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Abstract






The basis for determining the density of electrons in plasma is the study of the broadening of spectral lines, which is caused by the linear Shtark- effect. This article demonstrates the applicability of the classical theory of scattering, with the help of which, analyzing the spectral characteristics of spark plasma discharges in the electrochemical system of magnesium-electrolyte, determined that during the synthesis of oxide-ceramic coatings on a magnesium basis in discharge channels for electron density $(3.2...3.4) \cdot 10^{22} \text{ m}^{-3}$ and their temperatures $(1...1.1) \cdot 10^4 \text{ K}$ equilibrium, not degenerate, quasi-ideal plasma is formed with an ionicity (0.02...0.10). This temperature makes conditions for dissociation and ionization of electrolyte components and also ensures high chemical reactivity. At the same time, the speed of the reaction is much higher compared to classical electrochemical methods. The performed calculations provide the possibility of further correct thermodynamic calculation of the concentrations of the reaction products of the synthesis of oxide ceramic coatings in the electrolytic plasma of the magnesium alloy–electrolyte system.

Keywords
(separated by '-')

Product Innovation - Plasma-Electrolyte Treatment - Oxide Ceramic Coatings - Magnesium Alloys -
Electron Density - Electron Temperature - Industrial Growth



The State of Electrolytic Plasma in Synthesis of Oxide Ceramic Coatings on the Magnesium Basis

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Abstract. The basis for determining the density of electrons in plasma is the study of the broadening of spectral lines, which is caused by the linear Shtark-effect. This article demonstrates the applicability of the classical theory of scattering, with the help of which, analyzing the spectral characteristics of spark plasma discharges in the electrochemical system of magnesium-electrolyte, determined that during the synthesis of oxide-ceramic coatings on a magnesium basis in discharge channels for electron density $(3.2...3.4) \cdot 10^{22} \text{ m}^{-3}$ and their temperatures $(1...1.1) \cdot 10^4 \text{ K}$ equilibrium, not degenerate, quasi-ideal plasma is formed with an ionicity $(0.02...0.10)$. This temperature makes conditions for dissociation and ionization of electrolyte components and also ensures high chemical reactivity. At the same time, the speed of the reaction is much higher compared to classical electrochemical methods. The performed calculations provide the possibility of further correct thermodynamic calculation of the concentrations of the reaction products of the synthesis of oxide ceramic coatings in the electrolytic plasma of the magnesium alloy–electrolyte system.

Keywords: Product Innovation · Plasma-Electrolyte Treatment · Oxide Ceramic Coatings · Magnesium Alloys · Electron Density · Electron Temperature · Industrial Growth

1 Introduction

Last time, the plasma-electrolytic oxidation method on valve metals was intensively developed. Plasma electrolytic oxidation (PEO) includes complex chemical and electrochemical processes occurring in microdischarge zones with high temperatures and gas pressure on the border at the anode-electrolyte. Products of anodic oxidation and products of thermal transformations in these processes can change the properties of coatings. The PEO method makes it possible to obtain coatings on the surface of valve metals,

which in terms of physical and chemical properties (hardness, heat and wear resistance, corrosion resistance), are identical to refractory ceramic materials [1].

In the past decades, various traditional surface treatments, such as physical vapor deposition [2], chemical vapor deposition [3], ion beam-assisted deposition [4], and spraying [5], have been applied to metallic substrates to improve their general poor tribological properties [6, 7] for ensuring the reliability of highly loaded parts [8, 9]. However, most of the aforementioned methods involve high processing temperatures that can degrade coatings or substrates.

Understanding the type of plasma in the electrolyte will explain the processes of interacting electrolyte components with metal particles. Explanations of such processes help predict future properties of coatings.

Current theories do not allow determining the physical properties of the plasma. They exist only for partial cases, namely for specific temperature ranges. In the conditions of concrete temperature, fundamental properties of plasma as a Coulomb system depend on the amount of charged particles, size of the plasma channel, and time of existence. In the previous work [7], the temperature of plasma's electrons was determined during the synthesis of oxide-ceramic coatings on a magnesium basis, assuming that the energy distribution of the particles is described by the Maxwell-Boltzmann equation. To confirm or disprove these assumptions and to conduct further correct thermodynamic calculations of the output synthesis products, it is necessary to investigate the state of electrolyte plasma. The purpose of this work is to determine the state of electrolyte plasma in the magnesium-electrolyte system, namely, whether it is degenerate (non-degenerate), ideal (non-ideal), Debye (non-Debye), equilibrium (non-equilibrium), classical or quantum.

