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Micro- and nano-formations on the surface of plasma electrolytic oxide coatings on aluminum and titanium

V.S. Rudnev*

Institute of Chemistry, Far Eastern Branch, Russian Academy of Sciences, 159, Prosp. 100-letya Vladivostoka, Vladivostok 690022, Russia
Far East Federal University, 8, Sukhanova Str., Vladivostok 690950, Russia

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ABSTRACT

In the majority of cases electric spark and arc discharges developed in condensed media result in the formation of micro- or nanosized particles. Micro- or nano-formations of different types are always present on the surface of oxide coatings formed under effect of electric spark and arc discharges (the approach is referred to as the method of plasma electrolytic oxidation (PEO) or microarc oxidation (MAO)). These formations (particles) can be extremely active in physical–chemical terms and determine the properties of a system at large. Nevertheless, the structure, composition, and mechanism of formation of the above micro- and nanostructures and their control on the surface of PEO-coatings of different chemical composition as well as their effect on the functional properties of the systems obtained using the PEO-method have not yet been studied in sufficient detail. The present paper is devoted to the examination of micro- and nano-formations on the surface of PEO-coatings: their shape, composition, and effect on the coatings properties. The objective of the paper was to generalize our earlier obtained data on micro- and nano-formations on the surface of PEO-coating on aluminum and titanium and emphasize the importance of the poorly developed field of knowledge related to PEO-coatings, their structure and formation mechanisms, and the possibilities of controlling their composition and functional properties.

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1. Introduction

Recently, the method of plasma electrolytic oxidation (PEO) consisting in metal anodization in electrolytes under voltages of spark and arc electric discharges has been successfully applied in the formation of multicomponent oxide coatings of various functional purposes on the surface of metals and alloys [1–17]. In the literature the method is also referred to as microarc or microplasma oxidation or deposition (MAO). Through involving the electrolyte components into the process of anodic layers growth and initiating high-temperature processes, electric breakdowns result in the formation of multicomponent oxide coatings of certain, depending on the formation conditions (electrolyte composition and state, treatment duration, current value and form etc.), structure and chemical composition. The systems of the above type include coatings with calcium phosphate on aluminum and titanium for biomedical purposes [7,8], coatings with dispersed iron particles absorbing the super high frequency radiation of a specific wavelength range [9], coatings with ferromagnetic properties containing iron and/or nickel compounds [10], catalytically active layers with nickel, copper,

molybdenum, and tungsten oxides doped with platinum [11–13], hybrid polymer- or graphite-containing protective surface oxide structures [14,15], coatings increasing the heat emission (infrared radiation emission upon heating) of titanium alloys [16,17] etc.

It is generally known that electric spark or arc discharges in condensed media yield the formation of micro- or nanosized particles. Such particles could be very active in physical–chemical terms and determine the properties of a system at large. This is why the functional properties of complex surface oxide PEO-systems on metals formed under electric discharge conditions and containing, aside from the treated metal oxides, oxides and other compounds of different metals and nonmetals depend on many factors, in particular, organization of the coating surface and presence of characteristic formations (for instance, micro- and nanosized particles and crystals) caused by the process features on this surface as well as their structure, composition, location, and concentration. However, the structure, composition, mechanisms of micro- and nano-formations emergence on the surface of PEO-coatings of different chemical compositions, and their effect on the functional properties of metal/oxide layer PEO-systems still remain studied insufficiently.

The objective of the present work was to generalize our earlier obtained data on micro- and nano-formations on the surface of plasma electrolytic oxide coatings on aluminum and titanium, i.e., emphasize the importance of the poorly developed field of knowledge related to PEO-coatings, their structure and formation mechanisms, and the possibilities of controlling their composition and functional properties.

* Institute of Chemistry, Far Eastern Branch, Russian Academy of Sciences, 159, Prosp. 100-letya Vladivostoka, Vladivostok 690022, Russia.

E-mail address: rudnevsv@ich.dvo.ru.

