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## PROSPECTIVE APPLICATIONS OF TEMs

*Article*

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# A New Application of Track-Etched Membranes in X-Ray and Vacuum Ultraviolet Optics

Due to their unique structure properties, track-etched membranes are widely used in scientific and engineering practice to solve specific tasks and perform certain functions. Typical examples are diffraction filters, supports for thin-film X-ray filters and collimators in solar X-ray radiometers. In this paper new non-trivial application of track-etched membranes in optical instruments exploited in vacuum is suggested. Metal-coated track-etched membranes with modified architecture of pore channels can be used as air inlet/outlet elements that block the stray optical radiation within the inner space of the instruments. The membrane consists of two arrays of opaque channels intersecting at a certain angle inside its volume. Both surfaces are coated with light-absorbing and reflective layers of aluminum, which allows background optical radiation to be suppressed by several orders of magnitude. Residual air can pass through the membrane, which reduces the mechanical load on the sensitive elements of the device during fluctuations in external pressure. The developed "black" membranes are promising for use in various X-ray optical devices, including those for space purposes.

*Keywords*: accelerated ions, track-etched membranes, pore channels, X-ray optics, solar astronomy, air in-let/outlet, stray optical radiation, thin-film X-ray filters, solar X-ray radiometers

### *Introduction*

Irradiation of polymers with high energy heavy ions is widely used for nanostructuring of polymers, including production of the so-called track-etched membranes (TMs) [1, 2]. Membranes of this kind have straight pore channels the shape and the size of which can be varied at will. The main characteristics of TMs — thickness, pore density and pore diameter — can be precisely controlled. Due to the unique properties, TMs occupy many special niches in academia and modern technologies.

The design of X ray telescopes and solar radiometers often includes a means that provides the effective absorption of background electromagnetic radiation in a wide wavelength range from X rays to near infrared radiation and, at the same time, serves as air inlet and outlet which allows for fast pumping of inner vacuum space of the instrument. In some cases the means should protect detectors also from electrons and ions. Due to the elimination of atmospheric air from the apparatus, the gas dynamic load on the delicate parts such as thin-film X-ray filters under harsh conditions during the launching into Earth orbit is mitigated [3]. Quite often, relatively massive mechanical devices are used, made of blackened metal, and containing arrays of channels in the form of a multi-pass labyrinth. The channels provide a high flowrate of the residual gas and guarantee the fast vacuumation of the apparatus. Another existing approach is the LIGA technology allowing fabrication of thin plates with narrow channels [4, 5].

 

In the 1970-s and 1980-s, track-etched membranes were reported to be used as the key component of multi-layer thermal insulation [6, 7]. The idea was based on the fact that metallized TMs with submicron and micron pore channels constitute thin-film optical filters and, at the same time, are gas-permeable. This paper presents further development of the bifunctional filter, based on the ideas introduced in Refs [6, 7]. To this end, the morphology of pore space in polyethylene terephthalate track-etched membranes was modified such that to prevent direct propagation of optical radiation through the pore channels. In addition, the membranes were coated with light-reflecting and light-absorbing layers. Physical characteristics of the obtained structures are discussed.

Diffraction filters based on a metal-coated conventional track-etched membrane has been developed and described in previous publications [12]. The membrane having straight pore channels successfully blocked relatively long wave radiation but was not opaque to short wave lengths. In contrast, in the present paper we propose a distinctly different architecture of the membrane, namely the structure with labyrinth channels. This imparts the membrane a new functionality. To the best of our knowledge, such approach has not been reported previously.

*Experimental*

Polyethylene terephthalate (PETP) films with the thickness of 19 and 23 um were irradiated with accelerated xenon ions on the IC100 cyclotron of the Flerov Laboratory of Nuclear Reactions (JINR, Dubna) [8]. The ions impinged onto the film subsequently from both sides at angles of 20 or 45° relative to normal to the surface. (Fig. 1). The ion-irradiated films were treated with soft ultraviolet radiation in order to sensitize the ion tracks and etched chemically thus forming two arrays of mutually intersecting pore channels (Fig. 1). The range of Xe ions with the energy of 160 MeV in PETP was 20–21 um [9]. Therefore, the etched out straight pore channels did not penetrate the whole film thickness. However, the channels were connected to each other in the membrane depth, thus making possible the flow of air through the membranes.

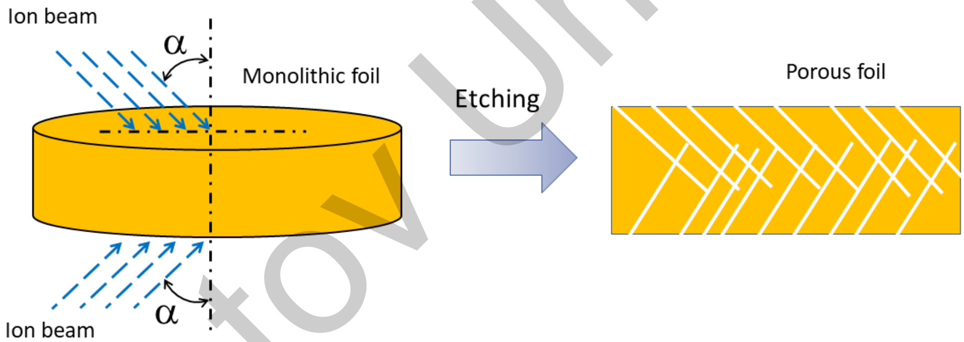


Figure 1. The principle of fabrication of a track-etched membrane having no straight through pore channels