2 Literature Review

It is known that the properties of coatings are influenced by synthesis modes [10]. In work [11], authors investigated by optical emission spectroscopy that the transition of a volt-temporal characteristic is associated with developing an intermediate coating layer with an average hardness of 270–450 HV0 HV_{0.05}. Also, research has shown that coatings grew at a rate in the range of 4.0–7.5 $\mu\text{m min}^{-1}$, depending on the substrate composition. Regardless of the substrate, the coatings consisted of MgO and Mg₂SiO₄, incorporating alloying element species.

Studies by authors [12] showed that introducing silica nanoparticles into PEO electrolytes leads to their reactive incorporation in coatings and thus influences the degradation behavior.

Xinyan Wang et al. described that the higher content of Cr₂O₃ particles led to the production of “green coatings” with improved thermal control and mechanical properties. The existence of particles has somewhat improved corrosion resistance and hardness [13, 14]. OCC-covering magnesium alloys are porous. The decrease in porosity can be achieved by adding a GeO₂ particle to the electrolyte, which significantly increases its corrosion resistance. The optimum concentration of CeO₂ particles was 2 g/l [15]. Adding reactive particles to PEO coatings aims to seal the porosity and provide a broader range of coating compositions. Experiments with nano- and micro-sized SiO₂ particles and different concentrations of KOH in the electrolyte provide new insights into possible

up-take and incorporation mechanisms of particles during PEO processing [16]. It was established that the radiation of electrolyte plasma in the valve metal-electrolyte system consists of a continuous spectrum of electron radiation, linearity – atoms and ions, striped molecules, and radicals. The continuous spectrum arises as a result of free-to-free transitions and free-bound transitions. Free-free transitions occur while inhibiting a free electron in the ion field. The photon, which is absorbed or emitted in this case, has an energy corresponding to the difference in the energy of two quantum states of ion and electron. The radiation that occurs when an ion braking on an ion or electron on an electron is much less intense than during electron inhibition in the ion field [17].

3 Research Methodology

The formation of OCC by the PEO method and the coating properties were investigated on samples of magnesium alloys.

The synthesis of OCC was carried out on the IMPELOM-1 installation (Fig. 1), which includes a current control unit and a power source (1), baths for electrolytes (2), and cooling units (3). The cathode was a stainless-steel bath where the electrolyte was located. With the help of measuring devices (voltmeter and ammeter), the value of the dependence of the voltage of the anode and cathode currents on time was recorded. The used electrolytes were formed based on distilled water and were constantly mixed with air (Fig. 1). A more detailed description of the making technology on the OCC surface is presented in [18].

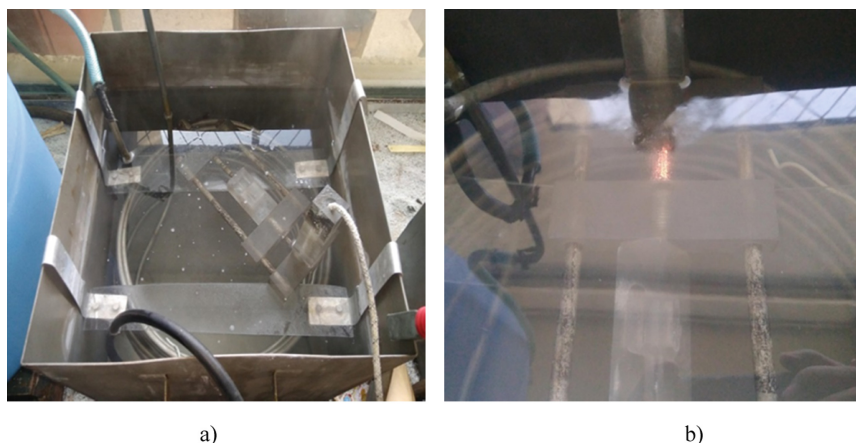


Fig. 1. Electrolytic bath with simple into the electrolyte (a) and the OCC formation process (b).

