

Peculiarities of electrophoretic deposition and morphology of deposited films in non-aqueous suspensions of Al₂O₃-Al nanopowder

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Abstract

The paper presents the results of a comprehensive study of the electrokinetic properties of non-aqueous suspensions of the Al₂O₃-Al nanopowder obtained by the method of electric explosion of wires with 0.3 wt.% of metallic aluminum in its composition. The dependence of zeta potential on the concentration of the Al₂O₃-Al suspension is revealed. The nature of long-term changes in zeta potential and pH in suspensions is established. Appearance of bubbles in the deposited coatings due to the interaction of metallic aluminum particles with the liquid suspension medium during electrochemical reactions on the electrodes is defined as the main feature of the electrophoretic deposition process in the Al₂O₃-Al suspensions. The influence of the suspension preparation method on the deposited coatings' morphology is demonstrated.

Keywords

electrophoretic deposition
electric explosion of wire
alumina weakly aggregated
nanopowder
non-aqueous suspension
zeta potential

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1. Introduction

Formation of Al₂O₃-based ceramic coatings is of interest in diverse fields of mechanical engineering, electrical industry [1], as well as in the field of creating composite materials for electrochemical devices [2, 3]. Al₂O₃-based ceramics is of interest in the field of luminophore production [4]. Well-known methods for developing thin-film coatings such as pulsed laser deposition [5] or chemical vapor deposition [6] require the use of complex and expensive equipment and limit the scalability of the technology.

Electrophoretic deposition (EPD) from suspensions is an instrumentally simple method, which allows coatings' formation on the surface of substrates of various shapes and sizes at high rates, easily controlled by the strength of the applied electric field [7–10]. The EPD process in a liquid dispersion medium is caused by the motion of particles in the suspension under the action of an external electric field and their deposition on the electrode [9]. One of the possible deposition mechanisms is electrochemical coagulation of the suspension in the near-electrode region [11]. The movement of particles in suspension or electrophore-

sis proceeds due to the formation of an excess electric charge on the particles and the formation of an electric double layer (EDL) due to the specific adsorption of potential-determining ions, for example, protons, on the particle surface [12, 13]. The stability of the colloidal system [14, 15] and the electrokinetic properties of the suspension depend on the zeta potential value, which directly determines the electrophoretic mobility of particles in the suspension and the deposition rate [16, 17].

The EPD method was used to obtain coatings based on aluminum oxide using commercial powders [18, 19]. However, the properties of suspensions and obtained EPD coatings were shown to depend significantly on the type and size of particles of the powders used, which is probably related to the specific technologies for their production, used by the manufacturer. The method of electric explosion of wires (EEW) [20–23] allows obtaining weakly aggregated nanoparticles of nearly spherical shape. Suspensions based on such powders are characterized by the appearance of a self-stabilization effect [24] and high values of zeta potential, which makes it possible to use suspensions of the EEW nanopowders without the addition of

dispersants and charging agents. In our recent work, we demonstrated the efficiency of using Al₂O₃-based nanoparticles for the formation of bulk ceramics by the EPD method [25, 26]. When using the EEW Mg-doped Al₂O₃ nanopowder containing 0.3 wt.% of metallic aluminum, an improvement in the sintering properties of the ceramics was demonstrated, which confirms the relevance of the development of colloidal technologies based on ceramic nanopowders containing metal particles. The study of the features of the EPD kinetics during the formation of a YSZ (yttria-stabilized zirconium dioxide)/Al₂O₃ composite coating was carried out in [27], where the sintering additive containing Al particles was introduced to decrease the sintering temperature down to 1150 °C. Structural features of YSZ/Al₂O₃ composite coatings obtained by EPD from the suspensions containing metallic Al particles were studied in [28]. The study of the sintering behavior of individual (YSZ) and composite coatings (YSZ/Al₂O₃) was carried out in [29] with the use of the YSZ and Al₂O₃ nanopowders obtained by the laser condensation-evaporation and EEW methods. The influence of the EPD modes under the deposition process in dilute suspensions (1 g/L) of CuO_x with the addition of Al nanoparticles on the weight of the obtained coatings is discussed in [30].

