



Features of Porous Anodic Alumina Growth in Galvanostatic Regime in Selenic Acid Based Electrolyte



Y. Nazarkina^{a,*}, K. Kamnev^a, A. Dronov^a, A. Dudin^b, A. Pavlov^b, S. Gavrilov^a

^a National Research University of Electronic Technology (MIET), Bld. 1, Shokin Square, Zelenograd, Moscow, 124498, Russian Federation

^b Establishment of the Russian Academy of Sciences, Institute of Nanotechnology Microelectronics INME of RAS, Leninskiy Prospekt 32A, Moscow, 119991, Russian Federation

ARTICLE INFO

Article history:

Received 1 September 2016

Received in revised form 7 February 2017

Accepted 9 February 2017

Available online 11 February 2017

Keywords:

porous alumina
selenic acid
self-ordering
morphology

ABSTRACT

The features of porous anodic Al₂O₃ formation process in galvanostatic regime in selenic acid based electrolyte under a range of anodization conditions were investigated. The growth process kinetics of porous anodic Al₂O₃ was characterized by chronopotentiometric curves. The effect of electrolyte concentration, current density and anodization duration on the oxide porous structure ordering was studied by scanning electron microscopy. Approximate regions of self-ordered porous Al₂O₃ formation process conditions were estimated. It was found that anodization parameters, at which self-ordered porous structure formation is observed, correspond to the conditions of equilibrium of oxidation and etching rates. It is shown that deviations from these conditions lead to the disordering of the porous structure.

© 2017 Elsevier Ltd. All rights reserved.

1. Introduction

Porous anodic aluminum oxide (AAO) is a material that widely used as a matrix for the formation and investigation of nanostructures [1–5]. In particular, AAO is a useful matrix for spectroscopic investigations of nanoobjects due to its unique geometrical and optical properties. Commonly AAO is formed in three main electrolytes (oxalic, sulfuric and phosphoric acid solutions). However, for particular applications, there is a need in new electrolytes which allow expanding the range of porous alumina matrix properties.

Recently, porous anodic alumina films formation in the selenic acid electrolyte was reported [9–11]. The cell size of the AAO produced in selenic acid based electrolyte is similar to the cell diameter of AAO obtained in oxalic acid solutions. At the same time, its pore diameter is smaller than in the AAO formed in oxalic acid and it is similar to the pore diameter of the AAO formed in sulphuric acid. Thus, on the one hand, the AAO formed in selenic acid can be used as a substitute for the AAO formed in oxalic acid. Porous anodic alumina formed in oxalic or other carboxylic acids is fluorescent [6,7]. This circumstance can complicate AAO application as a matrix for spectroscopic investigations [8], for example, by Raman spectroscopy. In contrast to the AAO synthesized in

oxalic and phosphoric electrolytes, the porous film formed in selenic acid based electrolyte does not exhibit fluorescence background in Raman spectra [12]. Thus it can be used as a matrix for the study of embedded nanoobjects by spectroscopy techniques. On the other hand, the fact that porous alumina formed in selenic acid has small pores and relatively big cell size is notably interesting for its application as a matrix for metamaterials production. For example, in paper [13], an AAO matrix with the porosity of around 3.5% was obtained via Al anodization in the selenic acid aqueous solution. This porosity value is much lower than the one achievable by means of sulphuric or oxalic acid [14,15]. This unique porous alumina structure, determining nanorod concentration, has enabled the metamaterials' plasma frequency to be tuned throughout the visible and near-infrared spectral ranges [13].

Up to date, several papers concerning aluminum anodization in selenic acid electrolyte have been published. In our recent paper [12], the effect of electrolyte concentration on porous Al₂O₃ formation in galvanostatic regime at the current density of 5 mA cm⁻² was considered. It was shown that the best pore arrangement within the investigated range of experimental conditions had been obtained for 0.3–0.5 M H₂SeO₄. Anodization in the solutions with lower concentrations at 5 mA cm⁻² leads to the defective porous structure formation. However, it could be useful to find the anodization parameters at which AAO can be produced even in low concentrated H₂SeO₄. It is also interesting to

* Corresponding author. Tel.: +7 926 766 50 90.

E-mail addresses: engvel@mail.ru, yulia.nazarkina@org.miet.ru (Y. Nazarkina).

have a possibility to produce AAO at higher current densities, for example, to increase the oxide formation rate.

In the paper [11], porous structure self-ordering under various electrolyte concentrations and applied voltages was investigated. It was reported that porous structure ordering increases with the electrolyte concentration and applied voltage. However, this result could be explained either by optimal electric field and concentration values adjustment or by higher charge values passed through the anode and, consequently, the thicker oxide layer formed. Indeed, in paper [16], the number of defects in porous structure is shown to reduce with the anodization time increase. So it is still a question: is it necessary to use high concentrated solutions and high applied voltages or it is possible to produce ordered AAO under other anodization parameters, for instance, by the longer anodization process.

