1 Article

The application of X-rays for an electrodeposition of composite coatings with modified structure and properties

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12	Abstract: Experimental studies of X-rays effect on the process of electrochemical deposition of
13	composite coatings are reviewed in the paper emphasizing the X-rays application for modification
14	of microstructural characteristics and mechanical performances of protective coatings. Research is
15	covered the Co/SiO ₂ coatings electrodeposited from aqueous solutions under the effect of X-ray.
16	The results of elaborate investigations of dispersing ability of electrolytes with SiO2- nanoparticles
17	and mass rate of composite coatings Co/SiO2 point to the fact that the method of the electrochemical
18	deposition under the effect of X-rays during electrodeposition is considered as the method which
19	intensifies diffusion in the electrolyte volume and allows to obtain dense, morphology uniform
20	coatings with increased hardness and improved adhesion. It is shown that exposure of electro-
21	chemical system with X-rays during the Co/SiO2 coatings electrodepositing occurs the orienting
22	effect on the growth of crystal grains.
23	Keywords: composite coatings; X-rays irradiation; radiolysis; nanoparticles; structure; properties

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1. Introduction

Electrochemical deposited composite coatings with included nanoparticles occupy an important place among various types of protective coatings and thin nanocomposite films. The method of an electrochemical deposition is widely used in different fields of industry for objects of various sizes and it greatly cheaper compare with different methods of covering such as plasma-assisted chemical vapor deposition [1–5], the ion implantation [6,7], the ion-beam [8,9] and magnetron sputtering [10,11], or another plasma techniques and chemical methods [12,13,14] which can be used primarily for covering small objects.

Formation of composite coatings with included nanoparticles with required surface morphology, microstructure and excellent properties in different cases depends on the composition of a reaction medium, electrodeposition conditions and modes and in most instances, it depends on the sedimentation stability of nanoparticles in an electrolyte. The most common employed method for supporting the sedimentation stability of nanoparticles is using different additives prevented coagulation of nanoparticles. The most visible disadvantage of employing additives is a change in the chemical composition of electrolytes leading to narrowing a range of working current densities of electrodeposition. Hence, factors have been described earlier contribute to form composite coatings with deterioration of qualities and properties [15-17].

Alongside with multipurpose procedures of deposition by electrochemical

reduction on conductivity substrates from electrolytes with insoluble suspended nanoparticles the effect of external factors on the formation of coatings with required properties are widely applied [18, 19].

The well-known method of deposition composite coatings with included nanoparticles by electrochemical reduction from suspended aqueous solution under ultrasonic vibrations. The application of ultrasonic field for suspending liquid and manufacturing of new material has a long history and has revealed broad prospects in the microstructure control and performance improvement for composite metals and alloys electrochemical deposited. Despite abundant researches on the sedimentation stability of electrolytes with nanoparticles with help of the ultrasonic technologies, the refinement mechanism of ultrasonic treatment during the electrodeposition process is still controversial. Particularly noteworthy the surprising laboriousness and increasing energy consumption with the implementation of ultrasound into the technological process. This implementation is associated with the need to prevent a formation of standing sound waves caused by reflecting ultrasonic waves transferring back and forth from the surface of a cathode. The formation of standing waves also causes a decrease in diffusion to the cathode surface and promotes the accumulation of nanoparticles under the source of ultrasonic vibrations. It is obvious that accumulation of nanoparticles near the ultrasonic vibrations source leads to coagulation of insoluble particles and promotes to reduction of dispersing ability and to concomitant deterioration of coatings operational properties and subsequently to decrease in the economic efficiency of the technological process [20].

In order to overcome indicated limitations, the present research work focuses on considering the method of the electrodeposition of composite coatings with nanoparticles under the effect of X-rays [21–23]. The main mechanisms of X-ray irradiation of suspended electrolytes base on formation of active and mobile radiolysis products in volume of an irradiated electrolyte which contribute to natural mixing of electrolyte, lead to increase its dispersing ability and to intensification of the electrodeposition. This makes it possible to form dense, uniform in thickness, fine-grained coatings with improved operational properties [24].

Of particular interest are Co- coatings electroplated that use in the manufacture of reflectors, mirrors, jewelry, due to its high abrasion resistance and the lower cost enables use it in more applications.

The main aim of this study is the experimental investigation of X-rays effect on the process of electrochemical deposition of composite coatings Co/SiO₂ underlining X-rays application for modification of microstructural characteristics and mechanical performances of protective electrodeposited coatings.

