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Investigation of the features of the porous morphology of anodic alumina films at the initial stage of anodization

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Abstract. The work is devoted to the study of the porous structure formation of anodic alumina films at the initial stage of aluminium anodizing. SEM images of the surface morphology of the oxalic acid anodic films were analyzed. It was shown that at the initial stage, both major and minor pores are formed, the diameter ratio of which is about 1.16 and does not depend on the anodizing voltage. The results obtained indicate that the minor pores in the anodic films are located inside hexagonal cells composed of the major pores.

1. Introduction

One of the effective methods of forming a material with a controlled porous structure is the process of anodic oxidation of aluminum [1, 2]. Figure 1 shows a laboratory complex for the implementation of electrochemical anodizing processes. Films of porous anodic alumina (PAA) obtained as a result of this process are characterized by a regular porous structure with pore sizes proportional to the anodizing voltage.



Figure 1. Porous anodic aluminum.

The structure of PAA can be represented as a hexagonal close packing of cylindrical pores perpendicular to the film surface (Figure 2). For the widespread use of PAA films as nanostructures in optics, sensors, and nanotechnologies [3], an important condition is the production of anodic films



with high ordering and a given pore diameter. The key role in this is played by the initial stage of the formation of the porous structure of anodic films during anodizing of aluminum, which is poorly studied.

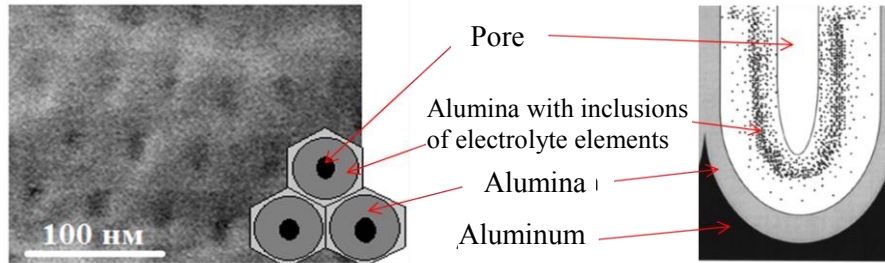


Figure 2. General view of the structure of the porous layer of aluminum oxide: SEM image and schematic representation of the pore profile [4].

At present, the following mechanism has been adopted to explain the pore nucleation process. In the first stage of anodizing (Figure 3), the aluminum surface is covered with a layer of barrier aluminum oxide. In this case, in the depressions and defects of the oxide film, the electric field strength increases sharply. This leads to local dissolution of the oxide layer due to temperature rise and dissolution under the action of an electric field. Due to the competition of neighboring points for charge drain, some of the pores stop growing and, as a result, a uniform growth of the PAA film begins with a constant distance between the pore centers. At the same time, it can be assumed that the development of pores in the initial region of disordered growth has its own characteristics.

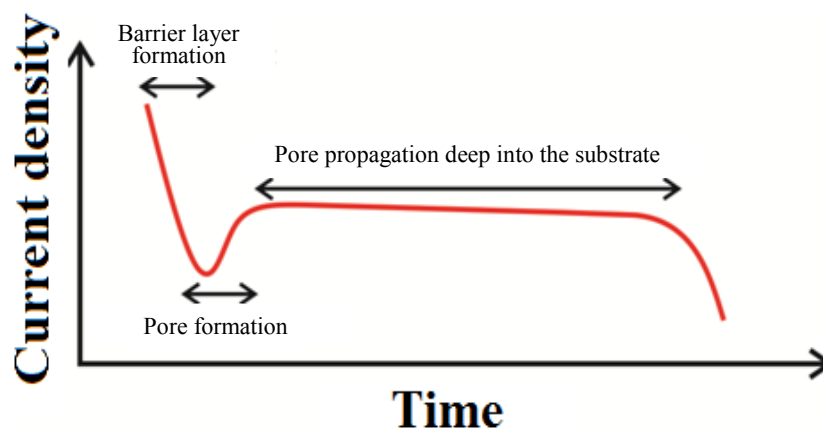


Figure 3. Kinetic dependence of the pore formation process.

The aim of this work was to study the features of the formation of the porous morphology of PAA films at the initial stage of aluminum anodization.

2. Results

Figure 4 shows an appearance of the thin film of PAA and AFM study of anodic alumina membrane synthesized in electrolyte solution based on oxalic acid.

