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Formation features of composite electrochemical nickel and nanostructured zirconium boride coatings[*]

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Особенности формирования композитных электрохимических покрытий на основе никеля и наноструктурного диборида циркония[***]

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Introduction. The electrodeposition of composite electrochemical coatings from electrolyte-colloid nickel plating containing ultradisperse zirconium boride powder is studied. The work objectives are as follows: to study mechanical-and-physical properties of the composites based on nickel and nanostructured zirconium boride, and to determine optimal conditions for the application of such electrochemical coatings.

Materials and Methods. Microhardness of composite electrochemical coatings was measured using PMT-3 microhardness tester on samples with the layer thickness of 30 µm under the indentation load of 100 g. A three-ball machine was used to determine wear resistance of the coatings. Sample tests were carried out under dry friction modes and with the use of 3% RV coolant. WSD values were measured by MIR-3 TU 3-3.1954-86 microscope. To determine the internal stresses in the coating, we used a flexible cathode method up to GOST 9.302-88.

Research Results. The electrolyte-colloid composition and modes of electrodeposition of composite nickel - nanostructured zirconium boride coatings are developed. Mechanical-and-physical properties (microhardness, wear resistance and internal stresses) of the obtained composite electrochemical coatings are analyzed. Recommendations for use of the developed electrolyte and the application of a composite coating on machine parts for their surface hardening are formulated.

Discussion and Conclusions. $Ni\text{–}ZrB_2$ CEC (composite electrochemical coating) has high microhardness (10–11 hPa at the indentation load of 100 g), which exceeds the microhardness of pure nickel by 1.5–2 times. As the microhardness increases, the internal stresses of $Ni\text{–}ZrB_2$ CEC decrease. The proposed coatings were compared to chromium ones deposited from the environmentally hazardous electrolytes. The wear resistance of

Введение. Исследован процесс электроосаждения композитных электрохимических покрытий из электролита-коллоида никелирования, содержащего ультрадисперсный порошок диборида циркония.

Цели работы: исследование физико-механических свойств композитов на основе никеля и наноструктурного диборида циркония, а также определение оптимальных условий нанесения такого электрохимического покрытия.

*Материалы и методы***.** Микротвердость композитных электрохимических покрытий измеряли с помощью микротвердомера ПМТ-3 на образцах с толщиной слоя 30 мкм при нагрузке на индентор 100 г. Для определения износостойкости покрытий использовали трехшариковую машину трения. Испытания образцов проводили в режимах сухого трения и с применением 3 % смазочно-охлаждающей жидкости РВ. Значения диаметра пятна износа измерили под микроскопом МИР-3 ТУ 3-3.1954-86. Для определения внутренних напряжений в покрытии воспользовались методом гибкого катода в соответствии с ГОСТ 9.302-88.

*Результаты исследования***.** Разработан состав электролита-коллоида и режимы электроосаждения композитных покрытий никель — наноструктурный диборид циркония. Проведен анализ физико-механических свойств (микротвердость, износостойкость и внутренние напряжения) полученных композитных электрохимических покрытий. Сформулированы рекомендации по использованию разработанного электролита и нанесению композитного покрытия на детали машин для их поверхностного упрочнения.

Обсуждение и заключение. КЭП $Ni\text{–}ZrB_2$ имеет высокую микротвердость (10–11 гПа при нагрузке на индентор 100 г), что превышает микротвердость чистого никеля в 1,5–2 раза. При возрастании микротвердости снижаются внутренние напряжения КЭП $Ni\text{–}ZrB_2$. Предлагаемые покрытия сравнивались с хромовыми, осаждаемыми из эко-

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$Ni–ZrB_2$ CEC is 2–5 times higher than that of chromium coatings. Thus, instead of chromic coatings, it is recommended to use the proposed composition for surface hardening of parts of the specialty machinery and industrial equipment.

Keywords: electrolyte, composite electrochemical coating, nickel, zirconium boride, microhardness, wear resistance, internal stresses.

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логически опасных электролитов. Износостойкость КЭП $Ni–ZrB_2$ в 2–5 раз больше, чем у хромовых покрытий. Таким образом, рекомендуется вместо хромовых покрытий использовать предлагаемый состав для поверхностного упрочнения деталей специальной техники и промышленного оборудования.

Ключевые слова: электролит, композитное электрохимическое покрытие, никель, диборид циркония, микротвердость, износостойкость, внутренние напряжения.

