Effect of Aluminum Foil Polishing on the Morphology of Membranes Based on Porous Anodic Aluminum Oxide

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***Abstract*— From the point of view of scientific and practical interest, one of the modern nanomaterials is porous anodic aluminum oxide (PAA), and especially membranes based on it. An important parameter is the morphology of the porous layer. By controlling the pore diameter, it is possible to create a filter for various substances, depending on the task. The structure of the porous layer is largely determined by the quality of the original material. This paper considers options for preliminary surface treatment, in particular, different polishing methods, and their effect on the geometry of the resulting porous layer. SEM and AFM images before and after surface polishing are presented.**

Keywords*— *porous aluminum oxide; surface roughness; mechanical, microemulsion, chemical and electrochemical polishing

I. INTRODUCTION

Membranes based on PAA are in demand in many areas of science and technology [1–5]. The uniqueness of aluminum is that in the process of electrochemical etching under certain technological conditions it is possible to obtain a layer of aluminum oxide with a self-ordered pore structure [6, 7].

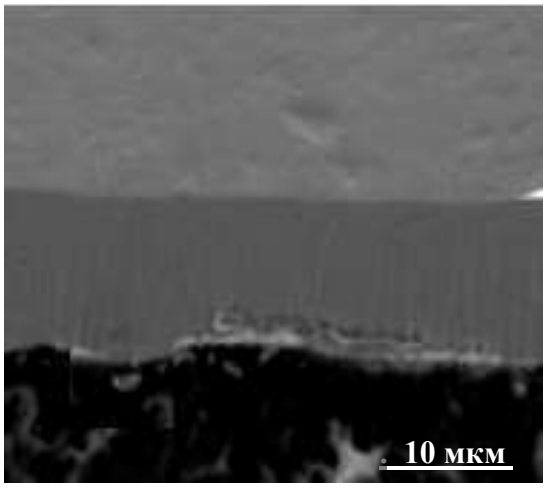
It is known that anodization of aluminum in acid electrolytes can occur with different speeds, efficiency, and stability. The formation of pores does not begin simultaneously over the entire surface of the metal, but in energetically more favorable places, which can be places where dislocations emerge on the surface, slip lines, grain boundaries, impurities, structural defects, etc. Thus, the initial distribution of pores on the surface of aluminum is random, chaotic, and does not obey ordering, but is determined by the homogeneity of the surface. This point plays an important role in the process of pore formation.

At the initial stages of anodic oxidation, a non-porous film is formed. The incorporation of electrolyte anions into the oxide leads to the formation of non-uniformity of the surface potential of the anodic oxide. This leads to the localization of the oxidation process. In the case of a nanostructured surface, the structure of the porous oxide is determined by the morphology of the working side of the original aluminum. This effect is determined by the fact that the change in current density in different areas of the electrode is associated with the non-uniformity of the electric field near this electrode.

The aim of the work was to study the processes of controlled formation of nanoporous membranes based on aluminum oxide with an ordered system of pores created by electrochemical anodization of aluminum foil.

II. SYNTHESIS OF THE STUDIED MATERIAL

Since the aim of the work was to form homogeneous nanoporous membranes based on aluminum oxide, the main object of the study was aluminum foil with a thickness of ~ 10 μm (Figure 1,a). Particular attention was paid to the methods of preliminary preparation of its surface and the actual process of electrochemical anodization, as well as to the study of the structure of the obtained porous membranes.



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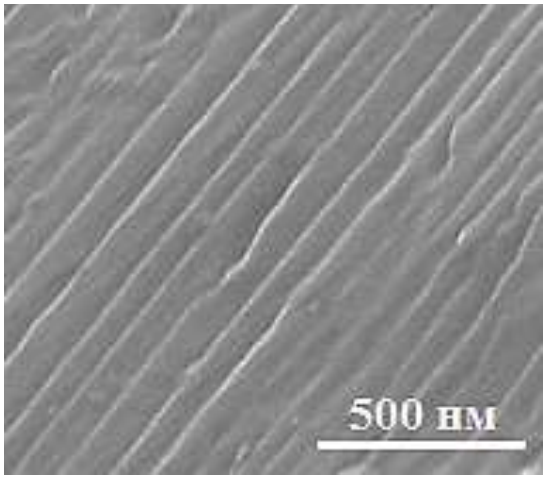

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Fig. 1. SEM image of the original aluminum foil: profile (b) and surface morphology (b)

Aluminum was obtained using the technology of multiple hot and then cold rolling. The original foil, as

shown in Figure 1, b, contains pronounced rolling lines, since the surface roughness of such a foil depends on the surface roughness of the rolling shaft, which in turn is manufactured using the method of turning smooth cylindrical surfaces. The surface profile of this foil has a high proportion of the periodic component in the surface roughness.