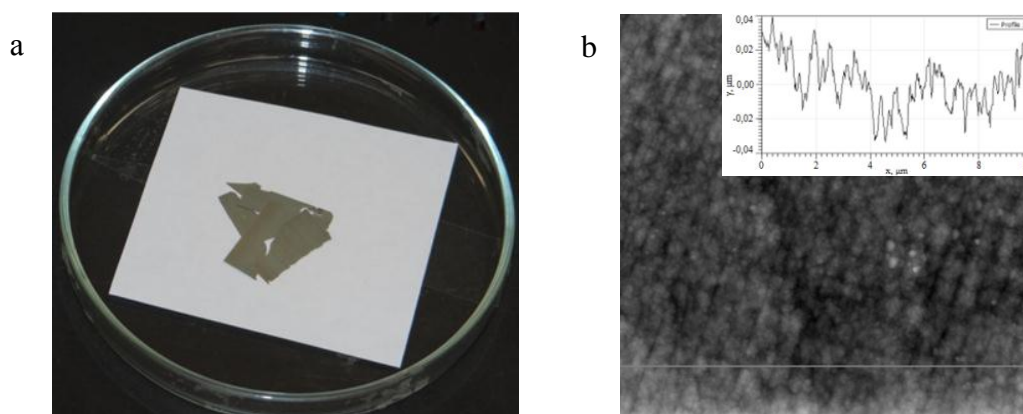


Figure 4. Photo (a) and AFM image (a) of the thin film of PAA formed in a solution of oxalic acid (the scanning area size is $10 \times 10 \mu\text{m}$, with profile line).

Figure 5 (a) shows SEM image of the surface morphology of a porous alumina film on an aluminum substrate obtained in a potentiostatic mode at 40 V in oxalic acid. To determine the pore diameter, computer processing of the surface morphology of the anodic film was carried out using the ImageJ program. Then, according to the processing results, a graph of the pore diameter distribution was plotted (Fig. 5b). As can be clearly seen from the graph, the pore diameter distribution has two distinct peaks at 22.5 ± 0.2 and 26.4 ± 0.2 nm. The major pores have a larger diameter of 26.4 nm, and the minor pores in the PAA film have a diameter of 22.5 nm. This indicates that at the initial stage of aluminum anodization, the simultaneous development of both major pores and minor pores with similar diameters is observed. The results obtained indicate that the ratio of the diameters of such pores in the anodic films does not depend on the anodizing voltage and is equal to approximately 1.16. It should be noted that the number of minor pores is of the same order as the major pores. Therefore, it can be argued with high probability that the minor pores do not have a chaotic distribution in the anodic film, but are located inside hexagonal cells composed of the major pores. At the same time, at the initial stage of anodizing, the shape of such hexagonal cells is strongly distorted.

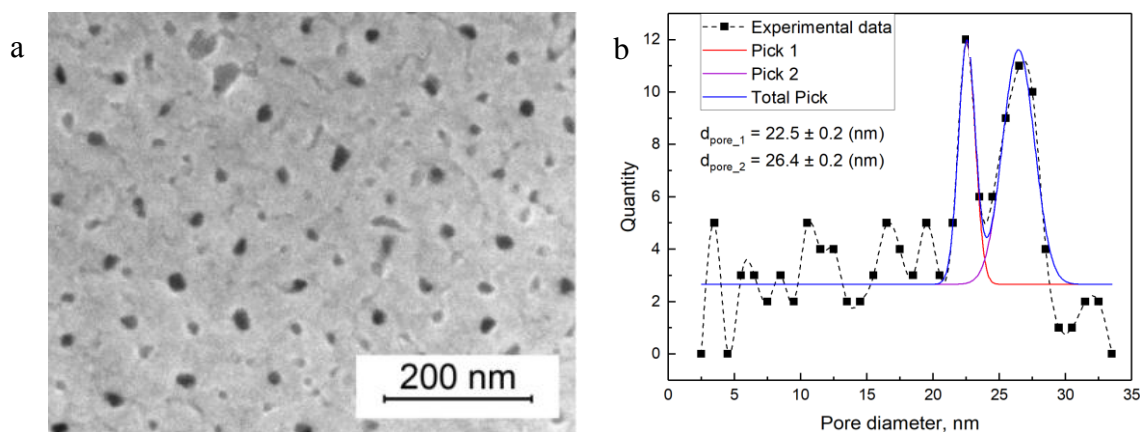


Figure 5. SEM image of the surface morphology (a) and pore diameter distribution (b) for a thin film of porous PAA formed on aluminum in oxalic acid at 40 V for 390 s

The results obtained indicating the presence of major pores and minor pores with similar diameters and the same density in the anodic film are in good agreement with the data presented in [5] for the pore size distribution determined by the method of capillary condensation of isopropanol vapors.

3. Conclusion

It was found that at the initial stage of aluminum anodization, the porous morphology of the anodic films is characterized by the simultaneous development of both major and minor pores of a certain diameter. The ratio of the sizes of the major and minor pores is about 1.16 and does not depend on the anodizing voltage in oxalic acid. The results obtained indicate that the minor pores do not have a chaotic distribution in the anodic film, but are located inside hexagonal cells composed of the major pores.

Acknowledgments

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