

Protective Coatings for the Elements of Ships Power Plants which Use Sea Water

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Abstract : In this paper we observe the protective coatings carbon for steel, aluminium and titanium alloys were obtained by plasma electrolytic oxidation (PEO) under unipolar and bipolar conditions. The anticorrosion properties and the thermal stability of the surface layers were studied by electrochemical impedance spectroscopy and potentiodynamic polarization. It was found that the application of the bipolar PEO mode enables one to synthesize the surface layers that possess enhanced anticorrosion and mechanical properties. results of research of antiscale PEO - coatings for marine power equipment are presented. The combined method of prevention of corrosion and scale formation was tested at the industrial plants of Russian Shipyard "Zvezda".

Key words : Seawater, Ships power plants, Coatings

1. Introduction

Plasma electrolyte oxidation (PEO) - one of the methods of surface treatment and strengthening of metals and alloys, which originates in traditional anodic treatment and, accordingly, is among the electrochemical processes. PEO allows to obtain multifunctional coatings, including wearproof, corrosion-proof, heatproof, insulating and decorative. PEO technology is relatively well perfected for the group of valve metals and alloys. The kind of plasma electrolyte oxidation is participation in process of forming of the microdischarges which have rather essential and specific (thermal, plasmachemical etc.) influence on forming surface and electrolyte, and as a result, the composition and the structure and, consequently, properties of

the obtained oxide layers essentially differ both from the natural oxide films (which are formed dew to metal oxidation in air) and from films obtained with usual anodic treatment.

Modern current supplies allow to fulfil the anodic-cathode (bipolar) modes of PEO, and at that it is possible to achieve the change of growth rate, increase of protective properties of the surface layers and decrease of power intensity of PEO process by control of repetition frequency of positive and negative current impulses and change of ratio of its amplitude and duration[1,2]. In the interval of cathodic polarization there occurs the reorganization of double electrical layers (Helmholtz and Gui-Chapman layers) which decrease the ionic transport and probability of penetration of the

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oxidizing reagents to the treated electrode. Moreover, alongside with the disorder transformation of the double electrical layer of the electrolyte at the cathodic period there occur changes of the double electrical layer in the material of the coating (the area of the spatial charge). By virtue of this reasons at the following displacement at the anode more intense plasma charges are realized. At that the large quantity of elements of the electrolyte comes upon the plasma, and it promotes the transition of metastable compounds of the coating to the stable state (for example, in $\alpha\text{-Al}_2\text{O}_3$ for the aluminum oxide). This reduces to the solidity enhancing of the forming surface layers, effects on its morphology and changes its anticorrosion properties.

Owing to this fact it is expedient to obtain the experiments directed to the researching of the influence of modes of forming on the mechanical and electromechanical properties of PEO-layers on such important constructional materials as aluminium, titanium and steel the later material is intractable by the electrochemical method.

2. Results and Discussion

2.1 PEO-coatings on low-carbon steel

The problem of obtaining the protective coatings, which could preserve the surface from different types of corrosion, on steel is actual for the shipbuilding, mechanical engineering and other productive industries. However this material is one of the electrochemically intractable. In a few researches at steel oxidation the alkaline solutions of electrolytes on base of natrium aluminate with liquid glass[7] or natrium hydroxide are applied[20]. Also there are methods of steel oxidation in electrolytes which contain liquid glass and potassium hydroxide[12]. In work[6] there was offered a procedure of obtaining of protective anticorrosion coatings on steel my PEO-method in

the electrolyte on base of natrium aluminate, carbonate and phosphate with potassium hexacyanoferrate (II) (yellow prussiate of potash). In the electrolyte of mentioned composition the results were encouraging for the practical use.

The purpose of the research was study of influence of bipolar mode of PEO on electrochemical properties of coatings on steel formed in solution of the electrolyte which contains liquid glass and natrium carbonate. The samples, which were exposed to oxidation, were made of low-carbon steel St3 and have shape of rectangular plates with size 40'8'1 mm[11, 12] .

