Nanoscale matrices to transport high-energy beams

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Abstract. It has been studied the process of charged particle transmission through nanoporous alumina membranes with a pore diameter of about 20 and 200 nm. Experiments with irradiation were carried out on the AN_2500 accelerator complex. It was shown that membranes based on nanoporous alumina provide transporting functions to the high-flow helium ions with energies of 1.5-2 MeV. It has been shown than it is possible to realize spatial localization of ion impacts on a substrate with a nanoscale resolution.

1. Introduction
Several recent papers [1-3] have considered the possibility of transporting the accelerated beams of charged particles using a dielectric channel without loss of energy and without losing the initial charge state. It has recently studied actively the use of nanoscale dielectric matrices for the Rutherford backscattering method (RBS). These matrices according to their geometrical parameters can empower the RBS. Namely, they can serve as the basis (template) for ion lithography, focusing the system for a charged beam or may be part of the construction for the study of complex objects to a vacuum (e.g., biological), promoting the exit of the ion beam into the air.

Nanoporous alumina is more and more popular in this area of research due to the technological, low time and resource-intensive, it is also possible to obtain a wide range of different geometrical parameters of porous layers [3–5].

2. Experiment
In order to obtain a porous anodic alumina membrane the electrochemical anodization of aluminum foil (40 μm) was applied in a potentiostatic mode in electrolytes based on aqueous solutions of phosphoric (H₃PO₄) and sulfuric (H₂SO₄) acids for 10–60 min at temperatures from 0 to 15°C. When using electrolytes with different acidities, the speeds of an anions introduction into the porous layer are different. This effect determines the voltage of the pore formation process and consequently the pore diameter. The anodizing voltage was within the range of 15–30 V (for H₂SO₄) or 130–160 V (for H₃PO₄) depending on the electrolyte type. The pore diameter is 18–25 nm and 90–200 nm, respectively.

Experiments with irradiation were carried out on the AN_2500 accelerator complex at Moscow State University [6, 7]. For carrying out the experiment, a special experimental chamber (Figure 1) was created, which includes a collimating aperture, a system of backscattered ion detection and a goniometer system. The intensity of the analysing ion beam was monitored by using a rotary aluminum propeller, its blades, which periodically block the beam, were spattered with a layer of gold a few hundred angstroms thick. Particles, back-scattered on the spattered layer of the monitor target
positioned to block the beam, are recorded by the silicon surface-barrier detector. On the basis of the registered back scattered particles, the intensity of the beam falling on the target is calculated. Ions having passed the monitoring system (while the position of the beam transmission) fall into the experimental chamber.

![Diagram](image)

**Figure 1.** Schematic representation of the experimental process.

The sample under study is installed in the chamber using a goniometer system. A semiconductor detector identifies the signal passed through the membrane of He$^+$ ions and back scattered ions. The angular position of the detector relative to the target can be changed in the scattering plane on a circle which center coincides with the center of rotation of the goniometer. 

Alumina membranes with small (20 nm) and large (200 nm) pores were chosen as researched samples. The foil thickness (10 micrometers) was chosen so that it exceeded the projective range of analyzing particles. According to the program TRIM 2011 calculations, the projective path of He$^+$ ions in amorphous alumina is 3.37 micrometers. The membranes were fixed in a Teflon frame to improve strength and for the ease of use [3]. In this paper the RBS spectra for the porous alumina membrane were measured at the detecting scattering angle 165° и 105°, the energy of the analyzing He$^+$ beam was 1.7 MeV, the ion beam diameter after passing the diaphragm system was 1 mm. The residual pressure in the chamber did not exceed 5•10$^{-4}$ Pa, the current density on the target was maintained constant and amounted to 4 nA.

### 3. Results and discussion

It is known that after anodizing, the oxide on the back side of the film remains “a barrier layer”. As passed through the membrane, the ions will lose energy and scatter at large angles, this layer will negatively affect the transport of ions through the dielectric matrix. Usually for the estimation of value either destructive techniques are involved (SEM, TEM AFM, etc.), or integral methods giving information on the depth (energy dispersive analysis, RSMA, etc.). The present work for a more detailed study used the Rutherford backscattering method. For this purpose the following experiments were conducted (Figures 2, 3): the membrane was located in the chamber so that the ion beam could fall on the front side of the membrane (up), then the sample was taken from the camera, it was turned upside down, so that the beam fell on the opposite side of the membrane (down). Thus, we could assess the elemental composition of the membrane on both sides.
Figure 2. The energy spectrum of backscattered He$^+$ ions with energies of 1.7 MeV for the scattering 120° angle through the membrane 200 nm pore diameter.