The purpose of this work was to study the electrokinetic properties of the suspensions based on the EEW Al₂O₃ nanopowder containing metallic Al (Al₂O₃-Al) and to determine the effect of the metal component in the composition of the nanopowder on the morphology of the EPD coatings. The regularities of aging of suspensions based on ceramic nanopowders with a metal component are not currently covered in the literature. Thus, as a separate task of the study, the issue of changing the properties of suspensions during their long-term storage and resulting aging effects were singled out. This can be important from the point of view of the practical application of the EPD technology, since the prepared suspensions are commonly stored for a long time to be reused for the deposition.

2. Experimental part

The Al₂O₃ and Al₂O₃-Al nanopowders were obtained by the EEW method using the wires made of metallic Al and Al-Mg alloy with a Mg content of 1.3 wt.%, respectively [20–22]. The specific surface area (*S*_{BET}) of Al₂O₃ and Al₂O₃-Al nanopowders was 42 and 40 m²/g, respectively; it was determined by the Brunauer-Emmett-Teller (BET) method using a TriStar 3000 vacuum sorption unit (Germany).

X-ray phase analysis of nanopowders was carried out using a D8 DISCOVER diffractometer (Bruker AXS, Germany). Processing of the XRD data was performed using the TOPAS-3 program. According to the XRD data (Table 1), the initial Al₂O₃-Al nanopowder did not contain magnesium as a separate crystalline phase. Probably, magnesium

is present in the form of interstitial or substitutional ions in the aluminum oxide crystal lattice. We have calculated the amount of Mg in the composition of the nanopowder with respect to Al₂O₃. Taking into account the proportion of Mg in the Al-Mg alloy, equal to 1.3 wt.%, the calculated content of Mg in the composition of Al₂O₃ is 0.67 wt.%.

Table 1 XRD data for the Al₂O₃ and Al₂O₃-Al nanopowders.

Crystalline phase (content, wt.%)	Lattice type (space group)	Lattice parameters, Å	CSR, nm
Al ₂ O ₃ powder			
γ-Al ₂ O ₃ (15)	cubic (Fd-3m)	a = 7.919(8)	18(1)
δ-Al ₂ O ₃ (85)	orthorhombic (P222)	a = 7.934(8) b = 7.956(8) c = 11.711(8)	18(2)
Al ₂ O ₃ -Al powder			
γ-Al ₂ O ₃ (31)	cubic (Fd-3m)	a = 7.950(10)	26(2)
metallic Al (0.3)	cubic (Fm-3m)	a = 4.054(4)	150(15)
α-Al ₂ O ₃ (0.4)	rhombohedral (R-3c)	a = 4.764(4) c = 12.990(20)	180(16)
δ-Al ₂ O ₃ (69)	orthorhombic (P222)	a = 7.934(8) b = 7.956(8) c = 11.711(8)	18(2)

According to the transmission electron microscopy study performed using a JEM 2100 transmission electron microscope (JEOL, Japan), the nanopowders' particles were weakly aggregated and had a spherical shape (Figure 1). Lognormal particle size distributions for Al₂O₃ and Al₂O₃-Al powders were obtained using graphical analysis of TEM images. The distribution function was as follows:

$$f(D) = \frac{1}{D\sigma\sqrt{2\pi}} e^{-\frac{(\ln D - \ln \mu)^2}{2\sigma^2}}, \quad (1)$$

where *D* is the diameter of particles, nm; μ is the distribution average value, nm; σ is dispersion of the ln*D* distribution. The values of the parameters were $\mu = 14.8$ nm, $\sigma = 0.608$ for the Al₂O₃ nanopowder and $\mu = 19.0$ nm, $\sigma = 0.632$ for the Al₂O₃-Al nanopowder, respectively.