At realization of experimental search of optimal composition of electrolytes with purpose of obtaining of surface layers on steel with improved anticorrosion properties we used water solutions which contain natrium carbonate Na_2CO_3 (concentration 20 g/l) and liquid glass $m\text{Na}_2\text{O} \cdot n\text{SiO}_2$ (module $m/n=2$) (concentration 30 g/l). Plasma electrolyte oxidation was accomplished both in unipolar mode and in bipolar mode. Time of forming of the coating was 10 minutes.

According to the results of x-ray phase analysis, the obtained coatings on steel are X-ray amorphous (Figure 1, a). For determination of composition of the X-ray amorphous stage there was realized the thermal annealing of the coating up to temperature of 400 and 1000°C in vacuum (for the exclusion of processes of additional oxidation of the coating's material and oxidation of the steel basis). After the annealing up to 400°C (Fig. 1, b) in the composition of the coating there were discovered traces of the ferric oxide (III), and after the annealing up to 1000°C (Figure 1, c) the crystallization of the X-ray amorphous stage with forming of oxide Fe_2O_3 and silicon dioxide SiO_2 took place.

For specification of composition of the coating the research[11] of allocation of elements both on the thickness and on the surface of the coating

were fulfilled by the method of X-ray spectrum microprobe analysis. In the composition of the coating the following elements were detected: Na, Fe, Si, C and O.

The analysis of allocation of elements on the thickness of coating allows to determine that in the outward layer of the surface concentration of silicon (31,8 at.%) is higher than in the inner one (29,5 at.%), which is conditioned by mechanism of implantation of this element from electrolyte and, accordingly, by its more active participation in forming of the outward part of the coating.

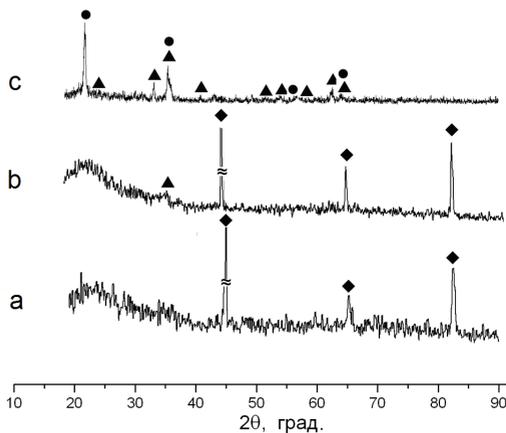


Figure 1: The roentgenogram of the sample made of steel St3 with PEO-coating: a - before the temperature effect; b - after the heating up to 400°C during 1 h; c - after the heating up to 1000°C during 1 h; ◆ - Fe; ▲ - Fe₂O₃; ● - SiO₂

Furthermore, in the outward layer of the coating (i. e. with taking into consideration the diameter of the electronic probe stain not more than 1.5 μm from the surface of the layer) there was detected rather high percentage of oxygen (64,3 at.%) and iron (3,7 at.%).

Concentration of sodium in the composition of coating is insignificant and is equal 0,7% (at.). Probably, sodium oxide, which is originally formed in a coating in PEO-process, then transfers to the electrolyte solution due to high solubility.

Allocation on the surface of elements which are the part of the coating has a discontinuous disposition and reflects the clustered construction of the formed PEO-layers. According to the microprobe analysis data, concentration of the elements on the surface is following, % (at.): Na - 0,3...3,7; Si - 20,2...29,8; Fe - 1,0...9,3; C - 8,0...20,0; O - 49,8...69,5. For the confirmation of microprobe analysis data and determination of the chemical state (oxidation rate) of the elements which are the part of the coating in this work we applied the method of X-ray photoelectric spectroscopy

The basic components of the surface layer are oxygen, silicon and carbon. The first two of them are mainly in form of SiO₂, and the evidence of this fact is value of E_{CB} and intensity of tracks [18]. The typical trait of another components is that carbon on the surface of the sample has at the minimum three different states (Figure 2):

1. In composition of aliphatic compounds, $E_{bond} \approx 285$ eV (C-C, C-H - the result of pollution of the surface during preparation of the coating).