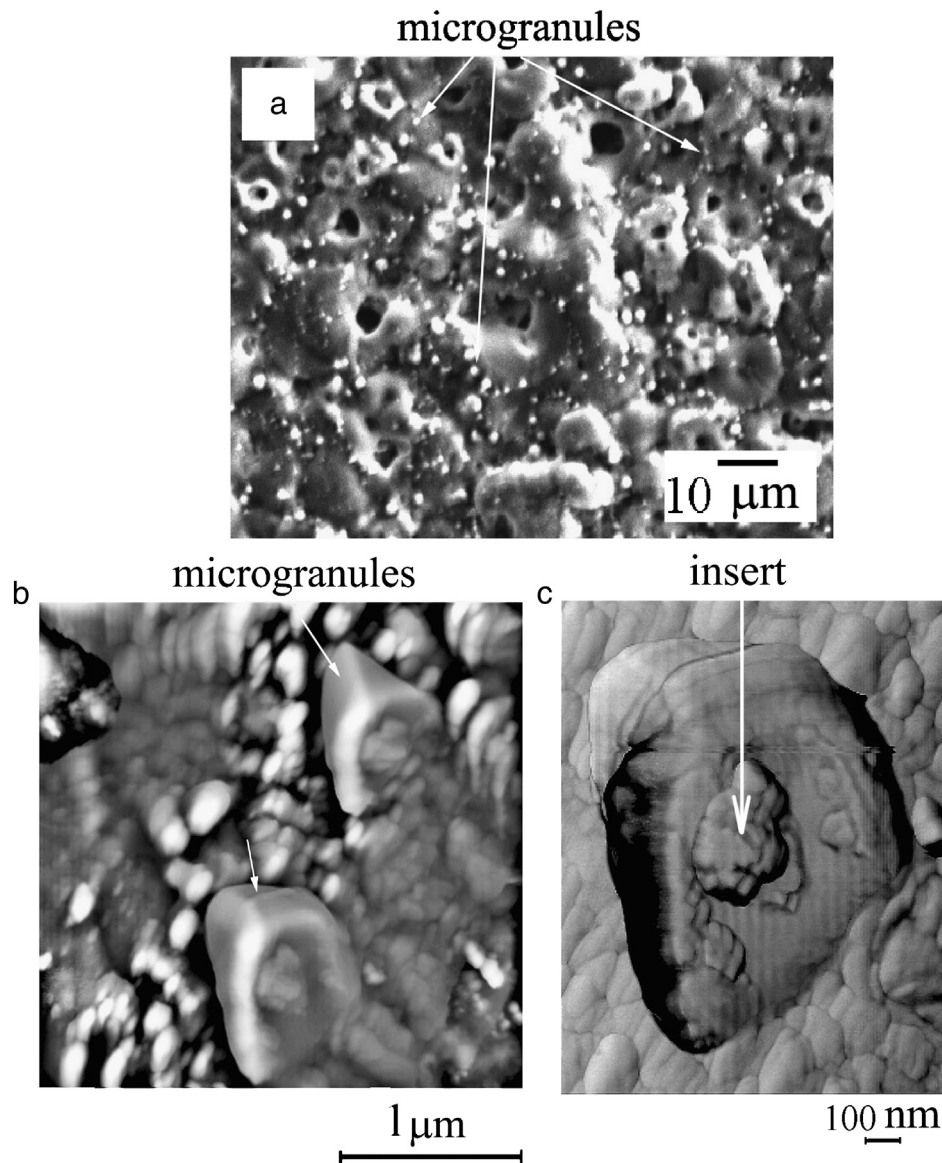


Fig. 1. a) SEM-images (upper view) of the surface of layers formed on the aluminum alloy AMg5 at anodic polarization in the electrolyte. AFM-images of microgranules: b) – amplitude representation, c) – phase representation. SEM – scanning electron microscope, AFM – atomic force microscope.

2. Results

2.1. Microgranules

Dispersed particles of regular or random geometric shapes (microgranules) are often observed on the surface of anodic coatings formed on aluminum and titanium by means of the PEO method [18].

We investigated the composition and organization of microgranules on the surface of coatings formed on aluminum alloy by the PEO method in an aqueous electrolyte $\text{Na}_3\text{PO}_4 + \text{Na}_2\text{B}_4\text{O}_7 + \text{Na}_2\text{WO}_4$ (hereinafter referred to as PBW [19,20]) with additives of nickel and/or copper acetates (respective electrolytes are marked as PBW_{Ni}, PBW_{Cu}, PBW_{NiCu}) that are catalytically active in oxidation of CO into CO₂ [21,22]. The anodic PEO-coatings were formed using the pulse unipolar current for 10 min at an effective current density of $i = 5\text{--}10 \text{ A/cm}^2$.

As seen from the electron microscopy images, the layers surfaces contain dispersed particles (microgranules) scattered with certain regularity (Fig. 1a). They are often located as chains over the perimeter of a contact of the type ‘elevation with a hole at the top/groove’ (Fig. 1a), i.e., in accordance with some order around the channels of electric breakdowns

(pores). The latter indicates to the correlation between their emergence and location with electric breakdowns in the near-anode area.

Fig. 1b and c shows the images of microgranules obtained using the atomic force microscope. The characteristic microgranule sizes are $\sim 1 \mu\text{m}$. As seen from the figures, in the case of coatings formed in the PBW_{Ni} electrolyte, these microgranules comprise vertically standing or lying on the side regular polyhedral tubes narrowing to the bottom. In the wide part, the tube contains an “insert” of a size of $\sim 0.1\text{--}0.4 \mu\text{m}$ (Fig. 1): the wall thickness is $\sim 0.1\text{--}0.3 \mu\text{m}$, whereas the microgranule length is $\sim 1 \mu\text{m}$. The microgranule walls are built from blocks of sizes of dozens nm.

According to the microprobe X-ray spectral analysis, for the coatings formed in the PBW_{Ni} electrolyte, the average (from 5 measurements) coatings and microgranules compositions can be presented as follows (at.%):

	C	O	Mg	Al	W	P	Ni
Surface	17.5	58.3	0.9	23.1	0.07	–	0.2
Microgranule	30.2	47.6	0.8	14.9	1.4	0.4	4.1

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