The foil surface pre-treated in this way subsequently contributes to the formation of artificially directed pores, limiting their shape and maximum size. In some cases, this can be successfully applied.

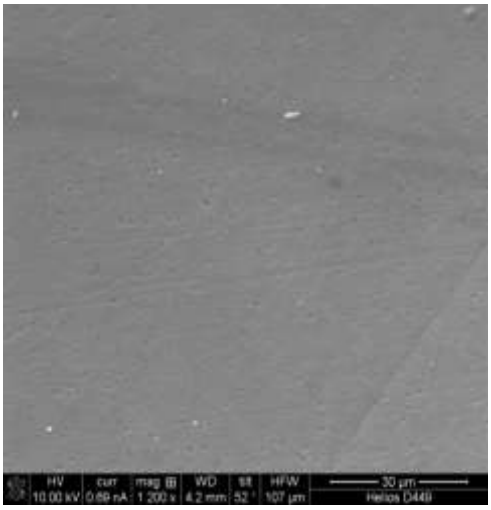
However, the most popular option is the porous structure of aluminum oxide with a hexagonal arrangement of pores. For this, special attention should be paid to reducing the surface roughness of the original foil.

In the work three types of polishing were investigated: mechanical, microemulsion, chemical and electrochemical. For mechanical polishing GOI paste No. 2 (fine) with an abrasive capacity of up to 1 μm was used. For microemulsion polishing a microemulsion in the system of sodium bis-(2-ethylhexyl)sulfosuccinate – kerosene – aqueous solution of acetic acid (1 mol/l) in mass ratios of 38/15/47, respectively, was used, in which an aluminum plate was immersed for 120 minutes at 60°C with stirring at 300 rpm [8, 9]. In the last two cases the aluminum foil was exposed to an electrolyte based on 20% phosphoric acid: for chemical polishing – at a temperature of 40-50 °C for 4 minutes; for electrochemical polishing - the process was accompanied by an applied voltage of up to 180 V.

The study of the original and polished by different methods aluminum foil was carried out using scanning electron and atomic force microscopy.

III. Results And Discussion

The obtained results (Fig. 2) showed that the type of polishing used has a significant impact on the surface quality, which in turn, together with preliminary mechanical faceting of the surface, imposes its own limitations on the pore formation process and on the pore habit.



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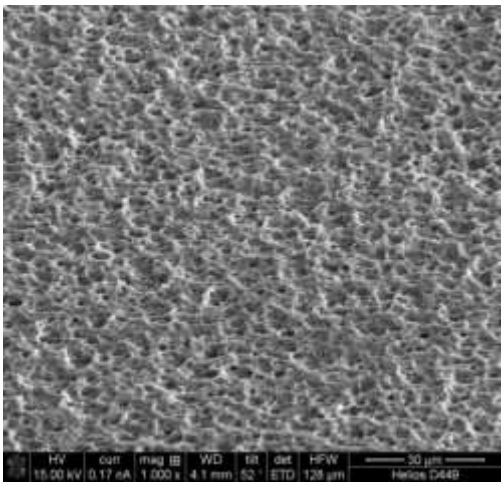

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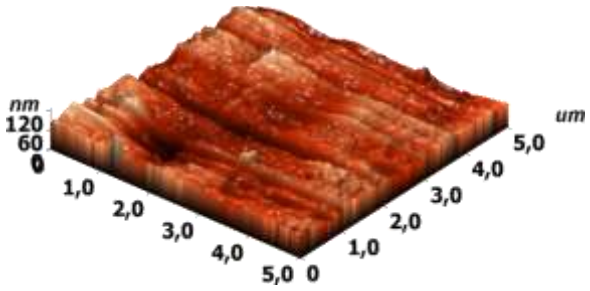
Fig. 2. SEM images of aluminum foil surface after mechanical (a) and chemical polishing.

The results of microemulsion polishing (Fig. 3) showed a reduction in the heights of surface irregularities by almost half while maintaining the periodic component of roughness.