The Al₂O₃ and Al₂O₃-Al nanopowders were used to prepare suspensions in an isopropyl alcohol medium without introducing dispersants or other additives. A suspension of the Al₂O₃ nanopowder was prepared with a concentration of 10 g/L. Initial suspensions of the Al₂O₃-Al nanopowder were prepared with concentrations of 25, 50, 100, 150 and 250 g/L. All the suspensions were sonicated using an ultrasonic bath UZV-13/150-TN (Reltek, Russia) for 125 min. In freshly prepared suspensions with different concentrations, zeta potential and pH were measured. The Al₂O₃-Al suspensions were stored for a period of time up to 250 days with following measurements of zeta potential and pH. Separately, a series of experiments was carried out for the Al₂O₃-Al suspensions with a fixed concentration of 100 g/L, both freshly prepared and aged for 100 days.

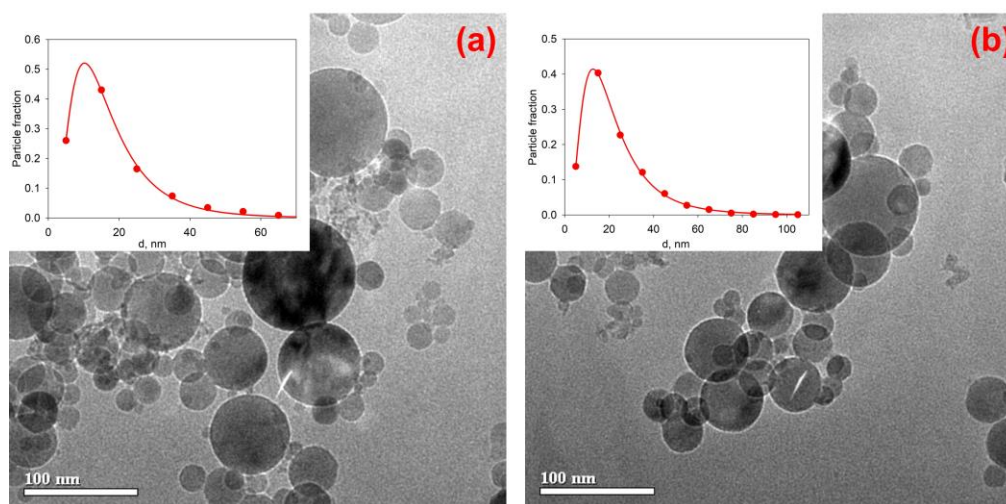


Figure 1 Morphology of nanoparticles and numerical size distributions for Al_2O_3 (a) and $\text{Al}_2\text{O}_3\text{-Al}$ (b) nanopowders.

Large aggregates remained in the suspensions after the ultrasonic treatment were removed by centrifugation using a Z383 centrifuge (Hermle Labortechnik, Germany) at a speed of 6000 rpm for 3 min. Measurements of the electrokinetic zeta potential and pH in suspensions were carried out by the electroacoustic method using a DT-300 analyzer (Dispersion Technology, USA). All measurements for the suspensions were carried out under isothermal conditions in air at 25 °C.

Electrophoretic deposition was performed using a specialized computerized setup providing constant voltage modes, which was developed and manufactured at the IEP, Ural Branch, Russian Academy of Sciences. EPD was performed with a vertical arrangement of the electrodes. The deposition was carried out in a cell with Ni-foil electrodes, the distance between them was 10 mm. During the EPD process, the voltage ranged from 5 to 50 V, while the deposition time was varied from 20 s to 15 min. The deposited coatings were dried on the electrode for a day at room temperature in a Petri dish. The thickness of the coatings was estimated by measuring their weight and taking into account the area and density of the coatings. Morphology of thin-film coatings obtained by the EPD method was studied using an optical microscope ST-VS-520 (Russia). The value of the current during EPD was measured using a UT71E digital multimeter (Uni-Trend Technology, China).

