

## Enhancing the corrosion resistance of oxide-ceramic composites by the cyclic anodic polarization technique

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### Abstract

The effect of cyclic electrochemical polarization on the corrosion properties of oxide-ceramic coatings consisting of transition metal oxides, boron carbide, and boron nitride was studied. The coatings were applied by laser melting of powder mixtures on the surface of low-carbon unalloyed steel using a fiber-optic laser. The resulting coatings feature enhanced tribological properties. The composition, surface condition, and resistance of samples to electrochemical corrosion were studied. Voltammetric curves were recorded in a neutral buffer solution. It was shown that the presence of boron nitride in the coating composition leads to depassivation of the steel surface. The conceptual possibility of enhancing the corrosion resistance of samples coated with B<sub>4</sub>C–BN–Bi<sub>2</sub>O<sub>3</sub>–MnO<sub>2</sub> by cyclic polarization was shown.

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**Keywords:** oxide-ceramic coatings, boron carbide, transition metal oxides, anticorrosion properties.

### Introduction

Industrial development urgently requires the creation of materials with new combinations of properties. Currently, the parts most commonly used in mechanical engineering are manufactured from iron alloys. Alternative materials for the manufacture of these parts cannot be used for a number of reasons; therefore, to endow steel with the desired properties, it is additionally subjected to physical or physicochemical treatment [1, 2]. Unlike most steels, oxide-ceramic materials feature both high hardness and a low coefficient of sliding friction [3]. These properties are primarily determined by the state of the surface. Therefore, to endow steel parts with the specified properties, it is sufficient to apply an oxide-ceramic coating to the surface of the steel [4, 5]. In particular, the laser cladding method is used to this end [6–8]. However, if such coatings are applied, the corrosion and electrochemical properties of the resulting material can be altered. This is due to the fact that the resulting coatings are porous, which allows the substrate material to contact with particles of oxide-

ceramic coatings. This can accelerate the partial cathodic reaction and thereby deteriorate the corrosion resistance of the resulting composite as a whole. The effect of carbide and oxide phases on the electrochemical properties of steels was studied previously [9–14]. The presence of these particles was found to change the electrochemical properties significantly, which may include deterioration of the corrosion resistance of the material. On the other hand, situations were observed where acceleration of the cathodic process could lead to the transition of the composite material to a passive state.

If composite materials are used in friction units during standby or routine maintenance of mechanisms, moisture can appear on the surface of these coatings. As a result, conditions for electrochemical corrosion are created. The metal phase of the substrate on which the coating is applied is primarily subject to corrosion. Due to this, the oxide-ceramic phase can get delaminated from the metal surface, resulting in the loss of the desired composite's properties. In this regard, an additional task, *i.e.*, enhancement of the corrosion resistance of metal-ceramic materials, arises. Previously, we found that the resistance of these materials to electrochemical corrosion can be increased by inhibitory treatment [12–15]. However, it may occur that the protective properties of the inhibitor treatment are lost due to wash-off of the inhibitor from the surface of the material or thermal destruction of the inhibitors due to elevated temperatures in the friction unit. Therefore, an urgent task is to find alternative options to enhance the corrosion resistance of materials. One such method may involve steel passivation by cyclic electrochemical polarization.

We studied the corrosion and electrochemical properties of samples with oxide-ceramic coatings of various compositions consisting of transition metal oxides, boron carbide, and boron nitride applied on a steel 10 substrate. Samples with the studied coatings feature improved tribological properties [4, 8]. The effect of electrochemical cyclic polarization in a borate buffer solution (BBS) at pH=7.4 under natural aeration on the surface condition and chemical composition of metal-ceramic composites was examined.

## Experimental

Oxide-ceramic coatings were synthesized by high-speed laser processing of a mixture of powders with various compositions on the surface of steel 10 plates with dimensions of 10×10×1 mm as a substrate. To ensure uniformity of application and preliminarily fix the powders on the steel surface, powder mixtures were applied as a suspension in 5% solution of rosin in ethanol. The compositions of oxide-ceramic coatings are presented in Table 1. The parameters of laser radiation for the treatment of materials were selected using the technique reported previously [16].