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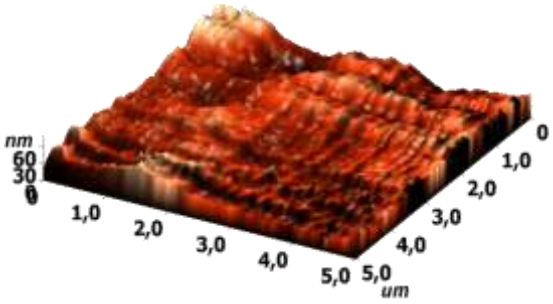

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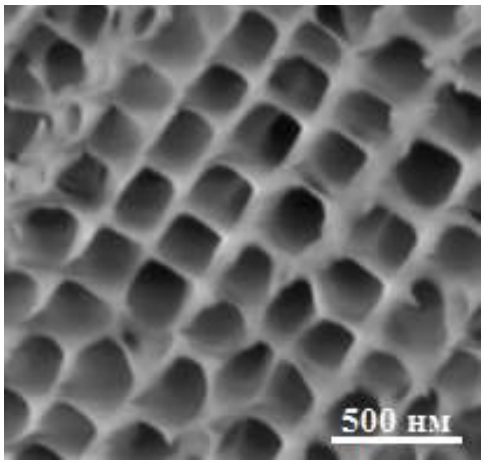
Fig. 3. AFM images of the surface of aluminum foil before (a) and after (b) microemulsion polishing.

To improve the reproducibility of the process of manufacturing nanoporous membranes based on aluminum oxide, it is necessary to use a source foil with uniform surface morphology.

IV. Conclusions

As a result of applying preliminary surface treatment stages, both individually and in combination with each other, PAA layers with an ordered structure were obtained. Typical images are shown in Figure 4.



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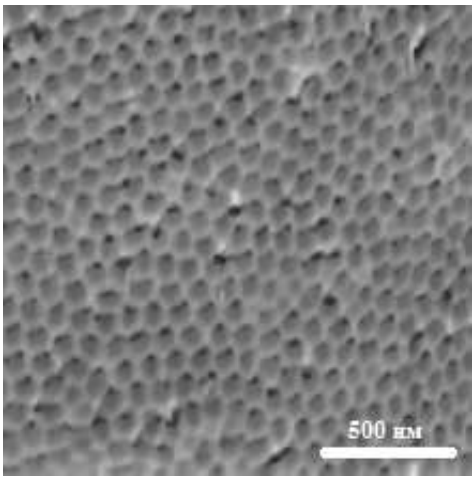

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Fig. 4. SEM images of PAA with different pore geometries

It is possible to control the shape of the pores (square, hexagonal, round) and their distribution relative to each other on the surface by choosing one or another method of preliminary processing of the original aluminum foil.

Thus, it has been established that the most important element of the technological process of forming a system of ordered membranes of nanoporous aluminum oxide by electrochemical anodization of aluminum foil is preliminary surface preparation.

It is shown how the morphology of the original foil affects the pore habit and the formation of a homogeneous structure. It is the morphological characteristics that are key from the point of view of the practical application of nanostructured PAOA membranes.

REFERENCES

[1] Muratova E.N. Micro- and nanosized 3D-ordered capillary matrices based on anodic aluminum oxide, Biotechnosphere. 2014, No. 6 (36), pp. 35–40 (in Russian)

[2] Spivak Yu.M., et al. Porous aluminum oxide for medical applications including targeted drug delivery. Nanobiotechnology Reports. 2023, V. 18, № S1, P. S37–S47

[3] Anufriev I.E., et al. Nanoporous membranes based on aluminum oxide for homogenization of liposomal solution. Vestnik of Novgorod State University, 2023, 1(130), 9–21. (in Russian)

[4] Matyushkin L.B., Muratova E.N., Panov M.F. Determination of the alumina membrane geometrical parameters using its optical spectra. Micro Nano Lett, 2017, V. 12, P. 100–103

[5] Muratova E.N., et al. The influence of the structural parameters of nanoporous alumina matrices on optical properties. Metals, 2024, V. 14, № 6, P. 651

[6] Muratova E.N., PhD thesis [St. Petersburg], 2014.(in Russian)

[7] Muratova E.N., et al. Influence of technological parameters of nanoporous Al2O3 layers' preparation on their structural characteristics. Glass Physics and Chemistry.2013. Vol. 39.No. 3.P. 320-328

[8] Levchishin S.Yu., Tyulyagin P.E., Subcheva E.N., Murashova N.M. Change in the roughness of the aluminum surface under the action of AOT microemulsion containing acetic acid. Successes in chemistry and chemical technology: collection of scientific Tr., 2016, V. XXX, No. 16 (181), P. 22-24.

[9] Tyulyagin P.E., Subcheva E.N., Polyakova A.S., Murashova N.M. Microemulsions of bis-(2-ethylhexyl) sodium sulfosuccinate for leaching and chemical polishing of metals. Chemical technology of functional nanomaterials, 2017, P. 273-275.