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Influence of substrate preparation on the shaping of the topography of the surface of nanoceramic oxide layers

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ABSTRACT

The paper discusses the shaping mechanism and changes occurring in the structure and topography of the surface of nanoceramic oxide layers during their formation. The paper presents the influence of substrate preparation on the surface topography of oxide layers. The layers were produced via hard anodizing on the EN AW-5251 aluminum alloy. The layers obtained were subjected to microscope examinations, image and chemical composition analyses, and stereometric examinations. Heredity of substrate properties in the topography of the surface of nanoceramic oxide layers formed as a result of electrochemical oxidation has been shown.

1. Introduction

Oxide layers produced through hard anodizing belong to materials with a strongly developed surface and their properties can vary widely, and depend mostly on the conditions in which they are fabricated [1–4]. The selection of the method of protecting the surface of aluminum and its alloys depends mainly on the purpose of nanoceramic layers. Electrochemical oxidation of aluminum and its alloys is one of the most optimal and frequently used aluminum surface protection processes. Aluminum oxide layers produced through electrochemical methods are characterized by a specific amorphous and columnar structure [5–8]. With their very good adhesion to the substrate [9–12] layers with such a structure not only effectively protect the surface of aluminum from corrosion [13–16], but can also be used in a number of technology fields. Different base materials, types and concentrations of electrolytes, current and temperature conditions and varied process duration are applied, depending on the anticipated properties of oxide layers. These parameters directly influence surface morphology and the structure, thickness and microhardness of layers [17–20]. In the case of tribological applications, it is very important to obtain an oxide layer with low values of surface roughness [21–23]. In studies of a number of authors, oxide layers are characterized by significant surface irregularities, which may to a large degree affect the considerable wear of the sliding partner in the first stage of interaction. In order to mitigate or eliminate the above-mentioned defect, the authors of the paper examined the formation mechanism and the influence of the substrate preparation on the shaping of the topography of nanoceramic oxide layers.

2. Material and methods

The object of the study comprised nanoceramic surface layers produced via hard anodizing on the AW-5251 aluminum alloy (Al 96.93%, Fe 0.447%, Si 0.318%, Cu 0.087%, Mg 1.798%, Mn 0.311%, Cr 0.038%, Ti 0.021%, Zn 0.05%). The selection of the alloy was dictated by its ease of oxidation and good results of previous studies where it was used. Plates of the alloy with the surface area of 0.05 dm^2 (0.1 dm 0.5 dm) were cut out with a water jet from a 1-mm-thick rolled sheet. The cathode consisted of a lead electrode with the same surface area as the aluminum alloy samples. Anodizing was conducted by means of a direct-current method, using a GPR-25H30D stabilized feeder at constant current density of 3 A/dm^2. The electrolyte was an aqueous acid solution: 33 ml/l H_2SO_4, 30 g/l $C_2H_2O_4{\cdot}2H_2O$, 76 g/l $C_8H_6O_4$, with a temperature of 303 K. Anodizing was performed in a cylindrical electrolyser with a diameter ϕ of 180 mm. During anodizing, the electrolyte was stirred with a mechanical stirrer at a rotational speed of 50 rpm. Before anodizing, samples produced in order to examine the formation and growth of layers were initially etched in a 5% KOH solution for

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Table 1
Conditions of oxide layers production carried out for the purpose of examining the shaping mechanism.

Sample	Etch	Current density	Temperature	Process time
A	KOH 40 minutes, HNO$_3$ 10 minutes	3 A/dm^2	303 K	Punkture voltage
B				Start of proper oxidation
C				5 minutes
D				10 minutes
E				60 minutes