2. Experimental

 Co/SiO₂ composite coatings have been electrodeposited from the additive-free sulfate electrolyte with following composition: $CoSO_4 \cdot 7H_2O - 200 \text{ g/dm3}$, $H_3BO_3 - 15 \text{ g/dm}^3$ and NaCl $- 10 \text{ g/dm}^3$. The concentration of SiO₂ nanoparticles was 0.5 g/dm3 and 1 g/dm3. Deposition was performed with a preliminary reverse during 5 minutes. The low-carbon steel with a carbon content of 0.05-0.12% was used as substrates. Before plating all substrates were polish with help of polishing machine BAINPOL VT Auto for preparing samples with average roughness 0,05µm. Coatings have been electrode-posited for 1h in the X-ray field (Peks =100 R/h) at controlled temperature of 23 °C [25].

Electrodeposition has been carried out in the thermostatic bath with two cells of 100 ml (figure 1). One cell was used for depositing coating under the effect of X-rays, exposing the electrolyte directly. Another sell was used for depositing reference coatings.



Figure 1. Schematic illustration of plating bath under effect of X-ray (a) cell for deposition of reference sample; (b) cell
for deposition of irradiated sample

97	Current densities were chosen in the range from 1 to 3 A/dm ² . The coatings formed in the
98	X-ray field, irradiated samples (Irr), and coatings formed without exposing with X-rays,
99	reference samples (Ref) were compared to estimate its structural variations. The thickness
100	was measured with a MTTs-3 meter (Minsk, Belarus). The mass gain and thickness of
101	Co/SiO_2 coating were determined as the average of 10 measurements (10 samples). The
102	surface morphology and elemental composition were studied using JSM-5610 LV scan-
103	ning electron microscope equipped with the EDX JED-2201 chemical analysis system
104	(JEOL, Akishima, Japan). Accelerating voltage was 20 keV. The estimated depth range of
105	electrons does not exceed 0.98 μ m. The energy resolution of the detector is about 0.137
106	keV. The phase Co/SiO2 composition was studied by X-ray diffraction (XRD) with help of
107	DRON-3M diffractometer in the CuK α radiation (Burevestnik, St. Petersburg, Russia).
108	Microhardness of the coating surface was measured with a CASON-59 HV microhardness
109	tester (Jinan Kason Testing Equipment Co, Jinan City, Shandong Province, China). The
110	adhesion of Co/SiO2 coatings was investigated by the scratch method based on applying
111	of a scratch grid on the surface of testing coatings. The adhesion tests were carried out in
112	accordance with the Interstate standard [26]. The dispersing ability of electrolytes was
113	studied with help of a Moler's cell [27]. The Moler's cell has a rectangular shape with a
114	collapsible cathode block. The cathode block consists of 10 indifferent aluminum cath-
115	odes. The main characteristic of the Moler's cell is that the cathode space is separated from
116	the anode space with a non-conductive partition. Between the partition and one of the
117	side bath walls must be a space of 1or 2 mm. In this case, the slight gap is a non-polarizable
118	anode that does not cause concentration changes in the solution. The advantage of the

Moler's cell is that the cathodic current distribution in it does not depend on the shape and does not depend on the anode location.

Value of dispersing ability is computed by measuring of mass distribution on all Al-cathodes according the Unified System of corrosion and ageing protection. Galvanic coating. Designation of dispersing ability of electrolytes during the creation of coverings [27]. The relative error of the method did not exceed 5 %.

3. Results

The Figure 1 shows the SEM images of surface morphology and cross sections of Co/SiO₂ composite coatings formed under the effect of X-rays and references sample formed from electrolytes with SiO₂ concentration of 0.5 g/dm³. As shown in Figure 1a, the reference sample formed without irradiation are characterized by needle-shaped crystallites normal oriented to the substrate plane and by coarse and inhomogeneous grains oriented lengthwise (parallel) to the substrate plane. This structure is typical for a cobalt microstructure [24]. By contrast, as can be seen from the Figure 1b, the irradiated samples differ from geometry surface with crystalline grains only parallel directed to the substrate. The observed differences in the surface morphology of exposed and reference samples caused by destruction of primary structure of the irradiated electrolyte and radiolysis transformations under the effect of X-rays.



(a)

(b)

Figure 2. SEM images of surface and cross sections of Co/SiO₂ formed at 2 A/dm² current density from electrolyte with SiO₂
 nanoparticles of 0.5 g/dm³ (a) reference sample; (b) irradiated sample.

139	Compare the Figure 2 and the Figure 3, it is obvious that well visible normal
140	oriented grains on the references samples gradually become smaller (fig.3.a) and almost
141	entirely eliminate on the surface of irradiated Co/SiO2 coatings (Fig.3.b). It should be
142	noted that coatings demonstrated on the figure 2 were formed from electrolyte with
143	concentration of SiO ₂ nanoparticles equaled 1 g/dm ³ .