3. Results and discussion

3.1. Influence of the concentration and exposure time of the $\text{Al}_2\text{O}_3\text{-Al}$ suspension on the zeta potential and pH values

The results of measurements of zeta potential (ζ -potential) of the freshly prepared suspensions of the $\text{Al}_2\text{O}_3\text{-Al}$ nanopowder with various concentrations (25, 50, 100, 150, 250 g/L) after ultrasonic treatment for 5 min are shown in Figure 2a. It can be seen that with increasing concentration of the powder in the suspension the value of ζ -potential decreases. This dependence is probably associated with an

increase in the interparticle interaction of nanoparticles with an increase in their concentration in the suspension due to overlapping electrical double layers [31].

ζ -potential and pH time changes in the $\text{Al}_2\text{O}_3\text{-Al}$ suspensions were studied for 250 days to determine the suitability of the suspensions commonly used in the technology of EPD coatings for an extended period of time. From dependences presented in Figure 2b, it can be seen that in the initial period of time up to 66 days the value of ζ -potential increased for all the studied suspensions of various concentrations. With a further increase in the storage time (more than 66 days), changes in ζ -potential became sign-alternating and chaotic. The most significant time changes in ζ -potential were noted for a suspension with a concentration of 100 g/L. Significant changes in pH of the suspensions were observed during the first 150 days of the (Figure 2c) followed by a return to the initial values, and then the pH values stabilized. The change in pH over time had a similar character at different concentrations of the suspensions. The noted peculiarities of the dynamics of pH and ζ -potential may be associated with the hydrolysis of aluminum cations with the formation of hydrated complexes $(\text{Al}_2(\text{OH})_2(\text{H}_2\text{O})_8)^{4+}$, $(\text{Al}_3(\text{OH})_4(\text{H}_2\text{O})_9)^{5+}$, as well as $(\text{Al}_3\text{O}_4(\text{OH})_{24}(\text{H}_2\text{O})_{12})^{7+}$, as shown in [32]. The suspensions of various concentrations are characterized by a high initial value of ζ -potential (fresh, not aged suspensions), which may be associated with the effect of self-stabilization due to the formation of aluminum cations in the dispersion medium as a result of the hydrolysis of trace amounts of nitrates, which inevitably appear on the surface of Al_2O_3 nanoparticles during their production by the EEW method [24]. According to the XRD data, the $\text{Al}_2\text{O}_3\text{-Al}$ nanopowder used for the suspensions' preparation contains 0.3 wt.% of metallic aluminum (Table 1), and, despite its small proportion, aluminum particles can actively react with a trace amount of water in the suspension. This can radically affect the electrokinetic properties of the suspensions and morphology of the deposited coatings, as well as cause slow changes in ζ -potential and pH over time.