The investigations mentioned above were carried out in the potentiostatic regime. The galvanostatic regime also has some advantages, for example, easier control of Al consumption and Al_2O_3 layer thickness that is more practical for industrial applications. However, the mechanisms of pore initiation and pore ordering have its specific features for galvanostatic and potentiostatic regimes [17]. As the pore and cell diameters depend on the applied field, these geometrical parameters tend to be constant while anodization is performed in potentiostatic regime [18]. In this way, relatively regular porous films can be obtained even under the anodization parameters that are not optimal for ordered porous films formation by long anodization process at constant voltage [19]. Contrarily, in the galvanostatic regime, the value of the applied voltage floats depending on the correlation between aluminum oxidation and alumina etching rates. Due to

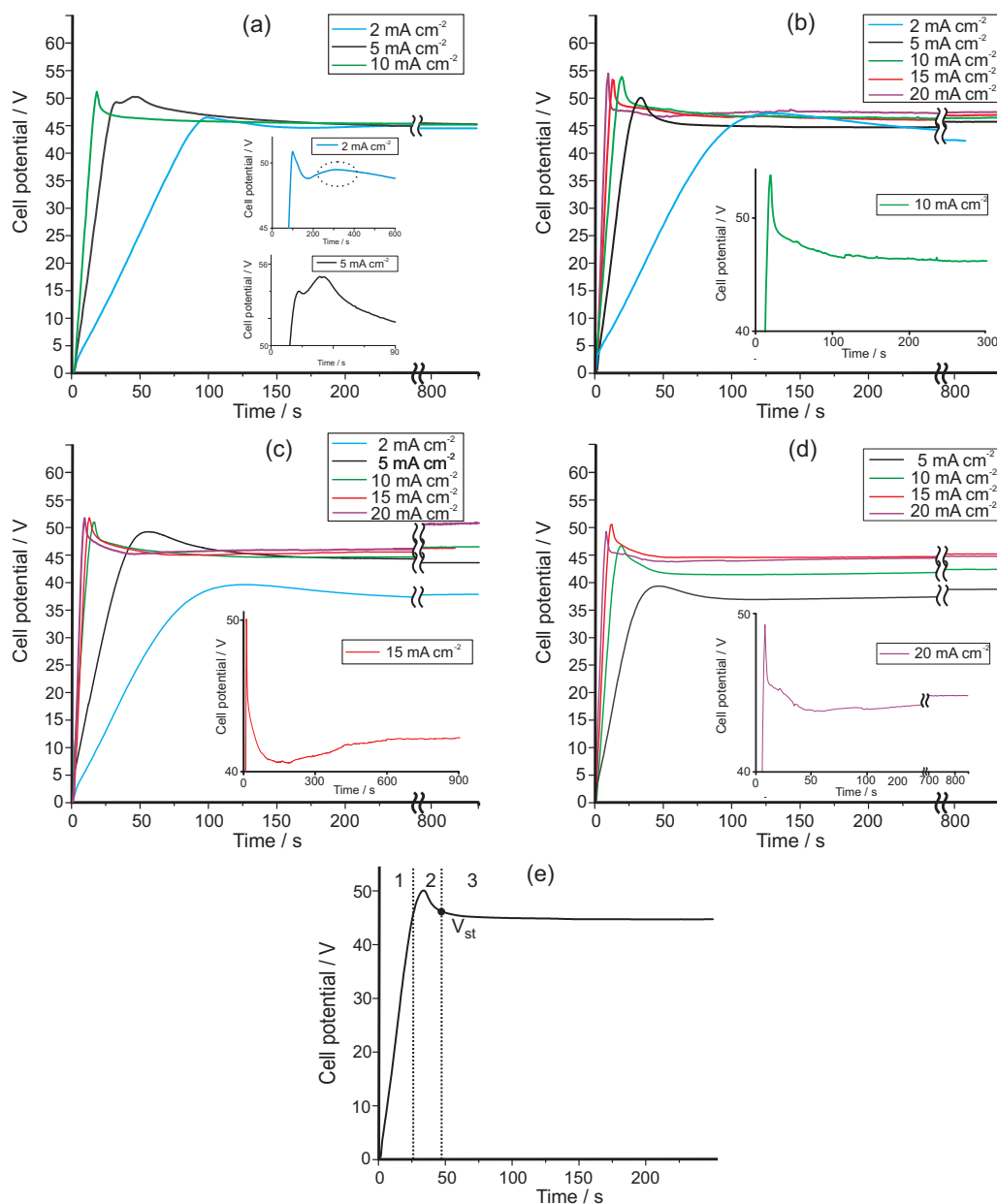


Fig. 1. Voltage-time behavior during anodic anodization at different current densities. Selenic acid electrolyte concentrations: 1 wt% (a), 5 wt% (b), 10 wt% (c) and 15 wt% (d). Schematic plot of the Al anodization stages, V_{st} – steady-state voltage (e). Top inset in the section (a): the appearance of the bulge at the beginning of the steady-state stage. Other insets represent the V-t curves fragments with features attributed to “burning”. The choice of the curve corresponds to the critical current density value, at which “burning” starts to appear.

Download English Version:

<https://daneshyari.com/en/article/6472015>

Download Persian Version:

<https://daneshyari.com/article/6472015>

[Daneshyari.com](https://daneshyari.com)