The physical parameters of the electrolytic plasma, in which oxides are synthesized on a metal substrate, were studied on a special low-power installation for oxide-ceramic oxidation equipped with a C-115 spectrophotometer (Fig. 2).



Fig. 2. C-115 spectrophotometer.

Under the action of the current on the sample located in the electrolyte bath, spark discharges occur, during which the plasma state is realized, and radiation enters the input window of the spectrophotometer through the created output window. In this way, the emission spectra of the electrolytic plasma of the magnesium alloy-aqueous solution system were studied.

The characteristic spectrum of electrolytic plasma radiation is shown in Fig. 3, a. An electrolyte was an aqueous solution of $1 \text{ g l}^{-1} \text{ KOH} + 1 \text{ g l}^{-1} \text{ Na}_2\text{SiO}_3$ (with a density of $1,38 \text{ g/cm}^3$). The oxide ceramic coatings were synthesized on an alloy of the 10-fold HDPE (Zn-0.8; Y-7.1–7.9; Cd-0.63; Zr-0.5; Mg-base). Current density – 3 kA/m^2 , the ratio of densities of the cathode to anode currents $J_c/J_a = 1$, pulse frequency 50 Hz. The spectrum of radiation was compared to the spectrums obtained in [11], where the plasma-electrolyte treatment (PEO) was carried out at a current density of 6.5 kA pulses of rectangular shape from the ratio of densities of the cathode to anode currents $J_c/J_a = 1.2$ with a frequency of 50 Hz in the water-electrolyte composition: $25 \text{ ml l}^{-1} \text{ Na}_2\text{SiO}_3$ та $15 \text{ g l}^{-1} \text{ Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$. The radiation spectrum entered a spectrophotometer with an optical fiber cable, allowing one to look after the radiation of magnesium in an area up to 300 nm (Fig. 3, b).

The lines of atoms were identified: hydrogen - H_αI , H_βI (656.28; 486.13) nm; MgI (333.21; 333.67; 382.93; 383.23; 383.83; 517.27; 518.36) nm; single ionized magnesium MgII -448,10; and electrolyte: NaI (589.00; 589.59) nm; radicals OH (306.4; 306.7) nm.

The breakdown of the tape formed in the poorly concentrated electrolyte occurs at a higher intensity of the electric field, which correlates with the radiation of a greater amount of lines that are observed in a spectrum (Fig. 1, a), compared with the spectrums given in [11] (Fig. 3, b), where highly concentrated electrolytes are used, and the dielectric properties of the coating and the breakdown stresses are lower.

According to the expansion of the hydrogen line H_α based on the Stark effect and the relative intensities of the radiation lines of ions and magnesium atoms and the methods outlined in [19], it was found that in the discharge channels, during the synthesis of oxide ceramics on a magnesium basis, a plasma with a density of electrons $n_e = (3.2 \dots 3.4) \cdot 10^{22} \text{ m}^{-3}$ and an electron temperature $T_e = (1.0 \dots 1.1) \cdot 10^4 \text{ K}$.

