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Polytetrafluoroethylene-oxide coatings on aluminum alloys

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ABSTRACT

Introduction of polytetrafluoroethylene (PTFE) in the oxide layers can significantly improve their practically important properties such as hydrophobic, anti-friction, and anticorrosion. The present work was devoted to the investigation of regularities of plasma electrolytic formation of PTFE-oxide coatings on aluminum in aqueous silicate alkaline electrolyte containing dispersed particles of polytetrafluoroethylene stabilized by siloxane-acrylate emulsion and the examination of their composition, structure, and properties. The coatings obtained have a structure, which is not a conventional one for PEO layers. The surface of the coating consists mainly of the products of decomposition of PTFE and siloxane-acrylate copolymer and additionally contains polytetrafluoroethylene particles. The conditions of fabricating wear-resistant and hydrophobic PTFE-oxide coatings have been established. The water contact angle of the coatings is larger than 90°. Such coatings can be recommended for application for the parts operating in friction units and fabrication of anti-icing coatings.

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1. Introduction

An intensive interest of researchers is concerned with composite (hybrid) coatings fabricated by the plasma electrolytic oxidation (PEO) method and containing, aside from an inorganic component (in particular, an oxide one), compounds of the nature similar to that of organic ones, such as graphite, polymers, and carbon nanotubes and nanofibers [1–15]. Polytetrafluoroethylene ($[-C_2F_4-]_n$, hereinafter referred to as PTFE) is a representative of the class of polymers characterized with high chemical inertness and good hydrophobic and antifriction properties [16]. Introduction of PTFE into oxide coatings formed using the method of plasma electrolytic oxidation (PEO) can significantly improve the level of their practical properties, including hydrophobic, antifriction, anti-scoring, wear-resistant, anticorrosion, dielectric, biocompatible etc.

The available literature sources describe a number of methods of formation of PTFE-oxide coatings on valve group metals [2,12–15]. The two-stage methods must have been studied the most thoroughly. In such cases, PTFE is deposited on the surface of the preliminarily formed PEO coating whether by mechanical rubbing or by impregnation in respective emulsions, often with subsequent thermal treatment [2,13].

It is evident that single-stage methods, i.e., formation of PTFE-oxide coatings directly by the PEO method are preferable in technological terms. For this purpose, the base electrolyte is added with dispersed PTFE particles, which can be embedded into the growing oxide coating

under effect of electric spark or microarc discharges [14,15]. In this case, an important and not yet solved problem consists in stabilization of dispersed PTFE particles in the bulk of an aqueous electrolyte. The researchers' attempts to solve it are related to addition of surfactants into the solutions [14,15].

For instance, the authors of [14] formed PTFE-oxide coatings on magnesium using the PEO method in an aqueous alkaline phosphate electrolyte containing 10 wt.% of polytetrafluoroethylene nanoparticles and sodium dodecylsulfate. To prevent exfoliation of the suspension electrolyte, it was constantly stirred. According to the X-ray diffraction analysis and X-ray photoelectron spectroscopy data, PTFE particles become embedded into the oxide coating. On the other hand, electrolytes containing PTFE nanoparticles and stabilized by anionic surfactants are insufficiently stable over time.

To stabilize dispersed PTFE particles in an aqueous alkaline electrolyte, the authors of [15] used an aqueous acrylate emulsion. The X-ray photoelectron spectra of the obtained coatings contain an F1 s peak with the bond energy equal to 690.0 eV corresponding to the CF_2 group, i.e., PTFE particles from the electrolyte become embedded into the oxide coating. This fact is also corroborated by the decrease of the friction coefficient and the increase of the coatings anticorrosion properties. In other words, application of the acrylate emulsion must be a rather efficient tool to stabilize dispersed PTFE particles in the electrolyte and to fabricate PTFE-oxide coatings.

In our opinion, application of the siloxane-acrylate emulsion can be even more efficient for stabilization of dispersed PTFE particles in aqueous alkaline electrolytes (see Fig. 1). Micelles of siloxane-acrylate polymers could be attached on the PTFE particle surface as a result of hydrophobic (intermolecular) interactions with the copolymer siloxane

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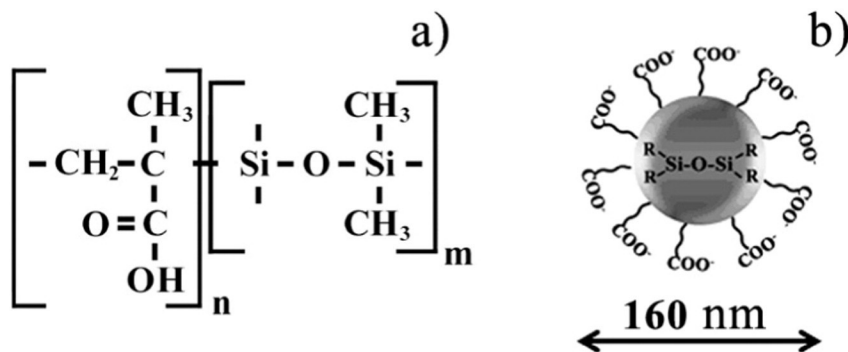


Fig. 1. Structural formula of siloxane-acrylate copolymer (a) and structure of the micelle of emulsion in water (b).

part (Fig. 2), thus providing the affinity to water and imparting a negative electric charge due to the presence of carboxylic groups in the copolymer acrylic part.