Chemical etching was performed under mild conditions (sodium hydroxide concentration of 1 mol/L, temperature of 60 °C) in order to provide a high track-to-bulk etch rate ratio and obtain the cylindrical pore channels. The scanning electron microscope SU8020 (Hitachi, Japan) was employed to image the membrane samples in the secondary electron mode. The fracturing technique was used for cross-section imaging. Prior to fracturing, the samples were embrittled using a controllable photo-oxidation process, most comprehensively described in Ref. [10]. The specimens were sputter-coated with a thin Pt-Pd layer. The air flowrate was measured on 1 $cm^2$ area of the fabricated membranes using spherical float flowmeters (Gilmont Instruments).

*Results and Discussion*

*Membrane Morphology*

The geometry shown in Figure 1 represents two arrays of parallel pore channels tilted at the angle α relative to normal, with the surface pore density *n* in each array. The ions impinged the foil surface from both sides so that their trajectories cross at an angle of 2π-2α in the side projection. After etching, the pore channels that belong to one array may intersect with the channels of the second array. In order to provide high air flowrate through the membrane, the conditions for multiple pore channel intersections have to be fulfilled.

In the case shown in Figure 1 the mean number of channel intersections (per one channel) *N* can be found using the following formula [11]:

$$N = 4\, n\, d\, H \tan \alpha, \qquad (1)$$

where $H$ is the thickness of the layer with intersections (see Fig. 2), $d$ is the channel diameter. Examples of the morphology of membranes obtained under such conditions are illustrated by the scanning electron microscope (SEM) images in Figure 2. Structural parameters of the membranes are shown in Table 1.

Table 1

**Structural parameters and airflow rates of track membranes used to fabricate the air inlet/outlet device**

| Membrane | Thickness, um | Pore density in each array $n$, $cm^{-2}$ | Tilt angle $\alpha$, ° | Pore diameter $d$, um | Mean number of channel intersections $N$ | Typical airflow at $\Delta P$ = 0.01 MPa, $cm^3\ cm^{-2}\ min^{-1}$ |
|---|---|---|---|---|---|---|
| A | 19 | $2\times10^9$ | 45 | 0.05 | 28 | 12 |
| B | 23 | $6\times10^7$ | 20 | 0.35 | 5 | 100 |
| C | 23 | $1\times10^8$ | 45 | 0.60 | 26 | 800 |

As can be seen in the microphotographs, the thickness of the layer within which the channels intersect one another is approximately 7, 17 and 11 um in membranes A, B and C, respectively. Using the known structure characteristics (Table 1) and formula (1) we can estimate the mean number of channel intersections. This quantity is approximately 28, 5 and 26 for membranes A, B, and C, respectively. In all three cases the number of intersections is large enough to guarantee that practically all channels of one array are connected with channels of the second array [11]. Accordingly, the membranes exhibited a substantial permeability for air in the tests performed under a small differential pressure $\Delta P$ = 0.01 MPa. By varying the channel density, channel diameter and the number of channel intersections, the permeability of the porous foils can be adjusted in accordance with specific requirements.

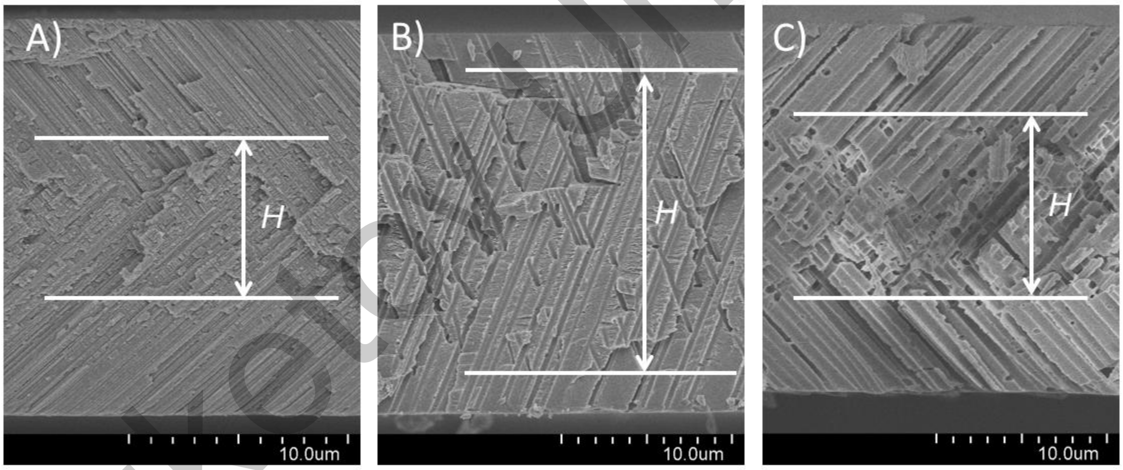


Figure 2. SEM micrographs of track-etched membranes with mutually intersecting arrays of tilted channels. $H$ is the thickness of the layer where the channels intersect. See text for further details

In order to exclude the possibility of formation of straight through channels in the membrane, the thickness $H$ should be markedly smaller than the total film thickness. On the other hand, the value of $H$ should be large enough to obtain a satisfactory number of pore intersections. The examples presented in Figure 2 have been fabricated following this principle.