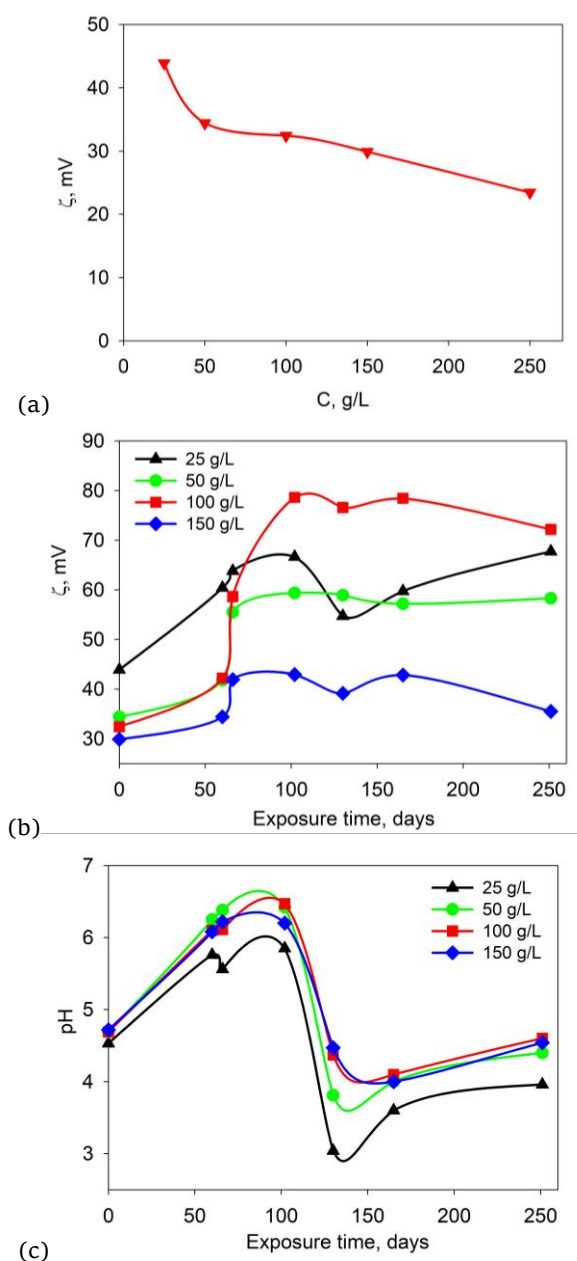


Figure 2 Dependences obtained for the suspensions based on the Al_2O_3 -Al nanopowders: influence of concentration on the ζ -potential value for the freshly prepared suspensions (a); influence of exposure time of the suspensions of various concentrations (25, 50, 100, 150 g/L) on the ζ -potential value (b); influence of exposure time of the suspensions of various concentrations (25, 50, 100, 150 g/L) on pH (c).

3.2. Influence of the method of the suspension preparation on the morphology of the EPD coatings

The studies in this section were carried out using the suspension with a concentration of 100 g/L of the Al_2O_3 -Al nanopowder. As noted in the previous section, this suspension had the most significant changes in ζ -potential with time. In this regard, a study was made into the effect of processing this suspension (centrifugation, dilution) on the EPD coatings' morphology. The scheme of the experiment is shown in Figure 3. The results of experiments are summarized in Table 2.

Ultrasonic treatment of the freshly prepared suspension with a concentration of 100 g/L of the Al_2O_3 -Al nanopowder was carried out for 125 min followed by the measurement of ζ -potential and pH, the values of which were +42 mV and 5.7 mV, respectively. The resulting suspension was used to conduct EPD on a model electrode (Ni-foil) in a constant voltage mode, varying the applied voltage in the range from 5 to 20 V and deposition time from 20 s to 1 min. The selection of the deposition mode in each case was carried out based on the necessity to obtain a continuous coating. Therefore, for each suspension, the lowest possible EPD voltage was selected at which it was possible to form such a coating (Table 2). After the EPD process conducted in the freshly prepared suspension at a voltage of 20 V and a time of 1 min, the resulting coating with a thickness of 20 μm was continuous, but contained cracks and a large number of bubbles. It should be noted that the appearance of bubbles in coatings during EPD is not characteristic of the deposition of ceramic powder materials from non-aqueous suspensions, and, usually, it is observed mainly for water-based suspensions [8, 33].

In order to determine the possible effect of the suspension concentration on the morphology of the deposited coatings and nature of the bubbles' formation, the freshly prepared suspension of 100 g/L was diluted to a concentration of 10 g/L followed by EPD on the Ni-foil (Table 2). The diluted suspension was characterized by a higher positive ζ -potential of +88 mV and $\text{pH}=6.4$. EPD from the diluted freshly prepared suspension showed that bubbles also formed in the coating, but in a smaller amount than under the deposition in the initial concentrated suspension (100 g/L). However, to obtain a continuous coating with a thickness of $\sim 5 \mu\text{m}$ for 1 min, it was necessary to increase the voltage up to 35 V.

At the next stage, the freshly prepared concentrated suspension (100 g/L) was centrifuged at 6000 rpm for 3 min. After the treatment, the resulting suspension had a concentration of 77 g/L, and the measured ζ -potential and pH values were +50 mV and 4.8, respectively. A continuous coating 8.2 μm thick was obtained at a voltage of 10 V and a deposition time of 1 min. The coating contained a large number of bubbles; therefore, centrifugation did not make it possible to exclude their formation. However, the number of bubbles became less in comparison with those formed during EPD from the initial suspension (concentration of 100 g/L).

