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Production of multi-, oligo- and single-pore membranes using a continuous ion beam

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A B S T R A C T

Ion track membranes (ITM) have attracted significant interest over the past two decades due to their numerous applications in physical, biological, chemical, biochemical and medical experimental works. A particular feature of ITM technology is the possibility to fabricate samples with a predetermined number of pores, including single-pore membranes.

The present report describes a procedure that allowed for the production of multi-, oligo- and single-pore membranes using a continuous ion beam from an IC-100 cyclotron. The beam was scanned over a set of small diaphragms, from 17 to ~1000 μm in diameter. Ions passed through the apertures and impinged two sandwiched polymer foils, with the total thickness close to the ion range in the polymer. The foils were pulled across the ion beam at a constant speed. The ratio between the transport speed and the scanning frequency determined the distance between irradiation spots. The beam intensity and the aperture diameters were adjusted such that either several, one or no ions passed through the diaphragms during one half-period of scanning. After irradiation, the lower foil was separated from the upper foil and was etched to obtain pores 6–8 μm in diameter. The pores were found using a color chemical reaction between two reagents placed on opposite sides of the foil. The located pores were further confirmed using SEM and optical microscopy. The numbers of tracks in the irradiation spots were consistent with the Poisson statistics. Samples with single or few tracks obtained in this way were employed to study fine phenomena in ion track nanopores.

1. Introduction

Artificial nanopores in polymer foils, fabricated using the track-etching method, have attracted tremendous interest due to their application in science and technology on the nanoscale [1–4]. Considerable research activity has been focused on single nanometer-sized asymmetric pores. The technique for producing single ion tracks in thin plastic films has become important in connection with extensive research in the field of novel nanofluidic phenomena and the development of miniaturized devices, such as molecular valves, ion pumps, and logic elements [1]. Apart from single-pore membranes, membranes with few pores (the number of which is known) are also useful in microfluidics [5,6]. The production of single- and oligo-pore membranes generally requires sophisticated equipment, including a heavy-ion accelerator, a specialized beam line with a detector to detect single hits and a beam switch capable of interrupting the beam after every hit [1,7,8]. A simple procedure allowing irradiation with a small number of ions, including single hits, was arranged on a U-300 cyclotron in the 80s [9]. The ion beam intensity was reduced by small apertures fabricated using the ion track etching technique. Separate irradiation spots (with few or single tracks) were obtained based on two factors: the target was transported across the beam, and the beam consisted of pulses 1.5 ms in length, with a duty factor of 25%. In the present work, we employ a similar approach with the difference that the beam is continuous and the interruptions are arranged using scanning at a certain frequency. We explore the potential benefits and drawbacks of this inexpensive and simple method.

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2. Experimental

2.1. Irradiation

The irradiation of polymer foils was performed in a vacuum chamber at the beam line of the IC100 cyclotron (FLNR JINR) [10]. The vacuum chamber includes a web transport mechanism to rewind a polymer foil, typically 300–320 mm wide, and pull it across the ion beam [11]. The foil can be transported continuously at an angle of 90° to the beam direction, at a speed of 1–100 cm/s. A mask with several small apertures was placed in front of the foil (Fig. 1). The beam was scanned magnetically over the mask. In this study, xenon ions with an energy of 170 MeV were used to bombard one or two layers of 12 μm thick polyethylene terephthalate (PET) foils. Two types of diaphragms were used to tune the ion beam intensity and obtain small numbers of ion tracks in the irradiation areas. Thin metal disks, with apertures 200–1000 μm in diameter (see Fig. 2A), were employed to create spots containing multi- and oligo-pore membranes. To obtain single-pore samples, diaphragms with apertures of 17–35 μm in diameter were used. These were fabricated using the track etching technique. Polycarbonate foils (Makrofol) with a thickness of 100 μm and PET foils (Hostaphan GN76) with a thickness of 76 μm, exposed to single ions at UNILAC (GSI, Darmstadt), were exposed to ultraviolet light and etched in 6 M NaOH in water + methanol (80:20, v/v) until the pores reached the desired size. An example of the fabricated aperture is shown in Fig. 2B. To increase the productivity of the irradiation process, the mask included a set of six holders (Fig. 2C), each consisting of a collimator and a diaphragm. In this case, six parallel lines of irradiation spots were obtained in the foil in one run. During irradiation, the beam current was maintained constant, typically within a range of ±10%. The current was monitored using a wire gauge with a transmission of 90% mounted at the entrance to the beam line.