To deposit oxide-ceramic coatings, an experimental setup was used that comprised a short-pulse ytterbium fiber laser (commercially available short-pulse laser LDesigner F1 with a maximum power of 50 W;  $\lambda = 1.065 \mu\text{m}$ ;  $\tau = 40 \text{ ns}$ ) and a deposition chamber. The latter was a vessel through which high-purity argon was passed. Laser deposition of a mixture of powders onto the steel surface was carried out in one pass by scanning a laser

beam over the sample surface. The technique for obtaining coatings is described in more detail in [14, 15]. The resulting thickness of oxide-ceramic layers was 5  $\mu\text{m}$ .

**Table 1.** Compositions of oxide-ceramic coatings.

Oxide-ceramic coating	Composition (wt.%)
B <sub>4</sub> C–FeO	B <sub>4</sub> C – 90, FeO – 10
MnO <sub>2</sub>	MnO <sub>2</sub> – 100
Bi <sub>2</sub> O <sub>3</sub> –MnO <sub>2</sub>	MnO <sub>2</sub> – 80, Bi <sub>2</sub> O <sub>3</sub> – 20
B <sub>4</sub> C–BN–Bi <sub>2</sub> O <sub>3</sub> –MnO <sub>2</sub>	B <sub>4</sub> C – 60, BN – 14, Bi <sub>2</sub> O <sub>3</sub> – 13, MnO <sub>2</sub> – 13

To determine the structure-phase composition, surface state, and chemical composition of the samples, X-ray diffraction analysis, scanning electron microscopy, and X-ray photoelectron spectroscopy (XPS) were used. X-Ray diffraction patterns of the samples were recorded using a DRON-3 diffractometer in Co- $K\alpha$  radiation at points in the range of Bragg angles 20–130° with a step of 0.05°; the pulse collection time in one point was 5 s. The surface condition and elemental composition of the samples were studied using a FEI INSPECT S50 scanning electron microscope equipped with an X-ray dispersion analysis system. The sample surface was studied by XPS with a SPECS electronic spectrometer using exciting Mg $K\alpha$  radiation in the mode of a constant transmission energy of 15 eV of a Phoibos-150 energy analyzer (equipment provided by the Shared Use Center of the Ural Branch of the Russian Academy of Sciences).

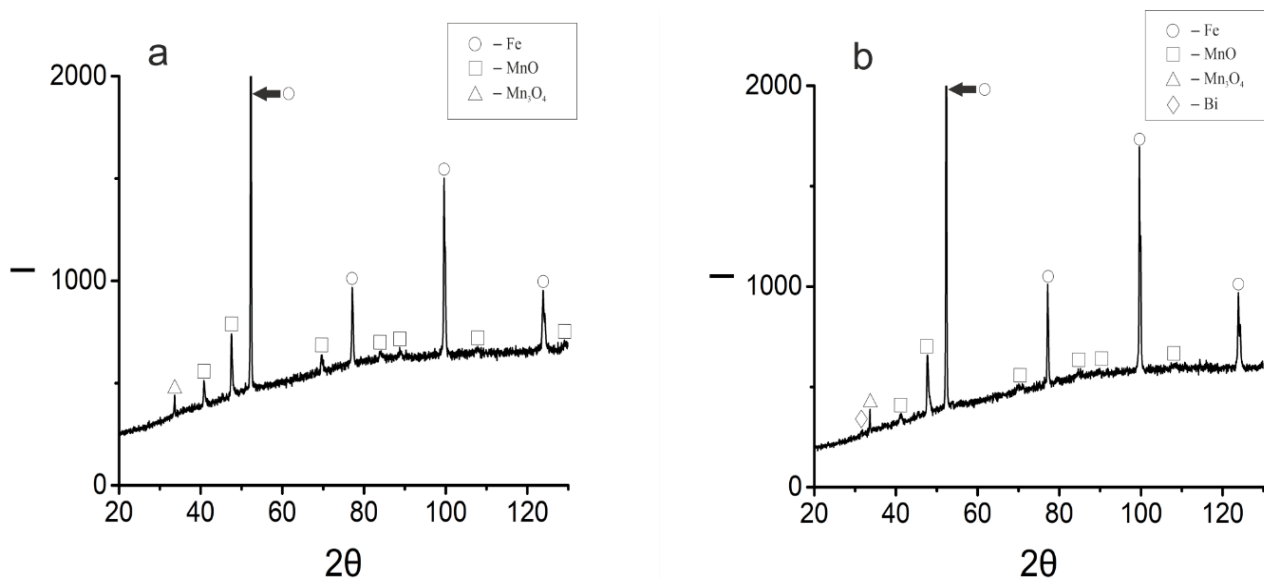
The corrosion and electrochemical characteristics of the samples were studied by cyclic polarization under potentiodynamic conditions using a technique described in more detail elsewhere [12, 13]. The non-working surface of the samples was insulated by coating with a non-conductive varnish. A linear potential sweep was carried out at a rate of 1 mV/s in the potential range from –700 to +1100 mV. An IPC-Pro L potentiostat and a standard YaSE-2 cell at an ambient temperature of (20±2)°C were used. A borate buffer solution with pH 7.4 (BBS 7.4) was used as the model electrolyte. This composition of the electrolyte was chosen since borate ions are indifferent with respect to the coatings synthesized and the substrate steel, *i.e.*, the electrolyte does not contribute to the transition of the material to a passive or active state. A saturated silver chloride was used as the reference electrode, relative to which the electrode potentials  $E$ , mV, of the polarized samples are presented. The current  $I$  was measured in  $\mu\text{A}$  units and was referred to 1 cm<sup>2</sup> of the electrode surface.