The number of channel intersections linearly depends on the quantities $n$, $d$, and $H$. The effect of angle $\alpha$ is non-linear in the range of practical interest, $\alpha \in$ [10 to 60°]. Increasing the number of intersections at the expense of an increase in $n$ and $d$ has a limitation caused by the fact that the volume porosity $P$ of the membrane matrix in two outer layers

$$P = n\, \pi\, d^2/4 \cos \alpha \qquad (2)$$

should not exceed 0.2–0.3 (for simplicity, here we neglect the effect of pore overlaps). The gas permeability of a membrane is proportional to the fourth and third power of $d$ at small and large Knudsen numbers, respectively. Therefore, a structure with larger pore diameters is favored with regard to the necessity of fast elimination of residual air. At the same time, the relatively large pore diameter, on the order of 1 um, is acceptable because of a strong suppression of electromagnetic radiation by the mechanism of diffraction filtering [12].

*Light Absorbing Coating*

Absorption of background radiation in different spectral ranges is governed by different mechanisms and caused by different reasons. To make the membranes non-transparent, a two-layer coating was deposited onto the surface of a porous membrane. Aluminum was RF-sputtered in argon plasma using the triode-type sputtering unit Sputron-2 (Balzers). The first layer 100 nm thick (so-called black Al [13]) was deposited at a relatively high pressure of residual gas (~$10^{-4}$ mbar). Then the vacuum chamber was pumped down to ~$5–7\times10^{-6}$ mbar and the second aluminum layer was deposited in an atmosphere of pure argon until the layer thickness reached 80–100 nm. The procedure was performed on both sides of the membrane; the total thickness of metal was ca. 200 nm on each side.

*Optical Properties*

The walls of pore channels were partially covered by the metal, which ensured formation of efficient absorbing optical wedge in the visible range of electromagnetic spectrum [12, 14]. Stray radiation in the red and near infrared ranges was cut by the metal-coated track-etched membrane by the mechanism of diffraction filtering [14, 15]. The specific geometry of the channels array in the membranes excluded the direct penetration of optical radiation. The considerable roughness of the channel walls additionally favored the losses of radiation due to its scattering during the transport through the channel.

The developed "black membrane" possesses two key parameters. The one is the gas flowrate through the membranes at a certain differential pressure and the other one is the optical transmittance. In contrast to the former parameter, which can be easily measured in a wide range, the estimation of the optical transmittance is not a trivial task. In the X-ray region, numerical modeling of distribution of the field amplitude inside micrometer-sized cylindrical pores in polymer track membranes is possible. The refractive index of the polymer matrix is close to 1 (with small real and imaginary additives) in this case, and the calculation of transport of radiation through the pore channels can be performed using a 3D parabolic equation [12, 16]. However, there are no suitable models for the visible and ultraviolet spectrum range and the non-uniform porous bodies. Direct experimental estimate of the optical properties of the black membrane is also not easy because of the necessity to measure light intensities that differ by many orders of magnitude. Our experiments have shown that ordinary track membranes with pores of 0.7–0.9 um in diameter covered by Al layers on both sides provide attenuation of optical radiation by 5-6 orders of magnitude. The measurements have been performed using a filament of incandescent lamp and a set of neutral density filters. In the case of the back membranes with non-through crossing pores the measurement of attenuation is beyond the capabilities of this method. Based on a rough extrapolation, suppression of optical radiation by 10–12 orders of magnitude seems to be plausible.

## *Conclusions*

We suggested a new non-trivial application of track-etched membranes. A structurally modified TM can act as an air inlet and outlet in the optical instruments exploited in vacuum. The membrane contains two arrays of non-through channels that intersect each other in the membrane bulk under a certain angle. Both sides of the membrane are coated with light-absorbing and light-reflecting aluminum layers, which ensures the suppression of background optical radiation by several orders of magnitude. At the same time the residual air can pass through the membrane, which reduces the mechanical load on the delicate parts of the instrument when external pressure changes. The developed "black" membranes can be employed when designing various X ray optical instruments, including those that are exploited in space.

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*Author Contributions*

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript. CRediT: **Alexandr Victorovich Mitrofanov** conceptualization, data curation, investigation, methodology, validation, writing-review & editing; **Pavel Yurievich Apel** conceptualization, formal analysis, validation, writing-original draft, writing-review & editing; **Oleg Mikhailovich Ivanov** investigation, writing-original draft; **Fedor Alekseevich Pudonin** investigation, technology, data curation, formal analysis, writing-review & editing.

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*Conflicts of Interest*

The authors declare no conflict of interest.

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