Образец для цитирования: Особенности формирования композитных электрохимических покрытий на основе никеля и наноструктурного диборида циркония / Л. А. Дегтярь [и др.] // Вестник Дон. гос. техн. ун-та. — 2019. — Т. 19, № 1. — С. 31–37 https://doi.org/10.23947/1992-5980-2019-19-1-31-37

Introduction. Composite materials are widely used to strengthen the surface of typical nodes and parts of the petroleum-gas field, processing and other industrial equipment. As a result of a combination (composition) of dissimilar substances, a new material appears whose characteristics differ from the properties of its components [1].

To improve the performance properties of electroplating, particulate fillers are introduced into the solution. Thus, a composite electrochemical plating (CEP) with a matrix of the base metal is obtained. Precisely the creation of the CEP is one of the current areas of modern electroplating. To obtain the CEP, dispersed particles of various sizes and types from electrolytes codeposit simultaneously with metals. These particles are included in the coating; they improve drastically the operational properties (hardness, wear resistance, corrosion resistance) of the coatings, and give them new qualities (anti-friction, magnetic, catalytic).

Various technologies of producing nickel-based CEP [2–5] are known. The selection of technique is determined by the operating conditions of the composite coating (CC). At this, wealth of fillers for obtaining coatings with specific performance properties complicates the choice of dispersed particles [3, 5–9]. In [2, 3, 6–8, 10], the physicomechanical properties of various nickel-based CC with particulate fillers (for example, nanocarbon material taunite, aluminum oxide, silicon carbide, graphite, etc.) are considered. In the mentioned works, in particular, it was noted what determines the structural and morphological characteristics of the CEP [7–9], their resistance to corrosion [7, 11], the coefficient of sliding friction [8], heat resistance, microhardness, and other properties [11] . Conditions and modes of CC deposition, composition of electrolytes, nature and degree of fineness of powder or other additive materials are mentioned as determining factors. The use of micropowders with a grain size of more than 1 μm and ultrafine powders or suspensions can change the nature of the codeposition of particles with the excreted metal, and, therefore, the characteristics of the CEP [9].

The use of micropowders with a grain size of more than 1 μm and ultra-disperse powders or suspensions can change the nature of the codeposition of particles with the excreted metal, and, consequently, the characteristics of the CEP [9]. During the dispersion phase, the properties of electrolytes and coatings from them gain a number of advantages [12, 13, 14] critical from the point of view of the applicability.

Nickel CC obtained from electrolytes with additives based on zirconium dioxide to improve surface morphology, microhardness, heat resistance and other properties [5, 15] are of interest. Zirconium boride (ZrB_2) as an additive to electrolytes for obtaining CC was not studied. However, it is known that zirconium and boron compounds improve drastically the mechanical and processing properties of electroplating [16]. . This work objective is to study the physicomechanical properties of the CEP based on nickel and nanostructured ZrB_2, as well as to determine the optimal conditions for its deposition.

Materials and Methods. Reagents of *Aldrich* Company and distilled water were used for the fabrication of electrolytes. The content of ZrB_2 in the coating was determined by the weight (gravimetric) method. The CEP microhardness was measured with PMT-3 microhardness tester. For this purpose, samples with the layer thickness of 30 μm were used under the indentation load of 100 g.

The current output of the coatings was obtained using a copper coulometer. The wear resistance of the coatings was determined on a three-ball friction machine. Samples were tested under dry friction modes and with the use of 3% RV coolant [17]. The wear resistance of the CEP was investigated as follows. The balls of steel ShKh 15 with the area of 0.05 dm^2 were covered with 30 μm thickness coating. Washers of St 45 steel served as a counterbody. The wear scat diameter was measured using MIR-3 TU3-3.1954-86 microscope. To determine the internal stresses in the coating, we used the flexible cathode method in accordance with GOST 9.302-88.

Research Results. The electroplating process and some features of the CEC with ultradisperse powder (UDP) ZrB_2 (0.04–0.06 µm) are studied. The creation of nickel-based CEC with certain physical and mechanical characteristics requires consideration of several factors. In this case, first and foremost, the challenge of the interaction of the dispersed phase, introduced earlier into the electrolyte, and colloid particles formed in the solution under the electrolysis or in the preparation of electrolyte is considered [12, 13].

