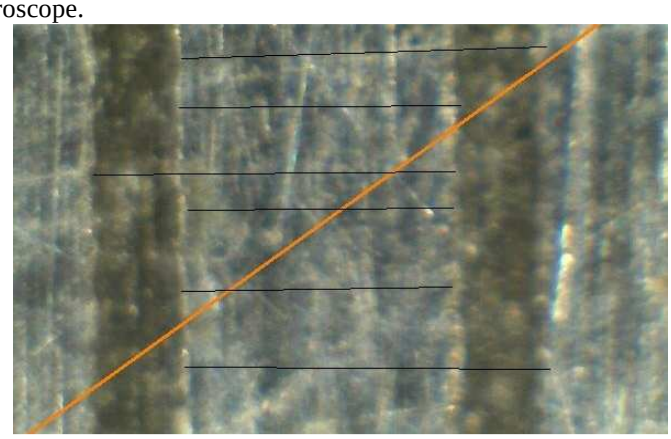

Fig. 4: Calibration with standard scale

RESULTS AND DISCUSSION

The main objective of the present study is to enlarge the tracks developed during irradiation in a controllable manner. Further, these tracks have sufficient size to visualise under digital microscope. Usually, the etching is employed for few minutes in case of polymer samples as initially tracks are at atomic level; they could not reveal under microscope. In this situation we must need a very high resolution microscope i.e. scanning electron microscope (SEM) or transmission electron microscope (TEM). To make them visualise under digital microscope, it is necessary to keep them for further etching. Secondly, for transparent membrane, it is hard to identify the tracks or pores hence the doping is very helpful to distinguish the track area from its surroundings.

The images for different etching time are compared with fresh (virgin) membrane. Fig. 5 (a) shows the image for virgin membrane as it is neither irradiated nor etched. Analysis of these images was performed using standard image analysis software, where a standard measurement was calibrated in terms of pixels and size of all other tracks are measured. On calibration we found that 1mm standard distance covers around 332 pixels as shown in Fig. 4. It was observed that the size of nuclear track increases as etching time increases, further the nuclear track density also increase with dose. A very high etching time will overlap the tracks. The average diameters were calculated and results are shown in Table 1.

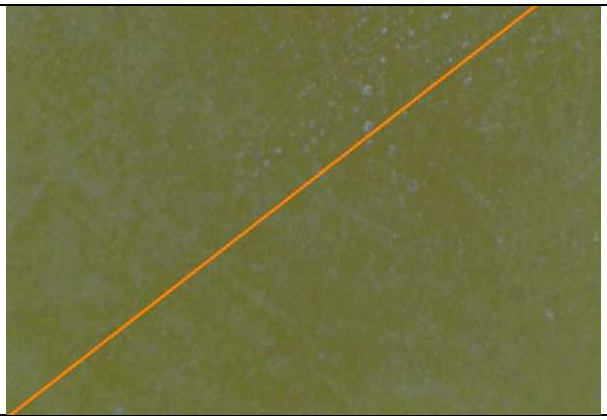

Fig. 5 (a) Fresh neither irradiated nor etched

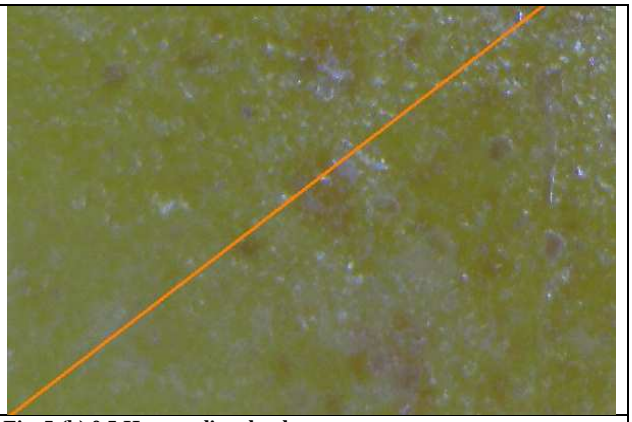

Fig. 5 (b) 0.5 Hour radiated only

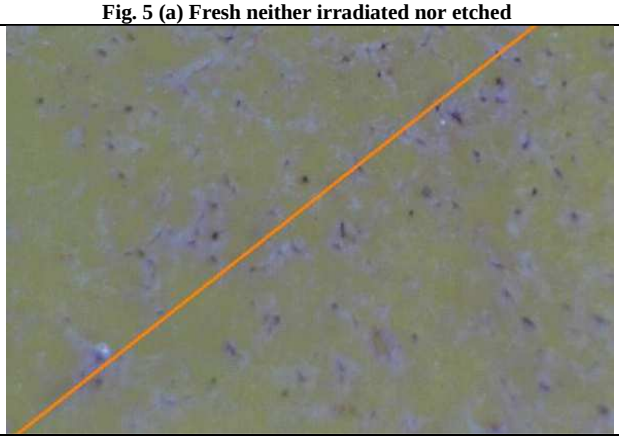

Fig. 5 (c) 0.5 Hour Radiated and 2 hour etching

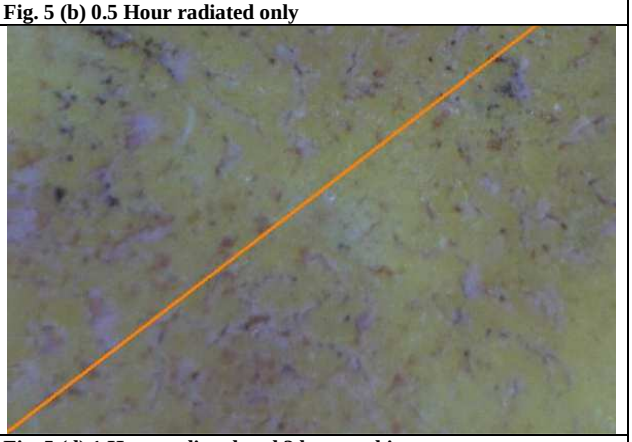

Fig. 5 (d) 1 Hour radiated and 2 hour etching

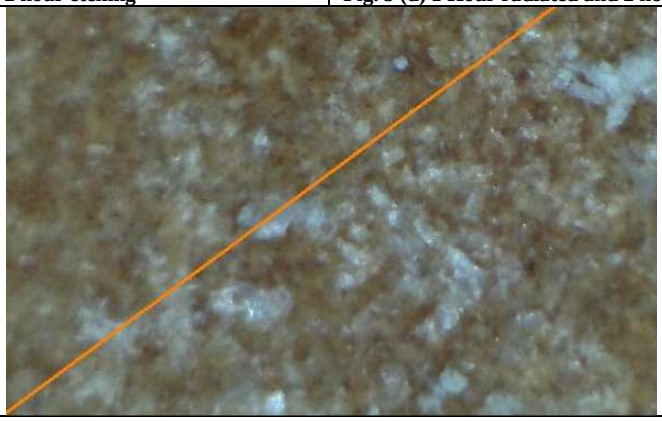

Fig. 5 (e) 0.5 hour radiated and 4 hours etching

Table -1 Average Track Diameter

Etching time	Track diameter (in micron)	
	30 minutes irradiation	60 minutes irradiation
120 minutes	25.55	135.42
240 minutes	321.93	--

CONCLUSION

It is concluded from above study that the size of nuclear track increases with increasing etching time, further the nuclear track density also increase with increasing dose i.e. irradiation time. A very high etching time will overlap the tracks.

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