## Results and Discussion

### Results of laser treatment

Figure 1 presents the results of X-ray diffraction analysis of the synthesized coatings consisting of MnO<sub>2</sub> and Bi<sub>2</sub>O<sub>3</sub>–MnO<sub>2</sub>. The results of the structural analysis of B<sub>4</sub>C–FeO and

$B_4C$ – $BN$ – $Bi_2O_3$ – $MnO_2$  coatings were reported in our previous works [14, 15]. A common feature of all X-ray diffraction patterns is the presence of peaks corresponding to the iron of the substrate. The  $B_4C$ – $FeO$ -coated sample contained phases of boron carbide, iron oxide, and iron carboboride. In the sample coated with  $MnO_2$ , no lines corresponding to manganese dioxide are observed, while there are lines corresponding to manganese oxides  $MnO$  and  $Mn_3O_4$ , suggesting that manganese dioxide undergoes decomposition during laser treatment. In samples with  $Bi_2O_3$ – $MnO_2$  and  $B_4C$ – $BN$ – $Bi_2O_3$ – $MnO_2$  coatings, in addition to the reduction of manganese oxides, bismuth oxide is also reduced to the metallic state. The powder mixture from which the  $B_4C$ – $BN$ – $Bi_2O_3$ – $MnO_2$  sample was synthesized included boron nitride and carbide; however, no peaks of boron compounds were detected in the diffraction pattern. In this sample, boron carbide exists in the form of an amorphous boron carbide phase with inclusions of boron nitride particles 1–10 nm in size [4].

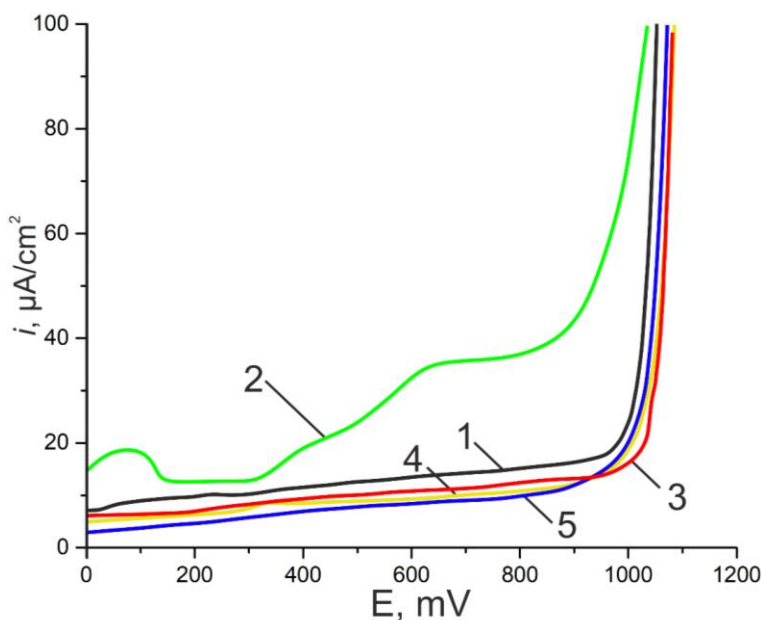


**Figure 1.** Results of X-ray diffraction analysis of coatings:  $MnO_2$  (a);  $Bi_2O_3$ – $MnO_2$  (b). The symbols at the top of the figures correspond to the composition of the phases in the spectrum.