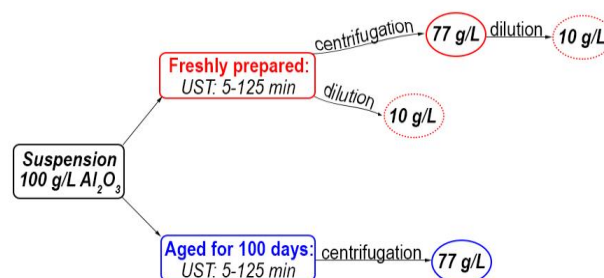


Figure 3 Experimental scheme: effect of centrifugation and dilution of the suspensions on the morphology of the deposited Al_2O_3 -Al coatings.

Considering the previously established tendency for the number of bubbles to decrease with decreasing the suspension concentration, a dilution of the centrifuged freshly prepared suspension (77 g/L) to a concentration of 10 g/L was performed. EPD was carried out at 40 V. The resulting coating with a thickness of approximately 4 μm , formed after 1 min of the deposition time, contained single bubbles, but their number was minimal compared to all previous experiments (Table 2).

The effect of centrifugation on the nature of bubble formation in EPD coatings was studied using the Al_2O_3 -Al suspension with a concentration of 100 g/L aged for 100 days (Figure 3, Table 2). The ζ -potential and pH values, measured in the suspension treated with UST for 125 min, were +50 mV and 4.1, respectively.

The resulting suspension was used to conduct EPD on an electrode (Ni-foil) in a constant voltage mode of 20 V and a deposition time of 1 min. The resulting film of 26 μm in thickness contained a network of cracks and a large number of bubbles. The centrifugation of the suspension aged for 100 days resulted in the formation of a continuous coating with a thickness of 15 μm during EPD, which was characterized by the presence of cracks and a reduced number of bubbles compared to the non-centrifuged aged suspension.

It is of interest to study the kinetics of current change during the EPD process in order to establish the nature of the charge transfer in the Al_2O_3 -Al suspension during the formation of the EPD coating. This study was performed during the EPD process at a constant voltage of 20 V and a deposition time of 15 min from various types of suspensions (with concentration of 100 g/L – freshly prepared and aged for 100 days; with concentration of 77 g/L – centrifuged, freshly prepared and aged for 100 days), and also for pure isopropanol. The results are shown in Figure 4. It is seen that for pure isopropanol the current value does not depend on time and has a value of about 0.01 mA, which is lower than the current values in suspensions (~0.02–0.05 mA). The charge transfer mainly proceeds with the participation of particles in suspensions, and a decrease in current is due to the depletion of the suspension during EPD, as well as

because of an increase in the resistance of the deposited film [9]. With the use of the aged (100 days) suspension with a concentration of 100 g/L, the drop in current became sharper. Centrifugation of the aged suspension (77 g/L) led to the return of the current kinetics to the values of a freshly prepared suspension. In a freshly prepared suspension, centrifugation does not change the current kinetics.

3.3. Possible mechanisms of the bubble formation in the EPD coatings with the participation of a metal component in the Al_2O_3 -Al nanopowder composition

Figure 5 represents optical micrographs of the EPD coatings obtained from the aged suspension of the Al_2O_3 -Al nanopowder with a concentration of 100 g/L (Figure 5a), as well as the coatings obtained from the freshly prepared Al_2O_3 -Al suspension: with a concentration of 100 g/L (Figure 5b); from the centrifuged suspension of 77 g/L (Figure 5c); from the centrifuged suspension diluted to 10 g/L (Figure 5d). Analysis of the images shows that there is a tendency for a decrease in the number of bubbles when using centrifugation of the freshly prepared suspension, especially when it is subsequently diluted to 10 g/L.