For the studies, sulfate-chloride nickel plating electrolyte-colloid of the following composition was used, g/l: nickel sulfate – 250, nickel chloride – 60, α-aminoacetic acid – 20. ZrB_2 concentration varied from 1 to 60 g/l.

The optimal electrolysis conditions in the presence of ZrB_2 UDP are specified. For this purpose, ranges of the cathode current density and the electrolyte pH, in which the CEC of good quality is formed, are determined. In the electrolyte composition under study, α-aminoacetic acid was present as an additive agent. Its maximum buffer properties were shown at pH 2–2.5: in this case, light matte coatings of good quality were formed. The presence of an effective additive agent in the electrolyte allows for electrolysis at the high cathode current density.

If ZrB_2, is absent in the electrolyte, then the limiting value of the cathode current density is 2 A/dm^2. When this value is exceeded, a dark contour is formed on the test samples. This indicates alkalization in the cathode layer, the formation of nickel hydroxide in a coarsely dispersed form, and its inclusion in the coating [12]. In this case, the physicomechanical properties of the precipitation deteriorate, their fragility increases, and the coating cracks along the edges of the cathode.

The introduction of ZrB_2 UDP in the electrolyte affects drastically the limiting value of the current density. Good precipitates are formed above the permissible current density characteristic of the electrolyte under study without adding ZrB_2. In the experiments, coatings without a dark contour were obtained at 3; 4; 6 and 8 A/dm^2. At the current density above 4 A/dm^2, dendrites were formed at the corners of the cathode. Their appearance means that the delivery of nickel ions is the limiting stage of the process [12]. The concentration polarization is eliminated under increasing the concentration of nickel salts in the electrolyte. At the nickel sulfate concentration of 300 g/l and high current densities, dendrites were not formed at the cathode.

The change in the current efficiency of the coating depending on the cathode current density is due to the appearance of dendrites at the cathode. With increasing current density, the current efficiency increases, reaches the maximum value at 3 A/dm^2, and then decreases sharply (Fig. 1).

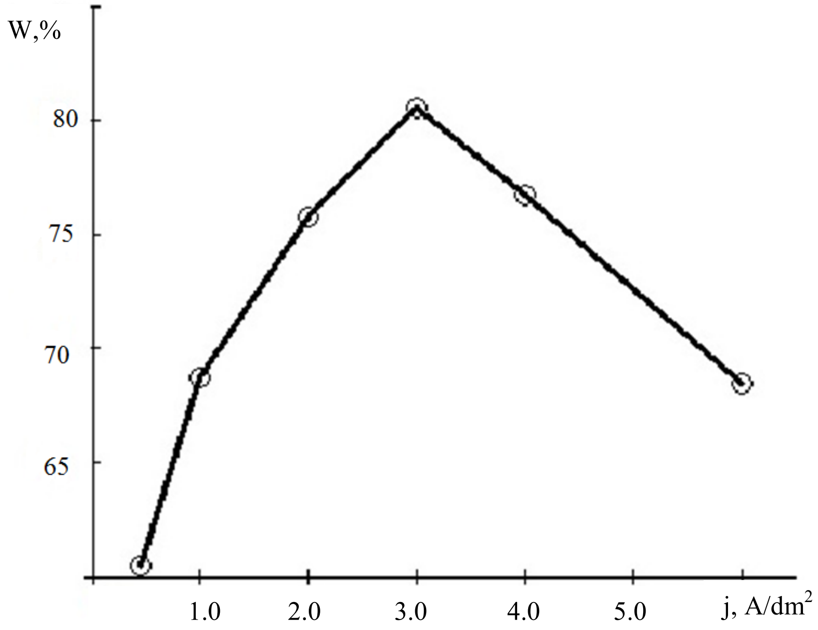

Fig. 1. Dependence of current efficiency of CEC $Ni–ZrB_2$ on cathode current density. Powder concentration in electrolyte is 40 g/l; electrolyte temperature is 50 °C; pH 2

When washing the samples, the dendrites are partially lost; as a result, the current output values of the coating decrease at the current density above 4 A/dm^2. The effect of increasing the upper value of the current density in the presence of ZrB_2 can be explained by the high degree of its dispersion. The particle size of the powder is comparable with the thickness of the electrical double layer, therefore, when particles move near the cathode surface, they can fall into the double layer zone. In this case, colloidal nickel compounds are involved in the cathode layer. They are formed in the electrolyte under the preparation and electrodeposition, and they prevent sudden alkalization in the process of electrolysis.