Figure 3. The energy spectrum of backscattered He$^+$ ions with energies of 1.7 MeV for the scattering 120° angle through the membrane 20 nm pore diameter.

The obtained results (figures 2 and 3) show that the first (up to channel 300) and the third steps repeat exactly each other (close to channel 500), but the second step (channel 300 – 470), corresponding to aluminum, has a “blockage”. This suggests that oxygen and phosphorus (sulfur) are completely uniformly distributed throughout the thickness, but for aluminum a certain concentration gradient is observed, by which the full or partial depletion of the aluminum substrate after the anodization can be judged. Based on these results, it is possible to evaluate the analyzed samples on elemental composition and its distribution in depth. To study the passage of the ions through the sample some samples with the most uniform concentration of the elements in depth were used.

To evaluate the transmission coefficient and the potential energy change of the ion beam transmitted through the nanoporous membrane is possible by measuring spectra of backscattered ions from the membrane surface and the target surface located outside the membrane. It should be noted that in this case we register only those ions that pass through the membrane.
During the first experiments the target was selected solid aluminium alloy (duralumin) sample. Figure 4 shows the spectra of backscattered ions from both the membrane and that have passed through the membrane and scattered from the target. The transmission coefficient is 0.28. The shift of the backscattered ions from aluminum spectrum from channel 420 (blue curve) to 450 channel (red curve) is due to the peculiarity of the experiment (the detector during the experiments is located at different angles). In addition, the spectrum for the membrane (red curve) shows that the edge of the step corresponding to the scattering of ions from aluminum is flatter. This indicates a decrease of the method resolution in energy. Estimative the resolution decreases at 40 keV.

![Figure 4. The energy spectrum of backscattered He\(^+\) ions from the membrane and passed through the membrane.](image)

It is known that beam energy resolution determines the RBS depth resolution, which depends on the mass of the element. The heavier the element is, the greater is the depth resolution. Therefore, for detecting the energy spectrum experiments on backscattering of ions passing through the nanoporous membrane were carried out with "heavy" elements. In further experiments as the "registering" environment - target a sample containing a heavy element, in particular hafnium (Hf) - HfO\(_2\) (24nm) / Si (100) was located behind the membrane. The given RBS spectrum (Figure 1) strongly pronounces the peak of HfO\(_2\) located behind the membrane. This indicates that in a direction to the normal to the surface the He\(^+\) beam passes through the membrane without losing energy. At that method the depth resolution worsened by only 24 keV. Probably part of the transmitted beam has deviated from the original beam axis, so that the resolution of analysis method deteriorated. The spectrum of a signal from the backscattered oxygen ions from the HfO\(_2\) films did not show. Probably this is due to the fact that some of the ions in the vicinity of the channel wall were scattered while moving through the nanoporous membrane. Thus analyzing ions approaches the target at different angles, which adversely affects the RBS resolution.
Figure 5. The energy spectrum of backscattered He$^+$ ions with energies of 1.7 MeV for the scattering angle $120^\circ$.

It is found that even a slight deviation of the membrane away from the normal (1.5–3 degrees) with respect to the propagation direction of the ion beam results in a reduction of backscattered ions exit HfO$_2$ target and hence to reduce the detected current. This allows to realize the spatial localization of the ion impacts on a substrate with nanoscale resolution.

4. Conclusion
It has been shown that the Rutherford backscattering method can help define the barrier layer thickness and define its composition with high spatial resolution without destroying the sample.

It has been shown that nanoporous membranes based on alumina containing nanoscale uniaxially-oriented system of capillaries provide the functions of transporting the He$^+$ ion stream with energy of 1.5–2 MeV.

It has been found that even a slight deviation of the membrane from the normal (1.5 to 3 degrees) relative to the direction of propagation of the ion beam reduces the yield of backscattered ions from the HfO$_2$ target and, hence, a reduction in apparent current. This enables the spatial localization of ion impact on a substrate with nanoscale resolution.

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