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Morphology and optical properties of aluminum oxide formed into oxalic electrolyte with addition surface active agents

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Abstract. The article discusses the results of investigations of porous films of alumina, formed into oxalic electrolyte with addition surface active agents, in particular, ordering structure, roughness of a surface, the optical transparency of the electrolyte concentration and surface active agents. Also discusses the features of the formation of porous films of temperature and IR radiation.

1. Introduction

Presently the quality of image, formed by devices on the basis of liquid-crystal materials became much higher and these devices fully forced out cathode-ray tubes. The best combination of electrical conductivity, optical transmission and processability processing using physicochemical methods etching demonstrates film of mixed oxide $\text{In}_2\text{O}_3\text{-SnO}_2$. However, the world's proven reserves of indium is very limited, and in the next few years the industry will inevitably face its significant deficit. Therefore, the search for new alternative materials with improved performance and processability, as well as economic parameters, is relevant and practically important problem [1-4]. The purpose of this research is to investigate the process of formation and growth of electrochemical-based porous structural alumina in the presence of additives and anisotropic surfactants.

2. Methodology

Nanoporous alumina formed into oxalic electrolyte with addition surface active agents. As additives used anisotropic ammonium salt 3- (4-hexylphenyl) -N, N-dimethyl-3-oksopropanammoniy chloride (A1) and 3- (4-methoxyphenyl) -N, N, -dimethyl-3-oksopropanammoniy chloride (A2) trimetildodetsilammony bromide (A3) and dimetilbenzildodetsilammony bromide (A4) in an amount of 0.1 g/l to 0.7 g/l. Material of cathode was stainless steel.

The samples were a glass substrate with a layer of aluminum 1 micron thick, deposited by vacuum evaporation. The dimensions of substrates was 20 x 10 mm. A layer of chemically resistant lacquer, which separated the direct surface anodizing and place of fixation plates, was deposited on the substrate.

For anodised samples used DC power supply brand MNIPI B5-78/7. To control the temperature values using a magnetic stirrer with heating DRAGONLAB MS7-H550-Pro. Digital registration of chronoamperogrammes made with a multimeter APPA 107. Microphotographs of the surface of anodized aluminum made on AFFRI-DM8 device at magnifications of X400. The morphology of the coatings were examined using a scanning electron microscope JSM-5610 LV chemical analysis system EDX JED-2201 JEOL. Optical transmittance measurements were carried out using a spectrophotometer SF-26.

Chronoamperogrammes of aluminum anodizing are shown in Figure 1.

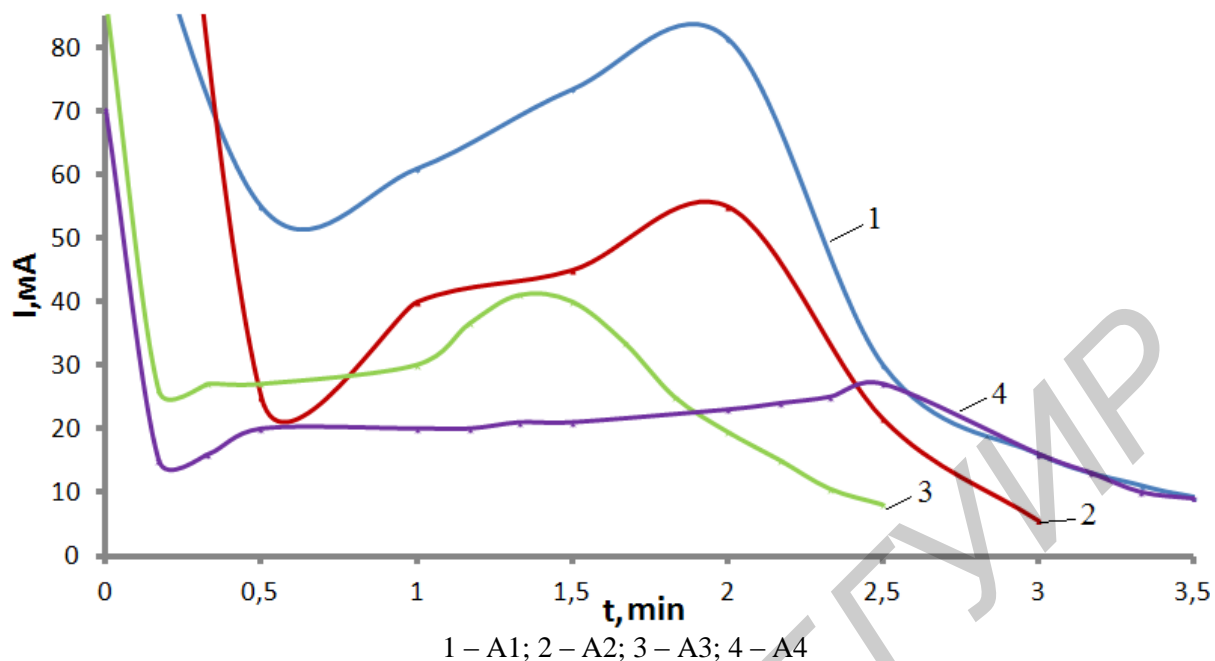


Figure 1. Chronoamperogrammes of anodizing aluminum at $U = 50 \text{ V}$ in a 0.5 M oxalic acid with the addition of additives.