The objective of the present work was to determine the composition and main physical-chemical characteristics of PEO formation of PTFE-oxide coatings on aluminum in aqueous electrolytes containing dispersed polytetrafluoroethylene and siloxane-acrylate emulsion and to study their structure and properties.

2. Materials and methods

2.1. Materials

Composite layers were formed on aluminum alloys samples (wt.%: 0.5 Fe; 0.5 Si; 4.8–5.8 Mg; 0.5–0.8 Mn; 0.1 Cu; 0.2 Zn, the remaining part Al) of a size of $20 \times 20 \times 0.5$ mm. The samples preparation included their mechanical grinding with subsequent chemical polishing in a mixture of acids (H_3PO_4 : H_2SO_4 : $\text{HNO}_3 = 4: 2: 1$) at 90–100 °C.

To prepare the base electrolyte, distilled water and commercial reagents of the chemically pure grade were used: 10.6 g/l $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$ and 2 g/l NaOH.

2.2. Formation of PEO-coatings

The electrochemical treatment of aluminum samples was carried out in the electrolyte inside a vessel made of thermal-resistant glass. A hollow coil made of nickel alloy, thorough which a cold tap water was fed, served as a counter-electrode. The solution temperature during the treatment did not exceed 30 °C. A PC-controlled TER-4/460N thyristor device (Russia) operated in the unipolar mode served as a current source. Anodizing of aluminum samples was carried out at a constant effective current density of 5 A/dm² for 20 min. The voltage on the

electrodes at the completion of processing was from 520 to 570 V. The electrolyte was stirred using a mechanical stirrer. The samples with coatings were washed in distilled water and dried in air.

2.3. Determination of coatings thickness and composition

The film thickness was measured using a VT-201 eddy current thickness meter (Russia). The device allows one to measure the thickness of the dielectric coatings (from 2 to 1000 μm) on electrically conductive materials.

The element composition was studied using a JXA 8100 Electron Probe X-ray Microanalyzer (Japan) and a SPECS X-ray photoelectron spectrometer (Germany). High-resolution surface images were obtained using a HITACHI S-55009 scanning electron microscope (Japan).

To determine the coatings polymer composition, a PY-2020iD Double Shot Pyrolyzer connected to a Shimadzu GCMS QP-2010 gas chromatograph-mass spectrometer was used. Pyrolysis, chromatographic peaks separation, and mass spectrometry detection were carried out under standardized conditions recommended in an F-Search “All-in-One” (Version 3.10) library for polymers identification.

The phase composition was determined using a D8 ADVANCE X-ray diffractometer (Germany) with the sample rotation in $\text{CuK}\alpha$ -radiation. An EVA search program with the PDF-2 database was used for the X-ray diffraction patterns analysis.

2.4. Determination of contact angle and coatings adhesion to the base

The coating/distilled water contact angle was measured by the method of ‘sitting’ drop [17].

The coatings adhesion was estimated from changes in the sample weight upon ultrasonic (US) impact in water according to the technique

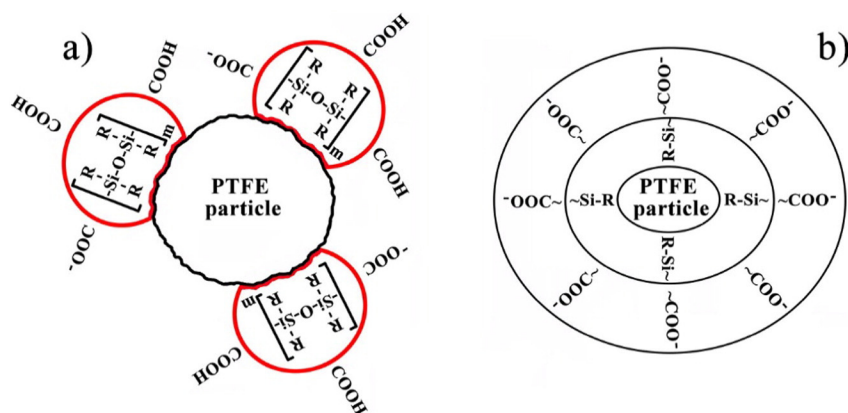


Fig. 2. Possible structural schemes (a, b) for dispersed particles in electrolyte.

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