In order to confirm the effect of the metal component in the composition of Al_2O_3 -Al nanopowder on the formation of bubbles in the EPD coating, the deposition was carried out from the suspension with a concentration of 10 g/L based on the Al_2O_3 nanopowder on the Ni-foil at a constant voltage of 50 V and deposition time of 1 min. The obtained continuous coating with a thickness of 10 μm did not contain bubbles, which confirmed the earlier assumption about the key role of metallic aluminum in the Al_2O_3 -Al nanopowder composition in the formation of bubbles in the structure of the EPD coatings. Metallic Al (0.3 wt.%) in the composition of the initial Al_2O_3 -Al nanopowder can interact with a trace amount of water contained in isopropyl alcohol to form gaseous hydrogen and aluminum hydroxide.

Table 2 Influence of the Al_2O_3 -Al nanopowder suspension preparation method on the ζ -potential value and morphology of the EPD coatings obtained under the selected EPD modes.

Method of the suspension preparation (concentration, g/L)	ζ -potential, mV (pH)	EPD mode (voltage, deposition time)	Thickness and morphology of the resulting EPD coating
Freshly prepared (100 g/L)	+42 (5.7)	20 V, 1 min	20 μm ; a significant amount of bubbles
Diluted from freshly prepared (10 g/L)	+88 (6.4)	35 V, 1 min	5.6 μm ; numerous bubbles
Centrifuged freshly prepared (77 g/L)	+50 (4.8)	10 V, 1 min	8.2 μm ; numerous bubbles
Diluted from centrifuged freshly prepared (10 g/L)	+80 (6.1)	40 V, 1 min	3.9 μm ; single bubbles
Aged for 100 days (100 g/L)	+50 (4.1)	20 V, 1 min	26 μm ; the largest number of bubbles
Centrifuged and aged (77 g/L)	+53 (5.2)	20 V, 1 min	15 μm ; numerous bubbles

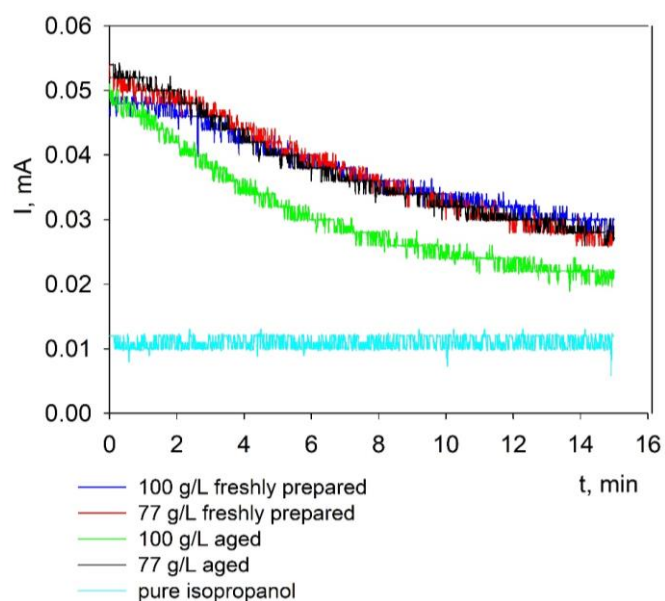


Figure 4 Time dependences of current intensity during EPD performed at an applied constant voltage of 20 V in freshly prepared and aged Al₂O₃-Al suspensions with different concentrations of 100 g/L and 77 g/L and for pure isopropanol.

