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Thermal Stability of Anodic Layers on Aluminum Alloys

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Abstract. The thermal stability of anodic alumina layers made by the open circuit potential measurements and electron microscopy is discussed. The crack growth resistance of the anodic alumina layers has been studied depending on the initial aluminum alloy composition and the anodization regimes.

Introduction

Anodized aluminum alloys are widely used in microelectronics for the manufacturing switching plates, multiterminal VLSI packages, multichip modules, various sensors, etc. [1]. The thermal stability of anodic alumina is an essential characteristic to use this material in various applications. Copper, magnesium, iron, and manganese are in the aluminum alloys as intermetallic compounds of type $CuAl_2$, $FeAl_3$, Al-Fe-Si, etc. At the alloy anodization, these compounds are dissolved more quickly than aluminum. So, the anodic alumina layers formed from aluminum alloys are more porous than these formed from pure aluminum. On the other hand, thermal stability of material is strongly dependent on its macro- and microstructure. Material with the porous structure, as a rule, is more thermally stable [2]. Taking aluminum anodic oxide to have mechanical properties of conventional bulk Al_2O_3, the growth of internal stress in the alumina layer can result in the alumina brittle fracture. In this case the Gryffitz's theory is applicable to the oxide layer. In accordance with this theory, the brittle fracture begins from the origin of microcracks. A crack will propagate when the reduction in potential energy that occurs due to the crack growth is greater than or equal to the increase in surface energy due to the creation of new free surfaces. The critical stress required for crack propagation in a brittle material depends on the modulus of elasticity, specific surface energy and half the length of an internal crack [3]. Cases of the internal stress growth with the increase of a dense oxide thickness resulting in the formation of microcracks at room temperature are known [4-7]. So, it is of importance to determine conditions at which stress in the anodic alumina becomes critical resulting in the crack generation to specify regimes of the anodization and thermal treatments preventing the anodic alumina layer from these microcracks.

Experimental

The surface condition (presence of microcracks) can be evaluated by an electric potential value. Defects in the layer correlate with values of stress and surface energy; latter is characterized by the electric potential. The methods based on the potential measurement are widely applied to study the surface solid state, research the oxidation mechanism, and determine antifriction properties of materials [8]. Nevertheless, the definition method for the microcracks nucleation discussed is of scientific and practical importance.

The method is based on the measurement of the aluminum electrode open circuit potential, which depends on the alumina layer thickness and oxide structure imperfection, i.e. the presence of defects such as microcracks. With the appearance of microcracks this potential becomes more negative [8].

Measurements of the open circuit potential were made using the device shown in Fig. 1. In our experiments we used an electrolyte based on the 1% aqueous solution of the citric acid. Voltmeter with the $\geq$ 100 MOhm input resistance was used for the measurements.