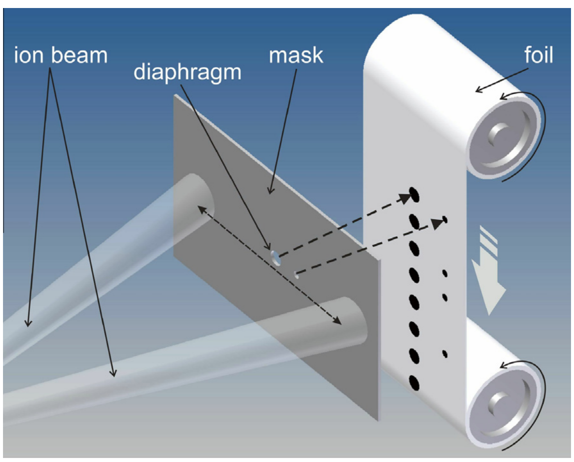

Fig. 1. Sketch (not to scale) of the irradiation process with a beam that is scanned over small apertures in a mask. Two extreme positions of the beam are shown. A larger aperture provides irradiation spots with many tracks. A smaller aperture provides mostly empty spots or single tracks.

2.2. Etching and registration of ion tracks

After irradiation, the foils were etched in aqueous 6 M NaOH at 80 °C to transform the ion tracks into pores. After etching, the thickness of the foil was reduced to 5 μm, thus indicating that pores as large as ~7 μm were developed. The foil was placed on a white web impregnated with a phenolphthalein solution. A thin layer of diluted alkaline solution (1 M NaOH) was uniformly spread over the upper side of the foil, as shown in Fig. 3. The pores in the foil became visible due to a color chemical reaction between the alkali and the chemical indicator. Small specimens, several millimeters in size, were cut from the foil and examined using a scanning electron microscope (Hitachi TM3000) and an optical microscope (Olympus TX43). When samples with non-etched ion

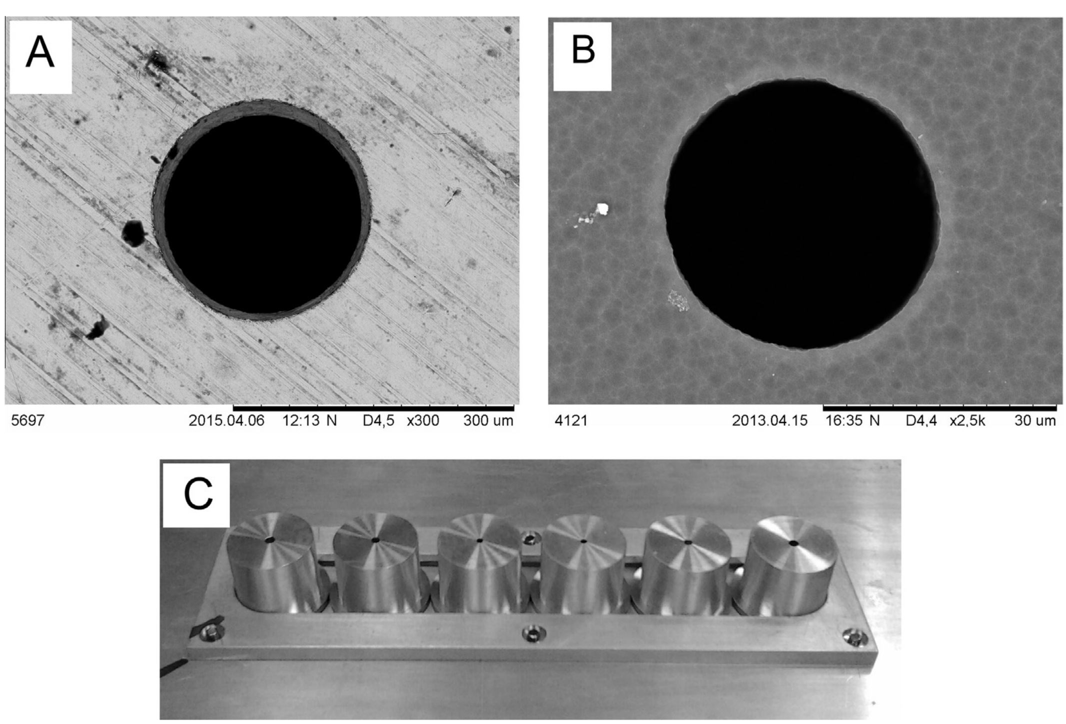

Fig. 2. (A and B) SEM images of diaphragms used to reduce the intensity of the ion beam. Copper foil 0.1 mm thick, scale bar 300 μm (A); PET foil 40 μm thick, scale bar 30 μm (B). (C) Set of six collimators, each with a diaphragm inside.