Figure 3. SEM images of surface of Co/SiO₂ formed at 2 A/dm² current density from electrolyte with SiO₂ nanoparticles of 1 g/dm³ (**a**) reference sample; (**b**) irradiated sample.

It should be noted that Co/SiO₂ coatings deposited from electrolyte with SiO₂ nanoparticles of 1 g/dm^3 characterize with more developed surface geometry in comparison with coatings which are deposited from the electrolyte with SiO₂ nanoparticles of 0.5 g/dm^3 So, it can be suggested that morphology and structure of Co/SiO₂ coatings affected by SiO₂ concentration. However it is clearly, that reference samples contain two types of crystalline grains, normal and parallel oriented to the substrate plane.

Comparative analysis of irradiated and reference cross sections of composite coatings Co/SiO₂ (Figure 2) allows to conclude that the rate of deposition under the effect of X-radiation is significantly higher. Moreover nonporous dense coatings are formed in X-rays field.

So, unlike the traditional electrodeposition process, the interfusion of the electrolyte with radical particles in X-ray irradiated electrolyte prevents the formation of cohesive contacts between SiO₂ nanoparticles and reduces the likelihood of coagulation and sedimentation. Due to this, the SiO₂ nanoparticles are transferred to the cathode, where they are overgrown with the reduced metal, forming their own finely dispersed agglomerate with an increased content of nanoparticles and with improved operational properties [21].

The tendencies to change the geometry surface of irradiated coatings Co/SiO₂ positively correlate with data of study mass rate per unit area and thickness per time. Comparative relationships between the results of mass rate study at different current densities of composite coatings Co/SiO₂ from electrolytes with different concentrations of nanoparticles are presented in the Figure 4.



Figure 4. Mass rate in a unit area of the Co/SiO₂ coatings formed at different current densities of 1–3 A/dm²: 1 – reference sample, formed from electrolytes with 0.5 g/dm³; 2 – reference sample, formed from electrolytes with 1 g/dm³; 3 – irradiated sample, formed from electrolytes with 0.5 g/dm³; 4 – irradiated sample, formed from electrolytes with 1 g/dm³.

As well seen, the mass rate per unit area increases with increasing of current density corresponding to Faraday's laws. It is evident, the mass rate of irradiated samples is higher than references samples. Dependencies of thickness over current densities are presented on the Figure 5. The analysis shows the correlation with investigation of mass gain . On one side, increasing in mass rate per unit area of samples deposited in X-rays is caused by increasing in number of ions transferred to a cathode surface through the diffusion layer due to solution radiolysis. On the other hand is caused by change of pH of electrolyte [27]. In particular, the pH reduction prevents formation of coagulants of hydroxide compounds. Hence lack of their absorption on the cathode surface promotes to formation of more perfect, compact and non-porous coatings than reference coatings [24]. From another side, an increase in mass rate of Co/SiO₂ coatings indicates the increment of number of nanoparticles included into the base metal matrix due to increasing the dispersing ability of electrolytes.



Figure 5. Thickness of Co/SiO₂ formed at different current densities: *1* – reference sample formed from electrolytes with 0.5 g/dm³; 2 – irradiated sample formed from electrolytes with 0.5 g/dm³;

3 – reference sample formed from electrolytes with 1 g/dm^3 ; 4 – irradiated sample formed from electrolytes with 1 g/dm^3 .

Figure 6 illustrates the dispersing ability of electrolytes used for deposition Co/SiO₂ coatings with concentration of SiO₂ nanoparticles in the electrolytes equaled 0.5 g/dm³ and 1 g/dm³. It is well visible in the Figure 6 that X-ray irradiation of electrolytes leads to the increase in the dispersing ability of electrolytes with suspended nanoparticles. As well known that the dispersing ability of electrolytes shows the uniform current distribution over the cathode surface causing the formation of homogeneous coatings. Thus, X-rays irradiation of electrolytes promotes to the formation compact Co/SiO₂ coatings.



Figure 6. The dispersing ability of irradiated and nonirradiated electrolytes with different concentration of SiO₂ nanoparticles.

As can be seen in the Figure 6 the addition in concentration of nanoparticles in electrolytes reduces its dispersing ability. Hence, obtained data are allowed to suggest that since SiO₂ nanoparticles have a high ability to aggregate in aqueous solutions, X-ray irradiation degrade nanoparticles clusters due to an increase in the dispersing ability of electrolytes and contributes to the formation of composite coatings with even distributed nanoparticles with improved structure and properties.