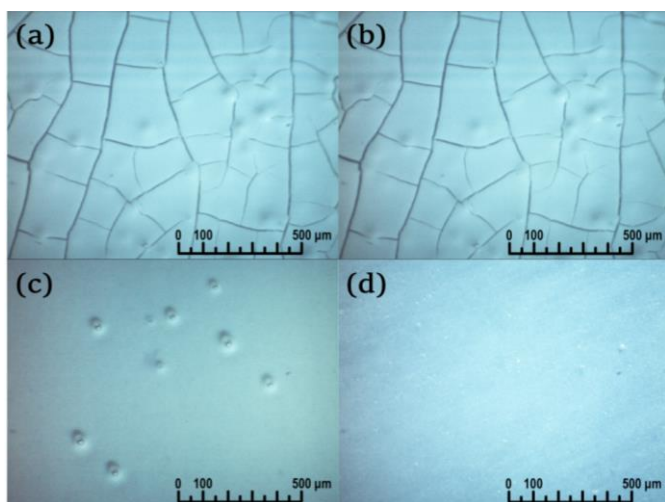


Figure 5 Optical micrographs of the Al₂O₃-Al coatings deposited from the suspensions obtained using different methods: from the aged suspension (100 days) with a concentration of 100 g/L (a); from the freshly prepared suspension with a concentration of 100 g/L (b); from the freshly prepared centrifuged suspension with a concentration of 77 g/L (c); from the freshly prepared centrifuged suspension diluted to 10 g/L (d).

Electrochemical reactions on the electrodes may enhance the interaction of the liquid medium with metallic aluminum due to the reaction occurring at the anode with the formation of water as a result of the oxidation of hydroxide ions: $4\text{OH}^- - 4\text{e}^- = \text{O}_2\uparrow + 2\text{H}_2\text{O}$. At the same time, water decomposes at the cathode with formation of the molecular hydrogen: $2\text{H}_2\text{O} + 2\text{e}^- = \text{H}_2\uparrow + 2\text{OH}^-$, and the reduction of H⁺ protons with the formation of the molecular hydrogen H₂ is also possible [33, 34]

As it was shown above (Figure 4), pure isopropanol is characterized by the time-independent current intensity, which indicates the absence of significant electrochemical reactions involving a trace amount of water in pure alco-

hol, since the conductivity of the liquid medium does not change for a long time (15 min). Therefore, it can be concluded that the main contribution to the electrochemical electrode reactions during EPD is made by ions present in the EDL of solvated particles in the suspension. Water can be introduced into the suspension during the process of the powder dispersion, as well as during the oxidation reaction of hydroxide ions at the anode. Exposure of the suspension over time has a negative effect on the formation of bubbles in the coating, which may be due to an uncontrolled change in the ionic composition in the suspension. The experiments performed show that the use of centrifugation of the Al₂O₃-Al suspension allows the formation of bubbles in the coatings to be significantly reduced, possibly due to the partial removal of aluminum metal particles during centrifugation. The particularities revealed in the use of the EEW Al₂O₃-Al nanopowder can be considered when using other ceramic powdered materials containing metal particles in the EPD technology of functional coating.

4. Conclusions

In the present work, a comprehensive study of the electrokinetic properties of the suspensions based on the Al₂O₃-Al nanopowder, obtained by the method of electric explosion of wires and containing a small fraction (0.3 wt.%) of metallic aluminum, was carried out with variation in their concentration and aging time. A decrease in zeta potential with an increase in the concentration of the Al₂O₃-Al suspension was shown, which is due to the influence of interparticle interaction in a concentrated suspension. The appearance of bubbles in the coatings obtained by EPD from suspensions of the Al₂O₃-Al nanopowder in isopropyl alcohol was found, while no bubbles were formed in the suspension of the Al₂O₃ nanopowder. Thus, the presence of metallic aluminum particles in the composition of the EEW nanopowder was revealed as the most possible reason for the appearance of bubbles in the structure of the Al₂O₃-Al deposited coatings, which, in turn, is enhanced by the electrochemical reactions on the electrodes. It was shown that the long-term storage of the suspensions of the Al₂O₃-Al nanopowder for their following usage for EPD to obtain coatings of the satisfactory quality should not exceed approximately two months (66 days).

Supplementary materials

No supplementary materials are available.

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Formal Analysis: E.G.K., D.S.R.

Funding acquisition: E.G.K.

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Conflict of interest

The authors declare no conflict of interest.

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