The buffer capacity of the electrolyte remains almost unchanged with the addition of ZrB_2. pH in the cathode layer seems to decrease due to the addition of new portions of ZrB_2 from the electrolyte volume. The probability of this phenomenon is confirmed by the fact that the upper value of the operating current density does not increase in the presence of ZrB_2 micropowders (5 μm) in the electrolyte of similar composition.

The optimal concentration of ZrB_2 in the electrolyte was determined from its content in the coating and from the microhardness values of the CEC. It was established that in the CEC under study, the content of ZrB_2 reached maximum (1.1 wt.%) when its concentration in the electrolyte was 40–60 g/l. With an increase in the cathode current density at all the studied concentrations of ZrB_2 additive into the electrolyte, the content of ZrB2 in the CEC increases to the maximum limit (1.1 wt.%). CEC with stable microhardness characteristics is formed when ZrB_2 concentration in electrolyte is 30–40 g/l.

For production and cost reasons, the powder concentration should not exceed 40 kg/m^3. Otherwise:

- mixing efficiency of the electrolyte decreases;
- it is difficult to exclude the presence of particles in the workpiece surface areas which are hard-to-reach for the electrolyte circulation;
- quality of coatings deteriorates.

The dependence of the ZrB_2 content in the CEC on its concentration in the electrolyte is considered. It is shown that when the concentration of ZrB_2 particles is 10 g/l with the grain size of 2–9 μm, the distance between the ZrB_2 particles in the coating is approximately 1/3 of the distance between the particles in the electrolyte. As the ZrB_2 concentration increases by 10–20 times, the distance between the particles in the electrolyte decreases, and the coating remains almost unchanged. The concentration of ZrB_2 in the electrolyte can be significantly reduced, and the content of particles in the coating will not decrease. Thus, the use of electrolytes with a high concentration of ZrB_2 is impractical, since the maximum content of ZrB_2 in the coating is reached at its lower concentration in the electrolyte.

The microhardness of the CEC with ZrB_2 rises with increasing the powder concentration in the electrolyte. It reaches its highest value (11 GPa) at the ZrB_2 UDP concentration of 50–60 g/l and the current density of 5–6 A/dm^2. At low current densities (0.5 A/dm^2 and 1 A/dm^2), the microhardness gradually grows with increasing the concentration of the second phase. At the current densities above 2 A/dm^2, the microhardness of the CEC increases sharply, and already at the ZrB_2 concentration of 1 g/l it exceeds the microhardness of pure nickel by 1.5–2 times.

The increase in the CEC microhardness in the presence of the ultradisperse second phase is explained not only by the inclusion of solid and colloidal nickel particles in the coating, but also by a decrease in the crystalline grain at high current densities, as well as by the dispersion hardening of the nickel matrix. There is an opinion that a necessary condition for the dispersion hardening of the coating is the occurrence of ultrafine particles in it, which prevent recrystallization and the formation of coarse grains [18]. On the one hand, submicrometer-sized particles inhibit the growth of crystal grains; on the other hand, they form agglomerates in the electrolyte and cannot penetrate into in the coating as separate particles. Evidently, some part of the second phase is included in the sediment (CEC) in the form of individual particles and stimulates the formation of a finely-crystalline structure of a nickel matrix with high hardness. The probability of this phenomenon cannot be denied if for no reason than because particles of the same nature, differing in the dispersion degree, exhibit different ability to increase the microhardness of the CEC.

The possibility of obtaining CEC with a low content of the second phase and high hardness takes on particular importance for increasing the wear resistance of the surface, since, in this case, the matrix must have specific elasticity. A significant increase in the volumetric content of solid particles causes the fragility of materials. Thus, the use of UDP to obtain wear-resistant coatings is based on the practice. Equally important is the reduction of the consumption of the second phase, and, consequently, the process efficiency.

It is known that the hardness of coatings allows for indirect assertions about some physical and mechanical properties of the CEC. In some cases, it correlates adequately with wear resistance [4]. However, maximum microhardness does not always correspond to high wear resistance. The latter depends on the interaction of various factors of the process, as well as on the specific friction conditions. Hence, the wear resistance of the CEC with ZrB_2 was compared to chrome galvanic coatings traditionally used as wear resistant [19]. At this, both lubricated and unlubricated friction modes were considered. According to the results obtained, the service lifetime of the parts with $Ni – ZrB_2$ coating is 2–5 times longer than that of the parts with hard chrome plating.