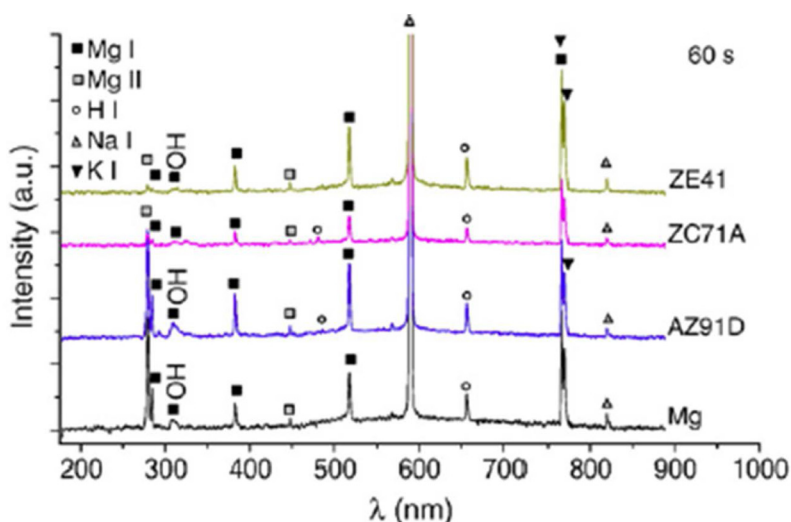
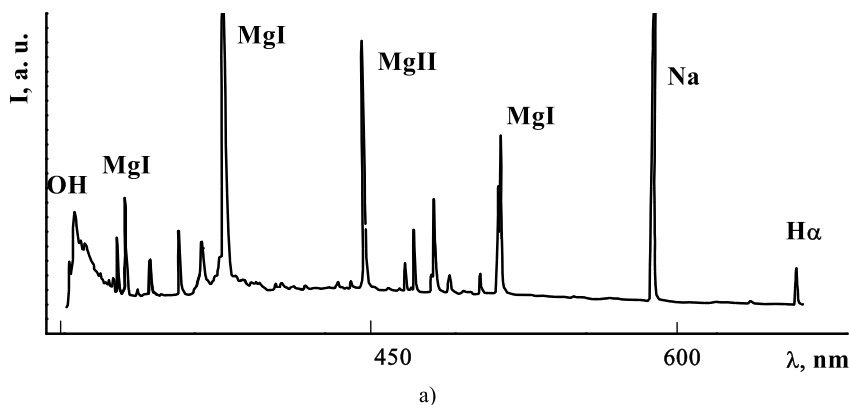


Fig. 3. Emission spectra of electrolyte plasma during the synthesis of oxide-ceramic coatings on magnesium alloys are given in works [19] a) [12] b).

4 Results and Discussion

If the density of electrons in the plasma and their temperature gradually change over time, then the equation of the state of an ideal gas can be fulfilled. The redistribution of energy between the particles in the plasma is due to the thermal motion, and the Coulomb interaction is small. It can be described by an expression $(\bar{Z}_e)^2/V_0 \ll kT$, where \bar{Z}_e - the average charge of particles; $V_0 \approx n^{1/3}$ - average distance between them; n - the amount of particles in 1 cm^3 of gas. This inequality can be written as:

$$n \ll 2, 2 \cdot 10^{14} (T_0/\bar{Z}_e^2)^3. \quad (1)$$

For $T_e \approx 10^4 \text{ K}$ та $n_e \approx n_i \approx 3.4 \cdot 10^{22} \text{ m}^{-3}$, the maximum value is $\bar{Z}_e = 2$, and condition (1) is executed. Coulomb interaction between particles is manifested in the fact that each

ion tries to draw particles of the opposite sign and has some energy of communication in the ionized gas. As a result, adding somewhat lower energy to the atom's ionization is necessary. This condition is considered in the modified Saha Eq. (2) when determining the electron temperature [19].

$$\frac{I_1}{I_2} = 2 \frac{f_1 g_1 \lambda_2^3}{f_2 g_2 \lambda_1^3} \cdot \frac{(2\pi m_0 k)^{3/2}}{h^3} \cdot \frac{1}{n_e} T_e^3 \cdot \exp \left[- \left(\frac{E_1 - E_2 + \kappa - \Delta\kappa}{kT_e} \right) \right] \bar{u} \cong e n_e^{3/2} \quad (2)$$

where h – Planck's constant; m_0 – the electron mass; f – force of oscillators; g – statistical weight of the upper levels; k – Boltzmann's constant; n – density of electrons; E – energy levels; K – ionization energy; ΔK – reduction of ionization energy; 1, 2 – the indices of the pairs of spectral lines of the ion and the magnesium atom on which the T_e was expected in plasma.

