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Etching technology in formation of self-organized aluminum mesh for sensor and display applications

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Abstract. The paper describes the investigations the temperature and concentration influence on etching of nanoporous alumina in technology of production of self-organized aluminum mesh for sensor and display applications. The pyramidal aluminum meshes was obtained by anodizing of thin film sputtered on sodium glass in oxalic acid solution at voltage of 50 V and then chemical etching of porous alumina in the mixture of phosphoric and chromic acid. It has been shown that the pyramidal morphology of Al meshes can be prepared by chemical etching at temperature of 60°C. This pyramidal morphology of Al mesh can provide good alignment properties and can be one of the promising solutions for production of sensory microsystems and displays without indium-tin oxide films.

1. Introduction

It has been reported that the electrochemical anodization of aluminum on a glass substrate can provide Al meshes with high optical transparency, a low electrical resistivity and good alignment properties for liquid crystal (LC) molecules on the substrate surface. This can be one of the promising solutions for the design and production of next generations of displays and sensors without indium-tin oxides (ITO) [1-4]. The anodization of aluminum is accompanied by the formation of porous alumina layer, which is necessary to remove for production of Al mesh [5].

In the last works [5, 6] for the formation of nanoporous alumina layers on glass substrate, we used the electrochemical anodization of aluminum layers at voltage of 50 V in oxalic acid solution. We founded optimal conditions of the anodization in oxalic acid solution, which allowed preparing nanostructured surfaces with a good optical transparency and conductivity, and good alignment properties of LC molecules on the substrate surface [5-8]. It should be noted that the short time of the anodization did not allow producing good quality transparent nanoporous alumina layers and long-continued time could lead to the formation of the layers with a high electrical resistivity and a low conductivity. Using trade-off between the transparency and conductance of the layers we obtained resistance of 10-20 Ω per square sheet for holes (sphere) with radius of 60-70 nm and interholes distance of 100 nm [7]. Moreover, the transmittance of such Al meshes depends on conditions of etching of anodic aluminum oxide. The research of etching process of anodic aluminum oxide is practically important problem [1-4] of development of technology of formation of self-organized aluminum mesh for sensor and display applications.

In this paper, we report about investigation of the temperature and concentration influence on etching of nanoporous alumina, which has been obtained by anodization in oxalic acid, for the technology of formation of transparent Al meshes.

2. Materials and Methods

Thin Al layers (thickness of 1000 nm) was sputtered on sodium glass by using magnetron spraying. The specimens with dimensions of 1 × 1.5 cm were cut off from sodium glass with thin Al layers and then they were degreased in ethanol. The area of 1.0 cm^2 was selected by insulation of the rest of the sample. An acid resistant paint was used for insulation [5].

Nanoporous alumina layers were made on the surface of aluminum by a simple anodization, which was carried out in 0.3 M oxalic acid at voltage of 50 V and temperature of 10^oC [6]. We used DC power supply brand MNIPI B5-78/7 for anodization. The current drop determinate the duration of anodization. The acid resistant paint was dissolved in acetone after anodization [6].

Oxide films were removed by chemical etching in a mixture of H_3PO_4 and H_2CrO_4. Experiments were performed at different temperatures (40, 60 and 80 °C) and different concentration of H_3PO_4 (0.3, 0.6 M) and H_2CrO_4 (0.2 and 0.4 M) in order to investigate the temperature effect on the conductivity of transparent Al meshes.

The optical transmittance of Al meshes on glass was measured by digital spectrophotometer Metash UV-6000pc in the wavelength range from 300 to 1200 nm. The morphology and cross-sections of the Al meshes were examined by Hitachi S-806 and Zeiss EVO MA 15 scanning electron microscopes.

3. Results and Discussion

For the investigation of etching in mixture of phosphoric and chromic acid were estimated the mass losing under dissolution of layer of the nanoporous alumina (Figure 1) which had been formed by anodization in 0.3M oxalic acid at voltage of 50 V and temperature of 10^oC [6].