### *Corrosion and electrochemical behavior*

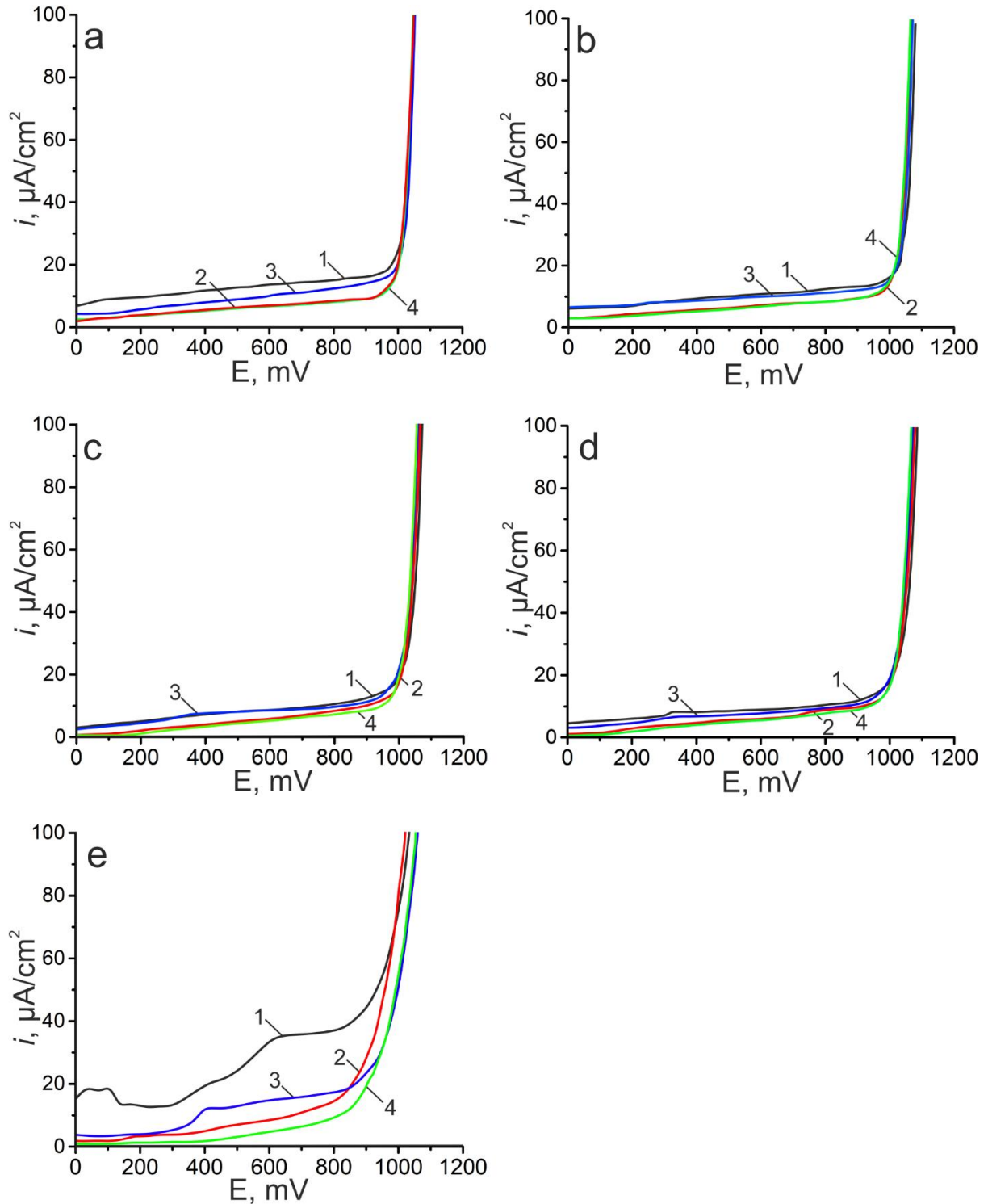
The anodic potentiodynamic curves of the studied samples made of steel 10 with oxide-ceramic coatings are presented in Figure 2. The anodic polarization curve of steel 10 shows that the material is easily passivated in a neutral borate buffer solution at pH 7.4. Application of oxide-ceramic coatings to steel 10 changes the shape of anodic polarization curves. On application of coatings with the compositions  $B_4C$ – $FeO$ ,  $MnO_2$ , and  $Bi_2O_3$ – $MnO_2$ , a slight decrease in the current in the passive state and an increase in the repassivation potential are observed (Table 2). Application of an oxide-ceramic coating consisting of  $B_4C$ – $BN$ – $Bi_2O_3$ – $MnO_2$  leads to a slight decrease in the repassivation potential and a significant increase in the anodic dissolution current at a potential of +400 mV. This observation is consistent with the conclusions of our previous work: application of coatings consisting of metal oxides