It is necessary to perform certain thermodynamic conditions to determine the temperature of plasma using radiation spectrums:

1. the plasma must be optically thin, which is manifested in the fact that the radiation that occurs in the internal areas of plasma emanates from it;
2. the plasma is in a state of complete, local, or partial local thermodynamic equilibrium. A complete thermodynamic equilibrium in a laboratory environment is never achieved. The condition for achieving local thermodynamic equilibrium (LTE) is described by the relation [19]:

$$n_e = 9 \cdot 10^{23} \left(\frac{E_2}{\kappa_H} \right)^3 \left(\frac{kT}{\kappa_H} \right)^{1/2} m^{-3}, \quad (3)$$

where E_2 – the energy of the first excited level; κ_H – ionization energy of hydrogen. In the case of spark discharge on a magnesium base in an aqueous electrolyte, the concentration of electrons in plasma is determined by the expansion of the hydrogen line H_α is $n_e \approx (3.2 \dots 3.4) \cdot 10^{22} \text{ m}^{-3}$, the electron temperature is $T_e \approx (1 \dots 1.1) \cdot 10^4 \text{ K}$, $E_2 = 10.15 \text{ eV}$ ($1.63 \cdot 10^{-11} \text{ erg}$) (for hydrogen), $K_H = 13,595 \text{ eV}$ ($2.18 \cdot 10^{-11} \text{ erg}$) [20, 21]. The condition of the LTE (3) is not fulfilled. Consequently, the Boltzmann distribution does not describe the population density in optically thin plasma. The kinetic energies of the electrons and atoms are not the same. In contrast to the previous case, a local thermodynamic equilibrium (LTE) may occur for all processes of collisions if we exclude atoms in the ground states from consideration [19]. In this case, talk about a partial LTE. The relation describes the condition of achievement of partial LTE:

$$n_e > 7 \cdot 10^{24} \frac{Z^b}{n^{17/2}} \left(\frac{kT}{\kappa_H} \right) m^{-3}, \quad (4)$$

where n – the main quantum number of the lower level, which is included in the condition of partial LTE.

For the plasma parameters given above, $n = 2$, $Z^b = 1$ and we obtain.

$3.2 \cdot 10^{22} \text{ m}^{-3} > 4.8 \cdot 10^{21} \text{ m}^{-3}$, that is, the condition of partial LTE is fulfilled and the plasma state can be described by parameters n_e , T_e and the reduction of ionization energy – $\Delta\kappa$, which is included in the modified Boltzmann and Saha formulas.

The degree of degeneracy of plasma (the degree of filling of possible quantum states by the particles of the system) can be found as the ratio of the number of possible energy states to the number of particles, comparing the mean de Broglie wavelength – λ ; with an average radius between the particles – r_0 .

For a given temperature T , the number of permissible states is equal to the ratio of the phase space, which corresponds to the mean particle pulse $p = (2mkT)^{1/2}$, to the volume of one state – λ^3 . Then the degeneracy criterion is described by the expression:

$$A_b = \frac{n\hbar^3}{(2\pi mkT)^{3/2}} = \exp(\mu/kT), \quad (5)$$

where \hbar – Planck's constant; m – the mass of the particle; k – Boltzmann's constant; n – the number of particles per unit volume of plasma; μ – the chemical potential of the particle.

The plasma state was analyzed according to the $n_e - T_e$ diagram (Fig. 4) using the methodology outlined in [21]. It calculated the parameters for the temperature of electrons $T_e \approx (1...1.1) \cdot 10^4$ K and the densities of electrons $n_e \approx (3.2...3.4) \cdot 10^{22} \text{ m}^{-3}$.

As at $A_b \ll 1$, the gas is non-degenerate, then the statistics of Maxwell-Boltzmann [13] are used. Calculations showed that in the interval $T_e (1...1.1) \cdot 10^4$ K the degree of degeneracy of plasma electron gas $A_{be} = (4.92...5.34) \cdot 10^{-8}$, hence $\mu = -(2.31 \cdot 10^{-11} ... 2.56 \cdot 10^{-11})$ erg. It is known that degeneration of plasma atoms is observed at absolute temperatures close to zero and densities $n \approx 10^{28} \text{ m}^{-3}$. In the range of temperatures $10^3 \text{ K} \leq T \leq 10^7 \text{ K}$, the degeneration of atoms and ions occurs at densities $n > (10^{32} ... 10^{40}) \text{ m}^{-3}$, which is consistent with the results obtained above (Fig. 4).

