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- Авторы: N. D. Sakhnenko, O. O. Ovcharenko, M. V. Ved, S. I. Lyabuk
Сахненко Николай Дмитриевич sakhnenko@kpi.kharkov.ua
Овчаренко Ольга Александровна
Ведь Марина Витальевна
Лябук Светлана Ивановна
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- Реферат: We describe an electrochemical method for the formation of composite coatings and foils based on copper reinforced by nanosized aluminum oxide and propose an approach to the chemical dispersion of aluminum oxide based on the principle “from top to bottom” and a composition of electrolyte guaranteeing the possibility of creation of composite materials with different contents of the modifying phase. The influence of the concentration of aluminum oxide in the electrolyte on the physicomechanical properties of the reinforced foil is established. The increase in the strength (and various other physicomechanical characteristics) of the synthesized composite materials is recorded.
Описан электрохимический метод формирования композиционных покрытий и фольги на основе меди, армированной наноразмерным оксидом алюминия, и предложен подход к химическому диспергированию

оксида алюминия по принципу “сверху вниз”, а также состав электролита, обеспечивающий создание композитных материалов с различным содержанием модифицирующей фазы. Установлено влияние концентрации оксида алюминия в электролите на физико-механические свойства упрочненной фольги. Отмечено возрастание прочности (и других физико-механических характеристик) синтезированных материалов.

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PHYSICOMECHANICAL PROPERTIES OF Cu–Al₂O₃ ELECTROPLATING COMPOSITIONS

M. D. Sakhnenko,^{1,2} O. O. Orsharenko,¹ M. V. Ved',¹ and S. I. Lyubak¹

We describe an electrochemical method for the formation of composite coatings and foils based on copper reinforced by nanosized aluminum oxide and propose an approach to the chemical deposition of aluminum oxide based on the principle “from top to bottom” and a composition of electrolyte guaranteeing the possibility of creation of composite materials with different contents of the modifying phase. The influence of the concentration of aluminum oxide in the electrolyte on the physicochemical properties of the reinforced foil is established. The increase in the strength (and various other physicochemical characteristics) of the synthesized composite materials is recorded.

Keywords: composite coatings, electrolytic foils, nanosized oxide, aluminum-oxide hydrosol, microhardness, ultimate strength, yield strength, corrosion behavior.

In recent years, the scientific research in the field of synthesis of functional nanomaterials is rapidly developed [1]. The contemporary investigations are focused on the creation of new approaches to the design and formation of multiphase nanostructures, which is connected with prospects of their application in various branches of science and engineering.

With the development of electroplating, composite coatings deposited from electrolytic suspensions containing highly dispersed powders, as a rule, aluminum, titanium, or zirconium oxides, are used more and more extensively. In the course of electrolysis, particles of the dispersed phase are not reduced. On the contrary, they pass into the cathodic deposits and are fixed in the matrix of the base metal. The collection of basic physicochemical and chemical characteristics of these composite electrolytic coatings (CEC) is better than the same collection for traditional metal coatings. The elevated corrosion resistance, friction resistance, wear resistance, hardness, and other operating parameters of CEC make them quite promising for the modification of the surface of metallic products. Since copper foil and copper coatings are important elements of numerous electronic and electric systems, the possibility of improvement of their strength and hardness with preservation of plasticity by the method of reinforcing of the metallic matrix by nanosized aluminum oxide is of significant interest [2, 3]. For this purpose, it is necessary to perform the electrochemical synthesis of the coatings and foils based on copper and containing Al₂O₃ nanoparticles and study the physicochemical properties of the obtained materials.

Experimental Procedure

Foils based on copper and reinforced by nanosized aluminum oxide were formed on carriers of Kh18N10T stainless steel. The adhered CEC of the same composition were deposited on 20 steel.

¹ Kharkiv Polytechnic Institute National Technical University, Kharkiv, Ukraine.

² Corresponding author, e-mail: mady@i.ua.fm.

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The foils and coatings were electrolytically deposited from a polyglut electrolyte for copper plating [4] of the following composition (g·dm^{−3}): 330–380 potassium dihydrophosphate, 70–90 copper (II) sulfate, and 15–25 sodium citrate with variable content of the dispersed phase. The solutions of electrolytes were prepared from certified chemically pure reagents in distilled water. For electrolysis, we used a stabilized DC source of BS-47 series. The current density was kept on a level of 2–3 A·dm^{−2}. The role of anode was played by plates of M1 copper and the ratio of the cathode area to the anode area was 1:5. Electrolysis was performed within the temperature range 20–25°C for 60–120 min. Depending on the time of electrodeposition, the thickness of the foil and CEC varied from 20 to 50 μm.

To form materials, we added aluminum-oxide sol containing 4.0–4.6 g·dm^{−3} of the dispersed phase to the base electrolyte in the ratio 1:2–5:1, thus varying the Al₂O₃ content of the electrolyte from 1.0 to 2.5 g·dm^{−3}.

Aluminum-oxide hydrosol was obtained from the high-temperature form of γ-Al₂O₃ with nonnormalized size of granules via the interaction with an aqueous solution of sodium hydroxide at pH ≈ 13 for 10–30 min and successive decantation of the colloidal solution. Particles of aluminum oxide were dispersed as a result of partial chemical dissolution of the amorphous aluminum oxide accompanied by the formation of hydroxo-complexes [Al(OH)₄][−] adsorbed on the surface of Al₂O₃ and specifying the charge of formed colloid particles. The stability of these particles

$$[m(\text{Al}_2\text{O}_3) \cdot n(\text{Al}(\text{OH})_4)^- \cdot (n-x)\text{Na}^+ \cdot y\text{H}_2\text{O}]^z \cdot x \cdot \text{Na}^+ \cdot z\text{H}_2\text{O} \quad (1)$$

is provided by [Al(OH)₄][−] charge-generating ions whose stability constant is quite high: $K_s \sim 3.2 \cdot 10^{30}$.

In the electric field, a negatively charged granule of a colloid particle (1) moves toward the cathode due to adsorption of copper cations on the surface. In the near-cathode layer and on the surface of the electrode, granules are adsorbed and dissociate with the release of Al₂O₃ nanoparticles of hydroxide ions and water:

The aluminum oxide from granules (1) and charge-generating anions (2) initiates the formation of nuclei at the sites of its contact with the surface of the cathode, and the parallel process of copper reduction leads to the overgrowing of these particles by the base metal [5, 6].

We studied specimens of foils 10 × 20 mm in size and 40–50 μm in thickness. The microstructure of the foil was studied by the transmission electron microscopy method on an EM-200 microscope under a voltage of 125–175 kV. We prepared objects by thinning the initial specimens in a PTF device. The micrographs were obtained with the use of bright and dark fields in order to improve the visualization of particles of the hardening phase. The grain sizes r were determined from the electron micrographs by the intercept method using the following relation:

$$r = \frac{\sum l}{k \sum n} \quad (3)$$