2. In composition of adsorbed substances, $E_{bond} = 288,0$ eV (the peak is caused by chemical adsorption on surface of the porous coating of oxygen or oxygenated components with forming of C-O connection). According to data described in [11] the typical atmosphere of residual gases of a thoroughly warmed vacuum system of spectrometer (summary pressure 10^{-9} Torr) consists of mix of H₂, CO, CO₂ (with prevalent concentration of CO) and elements of H₂O. As far as $E_{bond} = 288,0$ eV corresponds to carbon monoxide, it would be logical to explain presence of CO, which is in a vacuum chamber of spectrometer or in the atmosphere (where CO concentration is 0,01-0,9 mg/m³ according to [15]).

1. "Carbonate" carbon, $E_{bond} \approx 291$ eV (most likely, it is in composition of Na₂CO₃, because E_{bond} Na₂CO₃ is close to the corresponding value in

carbonate-ion). However concentration of Na in composition of sodium carbonate is approximately equal to concentration of “carbonate” carbon (and in Na_2CO_3 concentration ratio Na/C is equal to 2). This gives grounds to presume the presence of some quantity of sodium hydrogen carbonate or iron carbonate in a coating.

Small integral intensity of iron peak is an evidence of insignificant concentration of iron carbonate and oxides in a coating, and value E_{CB} Fe2p characterizes conditions of iron which is close to Fe^{3+} . Most probably, mainly iron is in composition of iron oxide Fe_2O_3 .

At comparison of elementary and concentration composition data which were obtained with two independent methods (X-ray spectrum microprobe analysis and X-ray photoelectronic spectroscopy) under the qualitative identity of results, the quantitative discrepancy is notable, though concentration values of basic elements are rather similar.

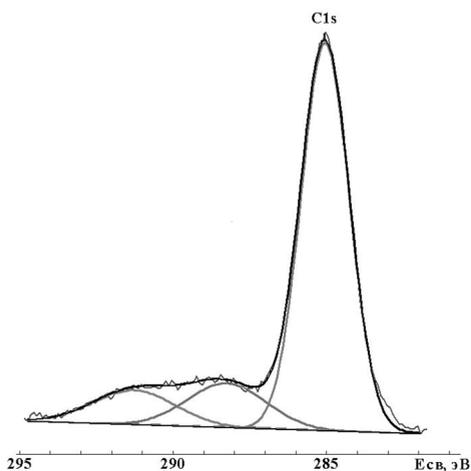


Figure 2: Overview RFES-spectrum of the carbon condition (C1s) in a surface on steel

Some discrepancy of the chemical compound data can be logically explained with clustered structure of the layer and with heterogeneity of its thickness.

Figure 3 shows SEM-picture of the transverse

sample of the obtained coating in steel St3. The thickness of coating is 30 mkm, it consists of light thick layer which contains detached dark-coloured dissemination on its surface.

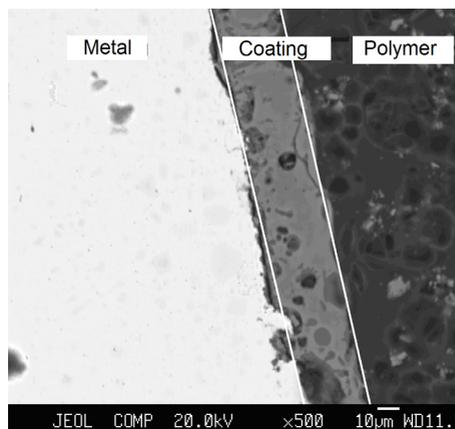


Figure 3: SEM-picture of the transverse side of the sample with the coating on steel St3

Thus, the heterogeneous properties of the layer's thickness are not confirmed with optical microscopy. In view of this fact and taking into consideration the surface's heterogeneity which was detected with X-ray spectral microprobe analysis we can make a conclusion about clustered structure of the studied coating on steel which consists basically from silicon and iron(III). Spatially localized allocation of the sorbed sodium carbonate from electrolyte on the surface (probably, in pores) intensifies heterogeneous nature of oxide PEO-layer.

It is logical to suppose that after the crystallization process due to possible differences of volume of amorphous and crystalline phases in the material of the coating there can appear macrodefects in the form of cracks and chips which appreciably decrease protective anticorrosion properties of PEO-layer which were determined before (Figure 4). The electrochemical impedance measurement data (Figure 4, curve 3) refutes this supposition.