The condition of application of classical scattering theory is written down in the form $R/\lambda > 1$, where $R = e^2/kT$ – is the mean radius of interaction, or otherwise, the Coulomb scattering amplitude, e – the charge of the electron, λ – the mean thermal wavelength of the de Broglie particle, which characterizes the width of the wave packet. For 10^4 K – $R = 1.67 \text{ nm}$, for $1.1 \cdot 10^4 \text{ K}$ – $R = 1.62 \text{ nm}$.

Thus, in the low-temperature plasma of the magnesium-electrolyte system, the interaction between heavy particles (ion-ion, ion-neutral, neutral-neutral) can be described with the help of the theory of classical trajectories, as $R/\lambda \gg 1$. In more detail, one should consider the interaction with the participation of electrons. The de Broglie wavelength is $(m/m_e)^{1/2}$ times larger than the length of this wave of ions or neutrals for a given average kinetic energy of particles. The interaction of an electron-neutral in a low-temperature plasma cannot be described without considering the effects of diffraction caused by the wave nature of electrons, which behave like waves when interacting with atoms or molecules (the Ramzauer effect).

The interaction of electron-electron and electron-ion is Coulomb, so there is a region of plasma parameters where the classical theory of scattering is used despite the large wavelength of de Broglie electrons. The criterion for this condition is written as follows:

$$A_k = \frac{e^2}{\hbar} \left(\frac{m_e}{kT} \right)^{1/2} \geq 1 \quad (6)$$

For the considered range of temperatures in our case $A_k = (5.36...5.62)$.

For a plasma with a density of electrons $n_e = 10^{22}\text{--}10^{23} \text{ m}^{-3}$, the values of the electron temperature and gas temperature are similar. Similarly, it is noted that at atmospheric and higher pressures, the thermal balance prevails for electric arcs.

As can we see from the invoices, in relaxation processes in electrolyte plasma, the limiting one is the single ionization of atoms. It means that for the determination of equilibrium, the duration of the discharge process must be greater than or equal to the characteristic time of ionization. The minimum discharge time in the initial conditions of the synthesis of oxides is $1 \cdot 10^{-4} \dots 2 \cdot 10^{-4} \text{ s}$. Using high-speed photography, the average duration of micro-particles in electrolytes of different compositions on the aluminum alloy AMG-6 was determined in the voltage range 440...540 V. The average lifetime is $2.3 \cdot 10^{-4} \text{ s}$. In another paper [21], using high-speed synthesis video, it is established that the initial lifetime of discharge channels for the magnesium alloys AZ31, AZ61, and AZ91D is $(0.5 \dots 1.9) \cdot 10^{-4} \text{ s}$ and with increasing synthesis time to $(10.0 \dots 40.0) \cdot 10^{-4} \text{ s}$. Thus, in the initial stages of synthesis, the time of existence of the plasma channel is slightly less than the time of establishing equilibrium water ionization $(4.59 \dots 5.11) \cdot 10^{-4} \text{ s} < (1 \dots 2) \cdot 10^{-4} \text{ s}$, but with the continuation of the synthesis time, the existence time of plasma channel increases on the order of magnitude $(10.0 \dots 40.0) \cdot 10^{-4} \text{ s}$. Thus, at the stabilization stage of the synthesis of oxide-ceramic compounds in the magnesium system, an electrolyte plasma in spark discharges can be considered equilibrium.

Spectral studies of spark PEO made it possible to establish the temperature in the center of the spark discharge channel and its periphery. In the pre-spark potential region, the cause of the glow is the luminescence of oxides. Still, as the voltage increases to spark breakdown potentials, separate components of the electrolyte and the electrode metal become radiation sources (Fig. 5).

The process of forming the coating occurs in an electric field, which is a source of energy for plasma-chemical reactions on the working electrode. An increase in the anode potential outside the Faraday zone until the breakdown of the dielectric or semiconductor film of the anode is formed when the Faraday zone is passed. Production breakdown due to the injection of electrons from the valence band into the conduction band. In the breakdown channel, the temperature rises strongly, dissociation and ionization of the electrolyte occur, and a plasma clot is formed, in which the plasma-chemical reactions of oxide synthesis are realized.