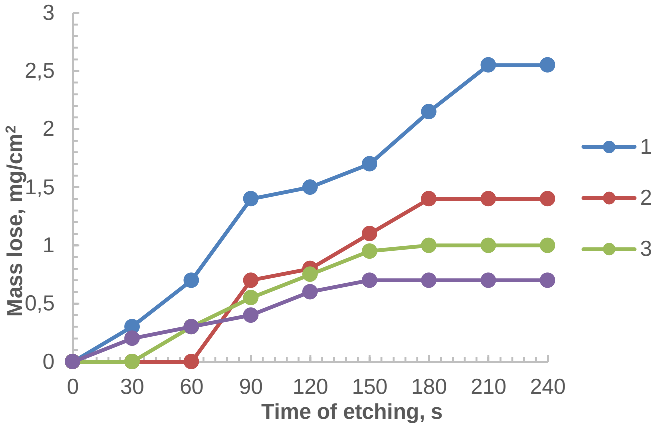


Figure 1. The effect of mass losing during chemical etching of nanoporous alumina (thinness of 1000 nm) at 60^oC in mixture 0.6 M H_3PO_4 and 0.2 M H_2CrO_4 (1), 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 (2), 0.3 M H_3PO_4 and 0.2 M H_2CrO_4 (3), 0.3 M H_3PO_4 and 0.4 M H_2CrO_4 (4).

It can be seen (Figure 1), that the raise of concentration of phosphoric acid from 0.3 to 0.6 M increased the value of mass losing of the nanoporous alumina. It may be connected with local dissolution of aluminum and alumina in the bottom of pore in the 0.6 M phosphoric acid. This fact indicated on the good etching ability of phosphoric acid for alumina and remanding aluminum [6]. Analyzing the etching dependences (Figure 1), it should be noted the decrease of mass losing with raising consecration of chromic acid from 0.2 to 0.4 M. Addition of chromic acid goes on to start the partly passivation of aluminum and to lower of etching rate alumina. The end time of etching was indicated by stop of mass changes (Figure 1). The optimal duration of etching of nanoporous alumina (thinness of 1000 nm) was 150 s in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4. These duration and mixture of etching allowed to safe transparent Al mesh, which had been formed by anodization in 0.3M oxalic acid at voltage of 50 V and temperature of 10°C [6].

The influence of temperature on etching process was exanimated by investigation of transmittance of Al meshes, which were obtained by etching in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 during 150 s (Figure 2).

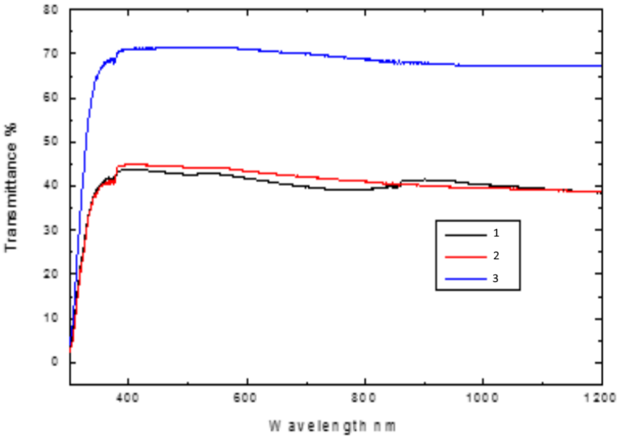
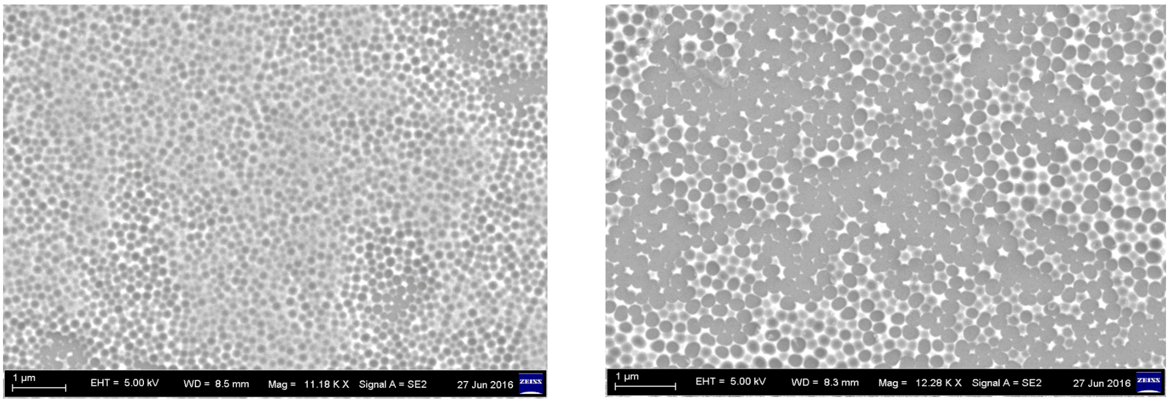