increases the corrosion resistance of the material [2], while the addition of boron nitride results in a decrease in corrosion resistance [13].



**Figure 2.** Anodic potentiodynamic polarization curves: 1 – steel 10; 2 – steel 10 with  $B_4C$ –BN– $Bi_2O_3$ – $MnO_2$  oxide-ceramic coating; 3 – steel 10 with  $B_4C$ –FeO oxide-ceramic coating; 4 – steel 10 with  $Bi_2O_3$ – $MnO_2$  oxide-ceramic coating; 5 – steel 10 with  $MnO_2$  oxide-ceramic coating.

We have also shown [2] that corrosion resistance can be enhanced by oxidizing the surface of the material. As a result of anodic polarization, irreversible formation of metal oxides can occur on the surface of the samples. As a result, passivation of the metal surface is possible that can increase the corrosion resistance of the material. Figure 3 presents cyclic voltammetric curves of steel 10 samples with oxide-ceramic coatings. Comparison of the anodic curves of the first and second cycles suggests a transition of the material to a passive state. This comparison clearly shows that the current density at equal potentials on the anodic polarization curves of the second cycle is smaller. A significant decrease in current density is observed on samples of steel 10 and steel 10 with all the oxide-ceramic coatings studied. The anodic polarization curves of samples with oxide-ceramic coatings  $B_4C$ –FeO,  $MnO_2$ , and  $Bi_2O_3$ – $MnO_2$  exhibit almost identical current densities at equal potentials. This leads to the conclusion that if coatings with this composition are applied, the steel surface is transferred to the passive state.



**Figure 3.** Cyclic voltammetric curves: a – steel 10; b – steel 10 with FeO–B<sub>4</sub>C oxide-ceramic coating; c – steel 10 with MnO<sub>2</sub> oxide-ceramic coating; d – steel 10 with Bi<sub>2</sub>O<sub>3</sub>-MnO<sub>2</sub> oxide-ceramic coating; e – steel 10 with B<sub>4</sub>C–BN–Bi<sub>2</sub>O<sub>3</sub>–MnO<sub>2</sub> oxide-ceramic coating; 1 – anodic curve of the first cycle; 2 – cathodic curve of the first cycle; 3 – anodic curve of the second cycle; 4 – cathodic curve of the second cycle.

The values of currents in the passive state and repassivation region are displayed in Table 2.

**Table 2.** Values of current in the passive state region and at the repassivation potential of samples with various oxide-ceramic coatings.

Oxide-ceramic coatings	Current in the passive state region (at $E = +600$ mV), $\mu\text{A}$		Repassivation potential, mV	
	Cycle 1	Cycle 2	Cycle 1	Cycle 2
Without coating	13	9	1004	1008
FeO-B <sub>4</sub> C	10	9	1024	1024
MnO <sub>2</sub>	9	8	1020	1020
Bi <sub>2</sub> O <sub>3</sub> -MnO <sub>2</sub>	9	8	1021	1021
B <sub>4</sub> C-BN-Bi <sub>2</sub> O <sub>3</sub> -MnO <sub>2</sub>	32	15	954	960