As a result, we get magnesium alloy samples with an oxide-ceramic coating, shown in Fig. 6.

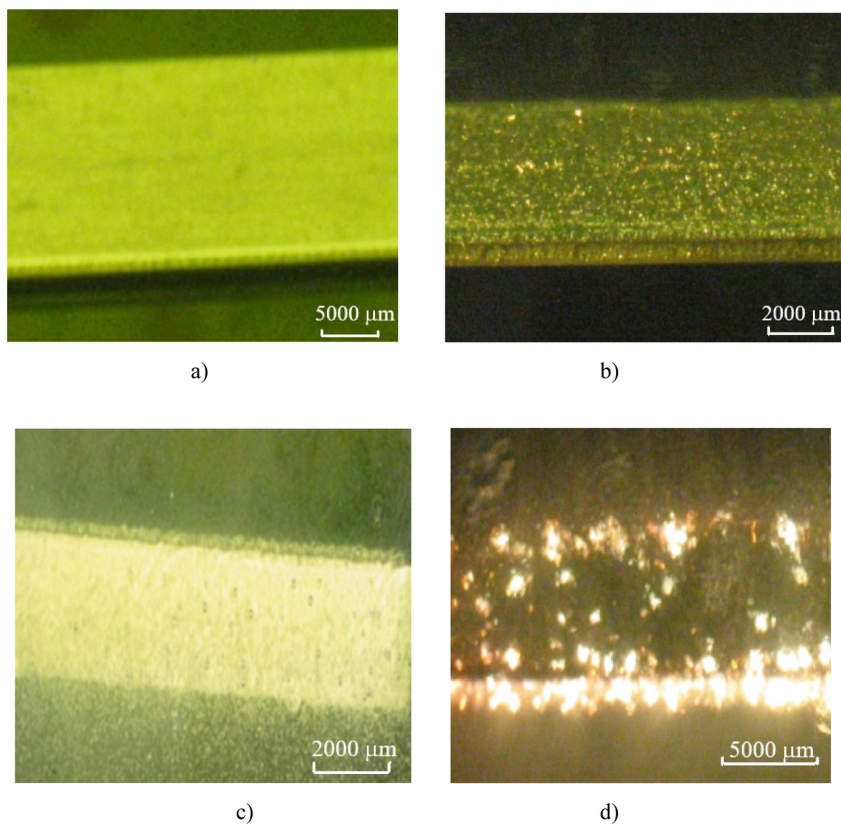


Fig. 5. Synthesis of oxide ceramic coating: a – sample in the electrolyte; b – the beginning of synthesis; c – stable growth of the cover; d – the end of the synthesis.

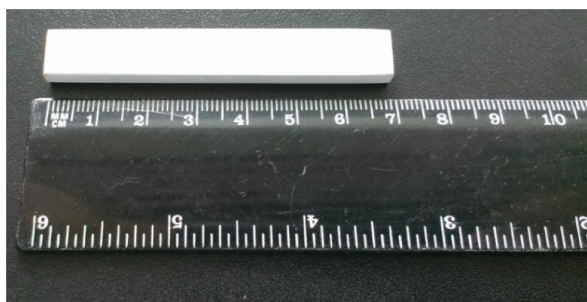


Fig. 6. Osidoceramic coating synthesized on a magnesium alloy.

5 Conclusions

During the synthesis of oxide-ceramic coatings in an electrolyte plasma at a temperature $(1 \dots 1.1) \cdot 10^4$ K with electron density $(3.2 \dots 3.4) \cdot 10^{22} \text{ m}^{-3}$ and a degree of ionization $(0.02 \dots 0.10)$ an equilibrium, non-degenerate, quasi-ideal plasma is realized on a magnesium basis in a discharge channel. These parameters indicate that the particle energy distribution is described by Maxwell-Boltzmann statistics. Accordingly, it is possible to carry out the correct thermodynamic calculations of reactions products concentrations of oxide-ceramic coatings synthesis in an electrolyte plasma of the magnesium alloy system is an electrolyte.

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Chapter 26

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