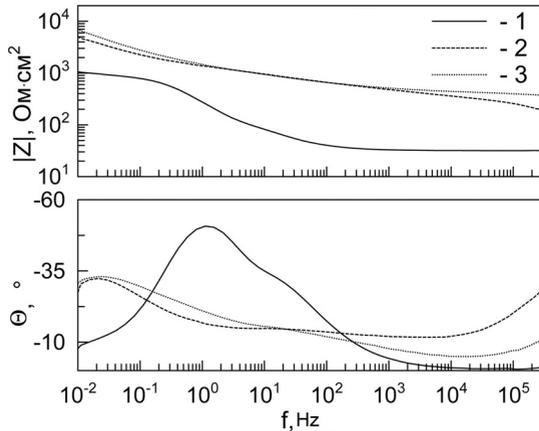


Figure 4: Bode diagrams for the samples of steel St3 made in 3 % solution of NaCl: 1 - samples without coating; 2 - sample with PEO-coating, 3 - sample with PEO-coating after annealing at 200°C

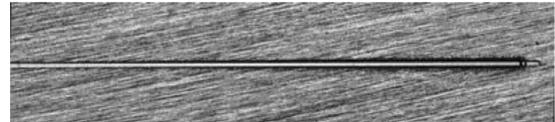
As a result of transfer of amorphous phase to the crystalline phase, which was triggered with warming, the impedance module increases, which in case of electrochemical changes (i. e. changes in the electrolyte solution) is the evidence of increase of anticorrosion properties of the coating on steel, which takes place dew to warming.

At the same time one should note that the value of the modulus of elasticity of the coating E is slightly small in comparison with metal without coating (26,6 and 33,6 GPa correspondingly) which may be explained with porous structure of PEO-layer as far as pores are the phase with minimal elasticity.

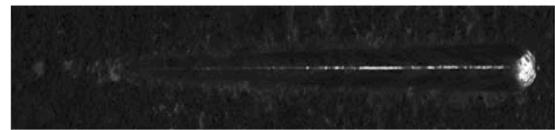
The realized research of the mechanical properties of the surface with method of dynamic microhardometry allows to determine that microhardness of the coating, which was measured with help of the restored print ($H = 11,5$ GPa), is more than 5 times higher than corresponding value of microhardness of the substrate material ($H = 2,1$ GPa).

During the scratch-test the acoustic emission data allowed to determine that indenter reaches the

substrate material at loading 55 N and depth 55 mkm (photographs of the scratches see Figure 10), which completely corresponds to the information about thickness of the coating, which was obtained as a result of the analysis of the optical photographs of the transverse шлиф of the coating (Figure 5).



a



b

Figure 5: Outlook of scratches after scratch-test on samples: a - St3 without coating; weighting effort $N = 5$ H; б - St3 with PEO-coating; weighting effort $N = 105$ H. Velocity of indenter 3 mm/min, line length 5 mm

2.2 PEO-coatings on aluminium

Coatings were formed on the surface of commercially pure aluminum A5N samples. The PEO process was carried out in different regimes: unipolar (potentiodynamic, galvanostatic, potentiostatic) and in an alternating bipolar (potentiodynamic, galvanostatic and combined) modes. Formation time was 5-20 min.

The resulting coating has dark brown color and the thickness of 30 microns. There is an easily removable white layer on the surface of the coating, which consists of the different phases of aluminum hydroxide: $\text{AlO}(\text{OH})$ and $\text{Al}(\text{OH})_3$. Dark dense layer, adjacent to the metal is composed of amorphous phase with inclusions of $\alpha\text{-Al}_2\text{O}_3$.

Coatings, having a typical for the PEO- layers developed surface, yet differ in the extent of

surface heterogeneity depending on the polarization mode. Performed analysis of the surface morphology gave reason to suppose that the formation of films in the bipolar mode which combines an anodic and cathodic pulses in a certain way results in a more uniform and less defective coating. The roughness of the samples decreases during the transition from unipolar to bipolar mode (from $R_a = 380$ nm to $R_a = 65$ nm). Differences in morphological structure (developed surface, porosity, thickness of the porous and dense poreless layers) investigated the coatings obtained in unipolar and bipolar modes entail changes causes the transformation of high-frequency part (10^4 - 10^6 Hz) of the phase angle θ from frequency f dependencies on the impedance spectra presented in Figure 6.