Figure 2. The transmittance spectra of Al nanomeshes obtained by chemical etching in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 during 150 s at temperature of 40°C(1), 60°C(2), 80°C(3).

The transmittance value of Al meshes obtained at 40°C and 60°C is lower on average 30-35% then the same value of Al meshes obtained at 80°C. This tendency can be explained by the enhanced dissolution of aluminum oxide and aluminum in 0.6 M H_3PO_4 and 0.4 M H_2CrO_4. This occurs by the increase of the hole diameter of mesh and to remove aluminum from the glass surface (Figure 3).



a) **b)**

Figure 3. SEM images of transparent Al meshes formed by chemical etching in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 during 150 s at temperature of 60°C(a) and 80°C(2).

Defects in hexagonal arrangement of Al mesh were caused by deference in rate of alumina etching in various regions of surface. It should been noted that the little fluctuation of temperature and concentrating of chemical agents in the process of the chemical etching could significantly disorganise hexagonal mesh of aluminium.

The SEM image of transparent Al meshes after anodization by method [6] and etching in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 during 150 s at temperature of 60°C are shown in Figure 4.

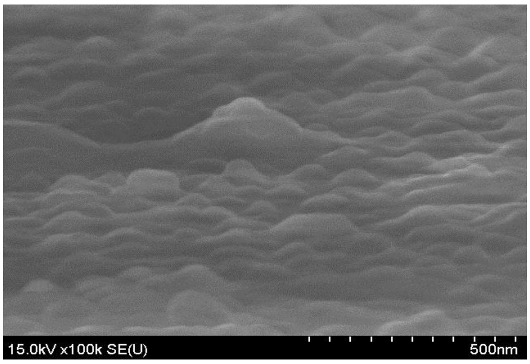


Figure 4. SEM image of transparent Al meshes after anodization in 0.3 M oxalic acid at 50 V and 10°C and chemical etching in mixture of 0.6 M H_3PO_4 and 0.4 M H_2CrO_4 during 150 s at temperature of 60°C.

The morphology of Al mesh was formed by pyramidal structures (Figure 4), which were organized in hexagonal arrangement onto glass surface (Figure 3 a). The morphology of Al mesh allows developing technique of synthesis of nanostructured functional films with changeable phase composition for chemosensitive layers of new gas sensors. This morphology of Al mesh can provide good alignment properties for LC molecules on the substrate surface and can be one of the promising solutions for the design and production of next generations of displays without indium-tin oxide films [8].

4. Conclusions

Thus, it has been shown that the nanoporous alumina can be used for the production of Al mesh with nanosize pyramidal structures by using chemical etching in the mixture of phosphoric and chromic acid.

The defects in hexagonal arrangement of Al mesh were caused by the little fluctuation of temperature and concentrating of chemical agents in the process of the chemical etching. It should been noted that the increase temperature up to 80°C could to remove aluminum from the glass surface.

These optically transparent Al meshes with good alignment properties for LC molecules can provide creation of new transparent electrodes for display devices without using ITO technology. The morphology of Al mesh allows forming new nanostructured films with changeable phase composition for promising gas sensors and sensory microsystems.

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