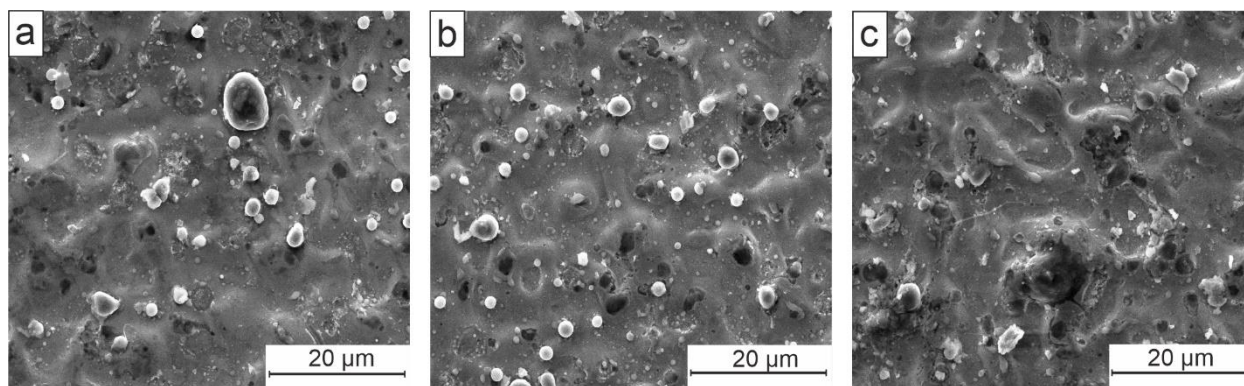
A slight decrease in the anodic current in the passivation region is observed during the second polarization cycle for all the samples studied, including the original uncoated sample. However, the greatest inhibition of anodic dissolution is observed for the B<sub>4</sub>C-BN-Bi<sub>2</sub>O<sub>3</sub>-MnO<sub>2</sub> composite. To find the reasons for the increase in corrosion resistance after electrochemical treatment, the surface of these samples was additionally studied by electron microscopy and XPS methods.

The state of the surface can change upon anodic polarization. Figure 4 shows an image of the surface of the B<sub>4</sub>C-BN-Bi<sub>2</sub>O<sub>3</sub>-MnO<sub>2</sub> oxide-ceramic composite obtained by scanning electron microscopy. One of the main advantages of such coatings is their heterogeneity, which allows the material to combine such properties as the high hardness of boron carbide and boron nitride and a low coefficient of friction due to the presence of oxide phases. After anodic polarization, the surface of the material does not undergo any significant changes (Figure 4b,c), with the exception of a decrease in the number of light inclusions. This observation suggests the conclusion that it is mainly the steel substrate that is subject to electrochemical corrosion. Based on the EDX analysis, it was shown that light inclusions on the surface of the material consist predominantly of manganese dioxide. This is due to the low melting point of this compound (535°C), which forms spherical inclusions upon solidification. Manganese dioxide has little effect on the tribological properties [4], so removing it from the surface of the material does not affect the operating characteristics of the material.

According to X-ray diffraction analysis, the resulting oxide-ceramic coatings are formed primarily by chemical compounds of the following elements: iron, oxygen, aluminum, manganese, silicon, and bismuth. There are limitations to this method: elements whose atomic weight is smaller than that of nitrogen cannot be determined. A comparative description of the elemental composition of the samples after anodic polarization is presented



in Table 3. After anodic polarization, an increase in the atomic fraction of oxygen and a decrease in the atomic fractions of other elements are observed, which may also indicate the formation of oxides on the surface of the material involved in the formation of the passivating layer.



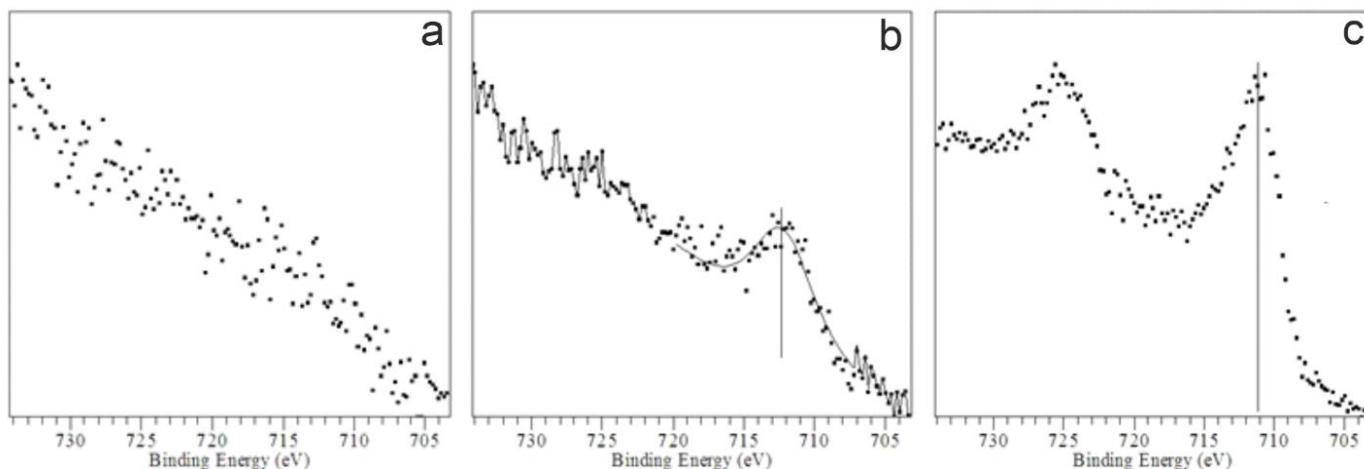
**Figure 4.** SEM image of the surface of samples with an  $B_4C$ -BN- $Bi_2O_3$ - $MnO_2$  oxide-ceramic coating: before electrochemical tests (a); after one half-cycle of electrochemical polarization (b); after three half-cycles of electrochemical polarization (c).