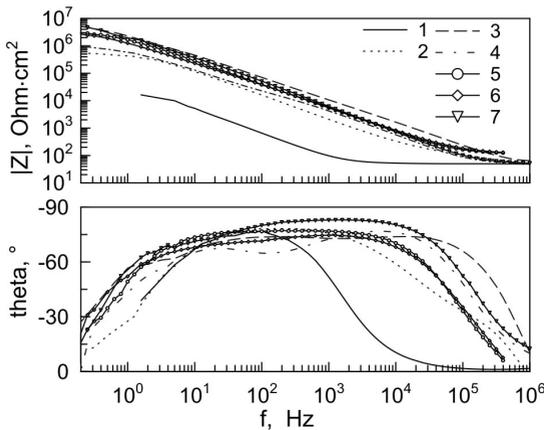


Figure 6: Impedance spectra, obtained for commercially pure aluminium in 3% NaCl: 1- bare metal; 2-7 PEO-coatings formed in different polarization modes

As follows from the analysis of impedance spectra, the formed PEO-coatings are two-layered: have a dense poreless sublayer and a porous surface layer (two inflections on the dependence of the phase angle of frequency), which agrees well with the results of previous studies on PEO-layers. Oxide heterostructures formed on the aluminum,

characterized by a significantly larger (by about three orders of magnitude) impedance modulus ($|Z|_{f \rightarrow 0}$) in comparison with the native oxide, the layers with the highest resistance were formed in a unipolar potentiostatic and bipolar combined modes (Figure 6, curves 7 and 3).

This is consistent with the well-known observations that characterize the potentiostatic mode or potentiostatic oxidation stage as the most optimal conditions for the polarization of insulating (dielectric) layers on metals. In the context of this discussion it should be noted also that the potentiostatic mode, as noted above, is included as a component in the bipolar combined mode. The analysis of the potentiodynamic polarization data obtained in 3% NaCl shows that we can strengthen the preceding conclusion, noting that the coating obtained in combined mode is more stable in the corrosive environment than the coatings obtained under different modes of formation, using both the anode and anode-cathode polarization. On the basis of the dynamic microhardness testing of PEO-coatings obtained under different conditions of formation, it can be concluded that the surface layers formed in the combined mode have the highest microhardness value (21 GPa) and the highest Young's modulus (158 GPa) as compared to coatings formed in other modes (H less than 12 GPa, E is not more than 69 GPa).

2.3 Composite antiscaling coatings on titanium

PEO-coatings have mesoporous clustered structure (Figure 7) which can serve a proper basis for additional coating with polymeric compounds aiming to change mechanical properties, increase hydrophobicity, reduce roughness and influence of various negative factors.

Such composite coatings can provide a very effective extending of the functional properties of metals and alloys. In this connection, the application

of polytetrafluoroethylene is of considerable interest due to the practical importance of the properties of this fluoropolymer.