Presented chronoamperogrammes have a classical form. When applying a DC voltage begins the growth of the barrier layer of the anode, creating significant resistance, which leads to a drop in the current start time of anodization.

3. Results

Due to the presence in the electrolyte of oxalic acid happens loosening dense layer and forming a porous nucleation structure, that is shown on an increasing anode current. Further current drop indicate complete transformation aluminum in anodic alumina.

Pore diameter in the investigated electrolytes on the surface of aluminum oxide was in the range of from 0.4 to 0.9 microns. With increasing concentration of additives increase the pore size is observed.

The optically transparent aluminum oxide film with a transmission coefficient of $50\text{-}55\%$ in the wavelength range $450\text{-}650 \text{ nm}$ obtained by introducing additives A1 into the electrolyte. Use A2 reduces transmittance by 10% .

Using A3 and A4 additives allows to obtain alumina having a degree of transmittance of 40 and 35% respectively. It should be noted that increasing the concentration of additives in the electrolyte is in the range of $0.1 - 0.5 \text{ g/l}$ leads to a decrease in the degree of light transmittance alumina with the additive A3 and increase with the additive A4.

Furthermore, when the anodizing of aluminum in solution with 0.3 M oxalic acid at a temperature of from 4 to $20 \text{ }^\circ\text{C}$ and a voltage of $30\text{-}80\text{V}$ for $3 - 420$ minutes under the influence of infrared radiation, we found that the formation of the small pore structure of the film is observed only at elevated temperatures, which is associated with an increase in the chemical reaction of aluminum oxide etch rate when heated.

The research data of the surface microprofile shows that an average of the samples with small pores roughness parameters $R_a = 0,015$ microns, $R_z = 0,0706$ microns, $R_{\text{max}} = 0,1009$ microns. The maximum depth of the pores of 0.03 microns.

The dependence of the optical density of the wavelength for the sample (anodization with 60V , $10 \text{ }^\circ\text{C}$) after the infrared radiation $10\,000 \text{ lm/m}^2$ are shown in Figure 2.

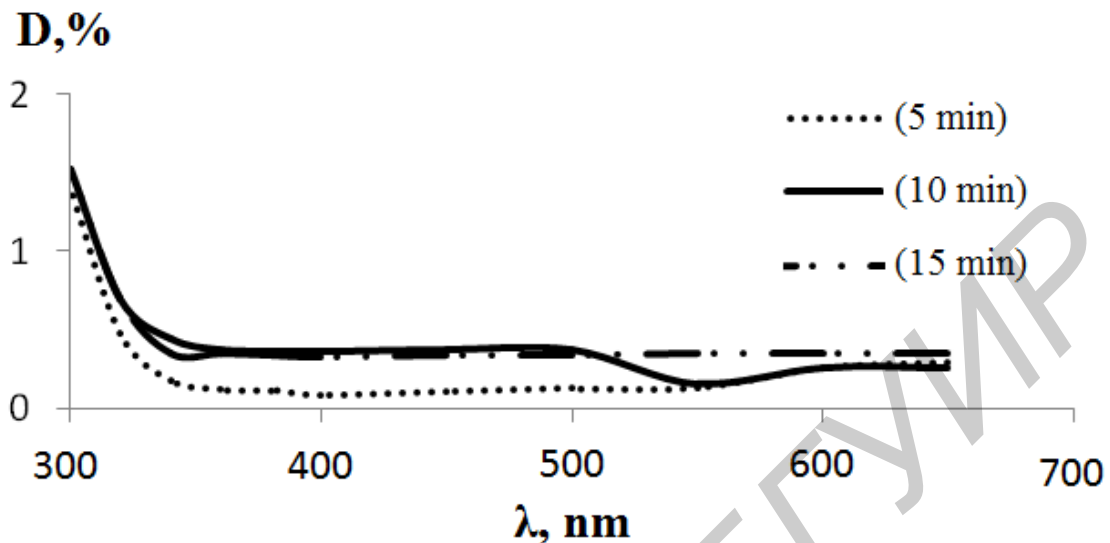


Figure 2. The optical density of the wavelength dependence of the sample anodized aluminum (anodization at 60 V, 10 ° C) after irradiation of infrared 10000 lm/m² for 5, 10 and 15min.

Figure 2 shows that the longer the sample is exposed to infrared emission, the greater its increased absorbance in the visible light spectrum. To restore the ability of transmitting the sample was kept in the dark for 5 minutes and recovered to a normal state. This fact indicates that the flow of the remaining adverse oxidation of aluminum with thermal stimulation.

4. Conclusions

Thus, researches have shown that the use of oxalic acid allows to obtain the most ordered, repeatable and uniform across the square anodized aluminum films. In this case variation of the additives and their concentrations allows you to change the ratio and pore sizes of aluminum films; that transparent nanostructured aluminum layers are obtained by self-organized growth of porous anodic alumina have a roughness parameters $R_a = 0,015$ microns, $R_z = 0,0706$ microns, $R_{max} = 0,1009$ microns and are characterized by high chemical activity in IR heating. These films can be used as a dimming glass IR optics, display technology for various purposes.

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