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STRUCTURAL AND FLUORESCENCE STUDIES OF POLYCRYSTALLINE A-AL_2O_3 OBTAINED ON ANODIC ALUMINA

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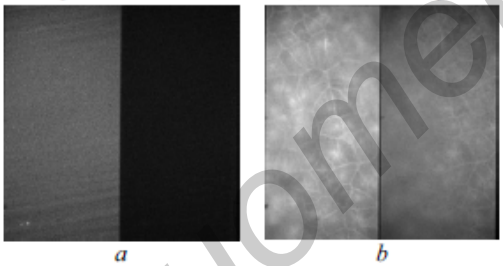
As is known, α-Al_2O_3 membranes due to their biocompability and high chemical resistance are of great demand [1]. Previous studies dealt with the investigation of physical chemical properties of anodic alumina. However, the formation of robust α-Al_2O_3 films from free-standing anodic alumina films and membranes remains quite a challenge [1]. In the present study we obtained robust α-Al_2O_3 films (144 μm thick) films by the heat treatment at 1200–1400 °C of free-standing anodic alumina and studied their physical chemical and fluorescence properties. The samples were obtained by double-sided anodizing of aluminum specimens in 2.0 M aqueous solution of sulfuric acid at constant current density of 32 A m^{-2} and temperature of (10.0 ± 0.1) °C up to the moment when aluminum was completely oxidized. At the steady state of oxide growth the voltage was at about 20 V, the anodizing time was of about 1.5 h. If indicated, the films were heat treated in the air at temperature range of 200–1400 °C.

According to differential scanning calorimetry data accomplished by *X*-ray analysis, the first step of crystallization occurs at around 944 °C, producing γ-Al_2O_3. The second one takes place at around 1236 °C, which corresponds to the formation of α-Al_2O_3. According to scanning electron microscopy analysis, the as-anodized anodic alumina films possess well-ordered porous structure with the pore diameter of 10.2 nm. After heat treatment at 1400 °C the samples lose their porous structure and certain crystallites with average size of 2.6 mm can be observed. In porous anodic alumina the surface structure transformation during high temperature treatment is caused by the reorganization of oxygen and aluminum sub-lattice and removal of impurities incorporated during anodizing. The changes in the surface structure of the films can be also observed by fluorescence microscope (Figure). The as-anodized film has uniform emission throughout the surface. At the same time for 1400 °C-treated film mainly emit only along the grain boundaries. It can be explained by the concentration of the defects of different type at grain boundaries of α-Al_2O_3. According to steady-state fluorescence measurements both the as-anodized and heat treated at 1400 °C films are characterized by a broad fluorescence in the wavelength range of 400–650 nm. The time-resolved fluorescence data show that with increasing treatment temperature the average life time of the centers increases from 3.5 to 4.5 ns. It is caused by the refining of alumina structure and formation of α-Al_2O_3. The origin of the fluorescence is believed to be due to the presence of hydroxyl groups.



a *b*

Figure. The surface appearance of the as-anodized (a) and heat treated at 1400 °C (b) sulfuric acid anodic alumina films observed by the fluorescence microscope in (430-480 nm)/(485-630 nm) diapason under 405 nm excitation.

References

1. T. Masuda, H. Asoh, S. Haraguchi, S. Ono. Materials 2015, 8, 1350–1368.