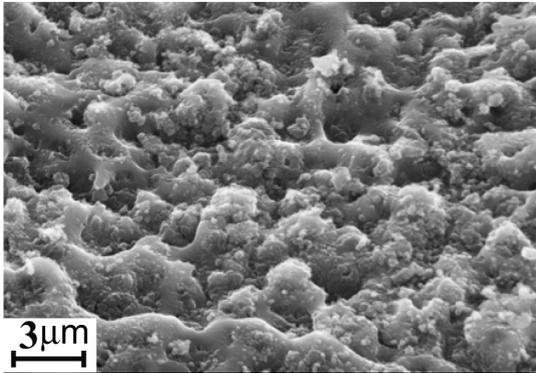


Figure 7: SEM-image of PEO-coating on titanium

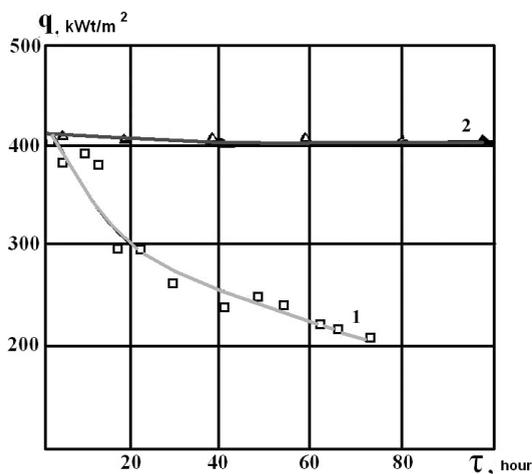


Figure 8: Alteration of heat flux on titanium heat-exchanger with different surface layers: 1 - natural oxide film; 2 - PEO-coating after treatment with small size PTFE powder

The method of polytetrafluoroethylene residue utilization developed in the Institute of Chemistry of the FEB RAS allows us to obtain superdispersed polytetrafluoroethylene (SPTFE, Forum®) with particle dimensions less than 1 μm by gas-dynamic thermodispersion. This material is promising for obtaining of thin composite coatings. At the same

time, when subjecting PTFE to repeated pyrolysis, one can obtain its different oligomeric fractions, which, in turn, may be applied to the structure of composite layers oriented toward specific operating conditions.

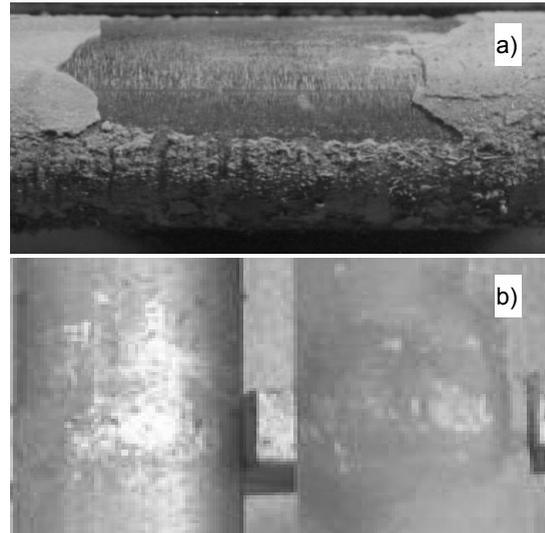


Figure 9: Optical images of titanium heat-exchanger with natural oxide film - a); with PEO-coating treated with small size polytetrafluorethylene powder - b)

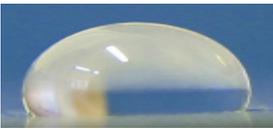
Further research was realized at the industrial test bench for the marine technique testing which was created on the Far Eastern shipyard “Zvezda” [21].

The treatment of PEO-coatings by polytetrafluoroethylene powder was made for creating of hydrophobic layer on the surface, decreasing its roughness.

The results are shown in Figure 8 and Figure 9.

The processing of non-oxidized surface by polymer powder is not effective because the powder has not good adhesion to metal. The PEO-coating, possessing some roughness, guarantees fine adhesion. The experiment for each type of surface layer was carried out during 110 hours.

Table 1: Effect of type of the surface treatment on the limiting wetting angle of the surface layer

Type of the surface layer	Limiting wetting angle, degr.
Natural oxide	52 
PEO-coating	87 
PEO-coating treated by PTFE	99-130 

Also PEO - coating use for the purpose of increasing of the hydrophobicity and decreasing of the surface roughness (Table 1), which reduce negative impact of the different levels, might be rather significant and effective.

4. Conclusions

Based on the results of the study we can conclude that on a commercially pure aluminum and low-carbon steel were formed protective coatings that possess improved anticorrosion properties and thermal stability over a wide temperature range in comparison with the substrate material. This behavior is explained by their low defect mesostructure and the stability of chemical composition of the surface layers.

Our investigations have shown prospects of the use of the UPTFE and high-temperature fraction of UPTFE to creation of composite polymer-containing coatings on metals and alloys, with a layers formed by plasma electrolytic oxidation as the basis. Repeated application of fluoropolymer and

subsequent thermal treatment allows to obtain defect-free composite coating possesses, which give the best protective properties. The investigations show that such composite coatings can be recommended as preventing scale formation and corrosion on heat-exchanging surfaces of equipment of Marine Engineering.

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