Internal stresses of the $Ni–ZrB_2$ composite coatings decrease with an increase in the ZrB_2 concentration in the electrolyte and the coating thickness. Differences in the internal stress values of nickel CEC grow with increasing the sediment thickness (Fig. 2).

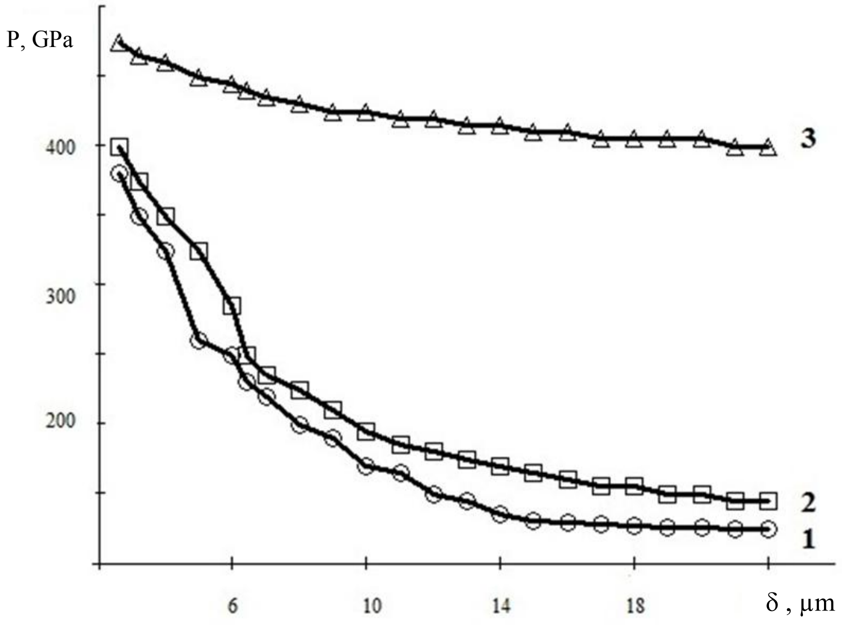

Fig. 2. Dependence of internal stresses on coating thickness and zirconium boride concentration in electrolyte, g/l:
1 — 30; 2 — 5; 3 — without second phase. Cathodic current density is 2 A/dm^2; electrolyte temperature is 50°C; pH 2

For galvanic coatings, the following regularity is peculiar: the finer the crystal grain and the higher the hardness, the greater the value of the internal stresses. In the presence of ZrB_2, the microhardness of the coatings increases sharply, and the internal stresses decrease. To explain this common pattern mismatch, it is necessary to consider the effect of the second-phase particles (particles of ZrB_2 UDP and colloidal particles formed in the electrolyte) on the precipitation hydrogenization ratio. The discharge of the hydrogen ions (and, consequently, a more complete removal of the molecular hydrogen) occurs faster on the second-phase particles than on the surface of nickel. So, the degree of precipitation hydrogenization decreases, which leads to a reduction of the internal voltage of the coating.

Discussion and Conclusions. In summary, the authors of the paper propose the following composition of nickel sulfate-chloride electrolyte-colloid for deposition of the nickel-based CEC and nanostructured ZrB_2, g/l: nickel sulfate - 250; nickel chloride - 60; α-aminoacetic acid - 20; ZrB_2 UDP - 30–40. The electrolysis mode is as follows: cathode current density is 2–4 A/dm^2; electrolyte pH is 2–2.6; temperature is 50° C; it is necessary to mix the electrolyte during the electrolysis process.

The electrolyte composition is developed; and the process condition for the application of the nickel-based CEC with the addition of ZrB_2 is established. The physical and mechanical properties of the obtained $Ni – ZrB_2$ CEC are studied: microhardness, wear resistance, and internal stresses.

CEC has high microhardness (10–11 GPa with the indentation load of 100 g), which is 1.5–2 times higher than the microhardness of pure nickel. The wear resistance is 2–5 times more than that of chrome coatings. As microhardness increases, the internal stresses of the $Ni – ZrB_2$ CEC decrease.

Considering the listed parameters, the proposed method can be used for surface hardening of parts for specialty machinery and industrial equipment.

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