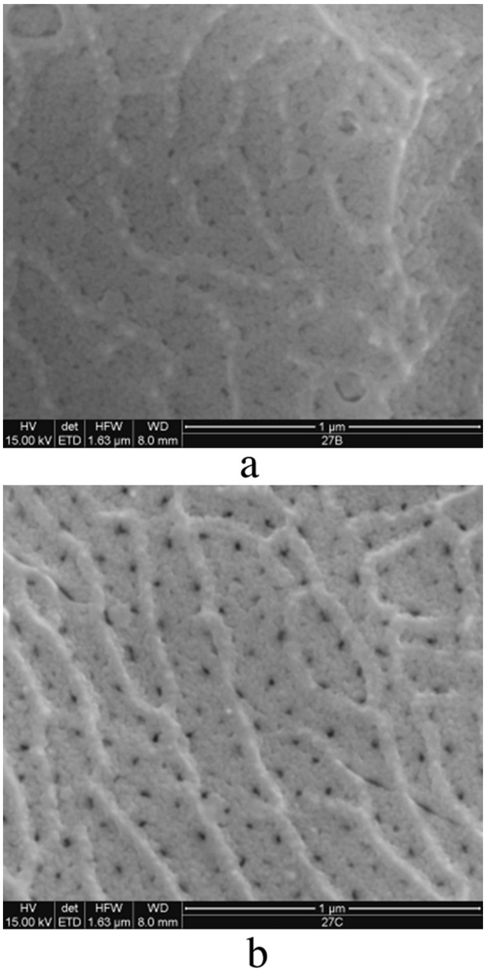

Fig. 1. Images of a nanoceramic surface layer acquired using a scanning microscope: (a) after 14 s, and (b) after 22 s from the beginning of the hard anodizing process.

40 min and then in a 10% HNO$_3$ solution for 10 min. The anodizing time of individual layers was determined based on the relationship between the changes in anodizing voltage and the duration of the formation of layers, and comprised various stages of layer formation (Table 1).

In the case of samples produced in order to examine the influence of the substrate preparation on the topography of the surface of layers (Table 2), the anodizing time was 60 min. The layers were produced without initial etching (rinsing in ethyl alcohol) of the surface of the EN AW-5251 alloy, and with initial etching for 40 and 150 min.

After anodizing, all the oxide layers were rinsed for 1 h in distilled water with the aim of rinsing out the electrolyte from the aluminum oxide pores. Surfaces of the same aluminum alloy rinsed in spirit and etched for 40 and 150 min in KOH and HNO$_3$ solutions were also examined.

The surface of layers was examined with a Philips XL30 scanning electron microscope, at magnifications of 1000 / 100,000*x*, and a MultiMode atomic force microscope with NanoScope 3D. Because aluminum oxide does not allow carrying away electrons knocked off during the interaction of a microscope beam with a sample, the layers were sputtered with Ag–Pd. The sputtering was performed with a PECS 682 device of the Gatan company in current

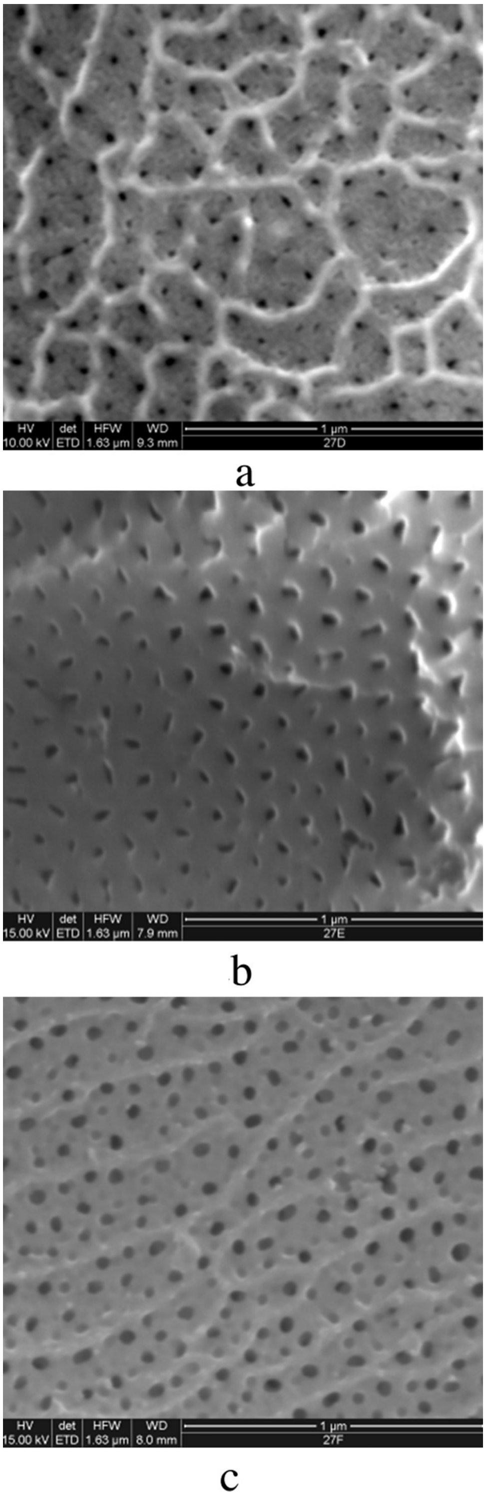

Fig. 2. Images of a nanoceramic surface layer acquired using a scanning microscope: (a) after 5 s, (b) after 10 s, and (c) after 60 min from the beginning of the hard anodizing process.