**Table 3.** Elemental composition of surface layers of samples with the  $B_4C$ -BN- $Bi_2O_3$ - $MnO_2$  oxide-ceramic coating.

Sample	Contents of chemical elements in the sample, at. %					
	Fe	O	Al	Mn	Si	Bi
Before anodic polarization	87.63	7.13	2.00	1.73	1.30	0.21
After one half-cycle of electrochemical polarization	87.09	8.43	1.50	1.63	1.21	0.15
After three half-cycles of electrochemical polarization	76.66	19.93	1.28	1.18	0.85	0.10

As an example, Figure 5 shows the results of XPS analysis of samples with the  $B_4C$ -BN- $Bi_2O_3$ - $MnO_2$  oxide-ceramic coating. During electrochemical polarization, a new characteristic line corresponding to Fe(II) and Fe(III) (Fe2p) from substrate iron appears. The Fe2p spectrum region of the sample that was not subjected to electrochemical tests does not contain components characteristic of iron. It was found that the intensity of the oxidized iron line in the sample subjected to three half-cycles of electrochemical treatment is higher than in that subjected to one half-cycle. The data obtained indicate that iron of the substrate undergoes oxidation; this process is electrochemically irreversible and contributes to the formation of the passive layer. A similar effect of anodic polarization indicating the formation of iron oxides on the surface of the composite is also characteristic of the other samples studied. However, the oxidation of the steel substrate is less pronounced, which is responsible for the lower inhibition of the anodic process.





**Figure 5.** XPS Fe2p spectra of iron samples with the  $B_4C$ –BN– $Bi_2O_3$ – $MnO_2$  oxide-ceramic coating: before electrochemical tests (a); after one half-cycle of electrochemical polarization (b); after three half-cycles of electrochemical polarization (c). The vertical line marks the  $Fe2p_{3/2}$  peak of oxidized iron in the Fe2p spectrum.

## Conclusions

1. The effect of applying oxide-ceramic coatings on the corrosion and electrochemical properties of steel 10 in a neutral borate buffer solution at  $pH=7.4$  was shown. Coatings with the compositions  $B_4C$ –FeO,  $MnO_2$ , and  $Bi_2O_3$ – $MnO_2$  enhance corrosion resistance, whereas the  $B_4C$ –BN– $Bi_2O_3$ – $MnO_2$  coating decreases the corrosion resistance of steel 10 under these conditions.
2. The effect of cyclic electrochemical polarization on the properties of oxide-ceramic coatings was studied. As a result of this treatment, the corrosion resistance of samples of steel 10 and steel 10 with the  $B_4C$ –BN– $Bi_2O_3$ – $MnO_2$  coating increases. Cyclic electrochemical polarization exerts a much smaller effect on the corrosion resistance of samples with the  $B_4C$ –FeO,  $MnO_2$ , and  $Bi_2O_3$ – $MnO_2$  coatings.
3. Enhanced corrosion resistance of steel 10 coated with  $B_4C$ –BN– $Bi_2O_3$ – $MnO_2$  is due to the passivation of the substrate material. The transition of the material to the passive state is caused by the irreversible formation of iron oxides, the presence of which was determined by electron microscopy and XPS spectroscopy.

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