Figure 7 presents the results of elemental analysis of composite Co/SiO₂ coatings. It shows that the effect of X-ray on the electrochemical cell during electrodeposition from electrolyte with nanoparticles of SiO₂ promotes to formation of composite coatings with a bit more SiO₂concentration compare with reference samples. It should be mentioned that difference in SiO₂ content is less than 0.1 % that why we can consider the data only as qualitative characteristic.



nanoparticles destroys nanoparticles clusters due to increasing in the dispersing ability of electrolytes. Obtained data correlates with investigation of mass gain, thickness and dispersing ability. Worth paying attention to the point of Fe content in the Co/SiO₂. The Fe content in the coatings formed from electrolyte with concentration of SiO₂ of 1 g/dm³ is less than in the coatings formed from electrolyte with concentration of SiO₂ of 0.5 g/dm³. The reducing of Fe content can be caused by rising of the thickness and mass gain of irradiated coatings. As for Fe content in the coatings it can appeer due to the preliminary reverse of current during 5 minutes before the electrodeoisition and some amount oFe ions transfered to the solution after reverse.

Figure 9 demonstrates the XRD patterns of reference Co-coatings, Co-coatings obtained in X-rays, Co/SiO₂ obtained from electrolytes with 0,5 and 1 g/dm² under irradiation and whithout it. It is seen, that all investigated samples have simple phase composition, containg onle Co-phase and oxide phase CoFe₂O₄. The last has appeared due to the preliminary reverse of current. Accoding to a little amount of SiO₂ there are no any peaks from SiO₂. But it is well visible that addition of some SiO₂ into an electrolyte leads to formation of CoSiO₂ coatings with modified structered characterizing with crystalline orientation (110). It should be noted that intensity of the main peaks of irradiated coatings is higher than intensity of reference sapmles. This proves the suggestion that X-rays irradiation of electrolytes during electrodeposition promotes to orienting effect on the formation of crystal grains of the Co/SiO2 coatings due radiation transformation in irradiated electrolyte.

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Figure 9-XRD patterns of CoSiO₂ coatings obtained in X-rays Reference Co-coating without SiO₂, 2- Co-coating, formed under irradiation; 3- CoSiO₂ deposited from the bath with SiO₂ concentration of 0.5g/dm³, 4- CoSiO₂ deposited from the bath with SiO₂ concentration of 0.5g/dm³ in the X-ray, 5– CoSiO₂ deposited from the bath with SiO₂ concentration of 1 g/dm3, 6- CoSiO₂ deposited from the bath with SiO₂ concentration of 1 g/dm³ in the X-ray.

e in the structure of composite coatings Co/SiO₂ generated the interest to investigate any operational proterties.

As seen in Figure 10 microhardness of irradiated samples markedly increased in comparison with reference samples. In particular the difference in microhardness of reference and irradiated Co/SiO₂ coatings, formed from the electrolyte with SiO₂ of 0,5 % is around 7%.

Finally, the adhesion of Co/SiO₂ coatings was investigated by the scratch method based on applying of a scratch grid on the surface of testing coatings. It was found that all irradiated composite coatings Co/SiO2 have excellent adhesion, equaled 1, according to the scale of scratch method (1 on a 4 scale, where 1 is the best rating). Unlike, reference

samples have adhesion only 2. The structural changes in matter can occur via primary radiolysis effects, which occur as a result of the adsorption radiation by aqueous electrolyte. Hence, these experimental results can be explained from the point of view of radiation chemistry and radiolysis of aqueous electrolytes have been admitted early.



Figure 6. Microhardness of irradiated (Irr) and non-irradiated (Ref) Co/SiO₂ formed at current densities of 1–3 A/dm² from electrolyte with SiO₂ nanoparticles of 0.5 g/dm³ (**a**) and 1 g/dm³ (**b**).

4. Conclusions

The results of elaborate investigations of the effect of X-rays on electrodepositing of composite coatings Co/SiO₂ from aqueous solution demonstrate increase in dispersing ability of electrolytes with SiO₂ nanoparticles, mass gain and thickness per time under irradiation due to the intensification of diffusion in the electrolyte by products of radiolysis.

It is shown that exposure of electrochemical system with X-rays during the Co/SiO₂ coatings electrodeposition occurs the orienting effect on the growth of crystal grains and allows to obtain dense, morphology uniform coatings with increased hardness and improved adhesion.

So, the authors have found enough evidence to support that the method of the electrodeposition of composite coatings under the effect of X-rays makes it possible to achieve the technical result that consists of increasing the dispersing ability of electrolytes containing nanoparticles, reducing aggregation, and increasing the sedimentation stability of nanoparticles, which leads to modification of the microstructure and enhancement of performances of properties composite coatings with included nanoparticles SiO₂.

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