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SURFACE MORPHOLOGY OF TUNGSTEN AND STAINLESS-STEEL SURFACES AFTER ELECTROLYTIC-PLASMA BORIDING

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The results of diffusion saturation of steel and tungsten surfaces with boron under conditions of electrolytic-plasma boriding EPB (anodic process) have been considered. The processing modes and electrolyte compositions, structural features of modified layers, and their microhardness are presented. A conclusion is made about the prospects of electrolytic-plasma boriding, and the advantages and limitations of the method are discussed. Surface morphology and chemical composition of the samples were investigated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS).

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INTRODUCTION

Borated steels have a fairly high wear resistance compared to carburized or nitrocarburized steels due to the formation of solid iron borides. Boridng can be carried out in various environments. Traditional methods include boriding in powder mixtures, electrolysis, liquid and gas [1].

Saturation with boron from the solid phase is carried out in powders of boron carbide, ferroboron or amorphous boron. This can be done in a vacuum as a result of evaporation of amorphous boron or boron carbide with condensation of these vapors on the surface of the workpiece and subsequent diffusion of boron into the metal. In this case, pastes containing boron carbide and a binder, such as cryolite, are used [2].

A relatively new EB technique, electrolytic plasma boriding (EPB), was lately introduced to conduct boriding in the aqueous solutions of borax with the addition of some additives (NaOH, Na_2CO_3, NaCl, etc.) to increase the electrolyte conductivity where a high DC voltage power supply (e.g., 30 kW – 500 V and 60 A or 50 kW – 500 V and 100 A, etc.) was applied to the electrodes to produce plasma on the surface of the workpieces [3–5]. Electrolytic-plasma boriding of metals and alloys is currently being successfully developed, which has certain advantages. The process takes several minutes, is combined with subsequent hardening in the same electrolysis. The use of toxic and explosive substances is excluded [6].

Another new method is laser irradiation. A layer of paste from nanodispersed powders containing amorphous boron with a dilute solution of polyvinyl alcohol is melted by a laser beam moving along the surface of the sample. This method made it possible to increase the hardness of austenitic stainless steel and improve the properties of low-carbon steel after its gas boron carburizing. It should be noted that electron beam treatment of borated samples causes modification of the crystal structure that formed after borating in boron carbide powder.

The listed boriding methods are characterized by both a fairly long exposure of the boron saturation processes (except for expensive electron beam or laser), and a high concentration of the reagents used.

In this research, the results of diffusion saturation of stainless steel and tungsten surfaces with boron under conditions of electrolytic-plasma boriding EPB (anodic process) have been considered.

1. EXPERIMENTAL SETUP

In all experiments a borax solution (sodium tetraborate) with concentrations 15…20% was used as an electrolyte. The conditions under which saturation was performed were as follows: the voltage on the electrolytic-plasma cell was 224…227 V, current density during the saturation process was about 1 A/cm^2, concentration of the solution was 15…20%. The exposure was 1…10 min. The temperature of the samples was estimated by pyrometry methods and ranged from 950 to 1000 °C.

Upon completion of the saturation process, the sample was removed from the solution and the discharge potential was then turned off. The second option is that the sample remained in the solution after the potential was turned off (hardening effect). The saturation modes of steel and tungsten samples did not differ.

To provide EPB, three various regimes were applied for the experiments:

1. EPB on tungsten surface – 3 min, S = 8 cm^2, U = 227…225 V, I = 8 A.

2. EPB on 12X18H10T SS substrate. EPB in 20% borax ($Na_2B_4O_7$) solution in water. Exposure – 10 min. S = 9 cm^2, U = 226…222 V, I = 8…9 A.

3. EPB on 12X18H10T. A solution of borax ($Na_2B_4O_7$) in water 15%. Exposure – 10 min, S = 9 cm^2. U = 224…220V, I = 9.5…10 A.

Structure and chemical composition of the samples were investigated by scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) by using ZEISS EVO® MA10.

Microhardness measuring microscope (PMT-3) was used for characterizing the samples physical properties.

2. RESULTS AND DISCUSSION

Fig. 1 shows the SEM surface morphology and EDS mapping after EPB of tungsten surface in 15% borax solution ($Na_2B_4O_7$) during exposure for 3 min. It should be noted that the temperature of the treated part during anodic treatment does not exceed 950…1000 °C, while during cathodic treatment, temperatures up to 3600 °C and higher were observed. In the case of anodic treatment, conductivity in the vapor-gas area is carried out by electrolyte anions, and in the case of cathodic treatment by cations also emitted from an aqueous solution. Oxidation processes during the anodic process are more intense than during the cathodic process (Fig. 2).

EDS spectra from EPB on tungsten surface as well as chemical composition are shown in Figs. 3 and 4, respectively. The chemical composition comprised 10.8 at.% B and 45.6 at.% O after EPB.

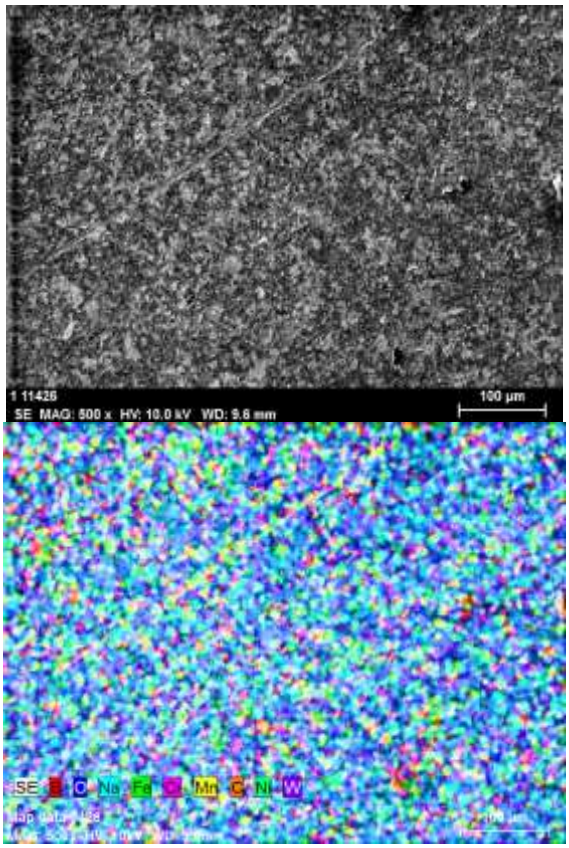

Fig. 1. SEM image after EPB on tungsten surface. 15% borax solution ($Na_2B_4O_7$) in water. Exposure – 3 min, S = 8 cm^2, U = 227…225 V, I = 8 A (top), EDS mapping (button)

Anodic dissolution of the sample surface helps to reduce surface roughness and round off the edges. The inevitable oxide layer in some cases increases corrosion resistance, but inhibits the diffusion of elements from solutions – the nature of the current is constant or pulsed.

In general, it can be noted that the specificity of the saturation of steel and tungsten samples with boron lies in the composition of the electrolyte used and, as a consequence, the boron potential of the resulting vapor-gas gap. Publications indicate various concentrations of borax for boriding steels, but the mechanism for the release of the saturating component is almost completely unknown. The problem is complicated by the strong dependence of borax solubility on temperature. In any particular electrolyte, the solution temperature varies from approximately room temperature far from the processing zone to the boiling point at the boundary of vapor-gas area.

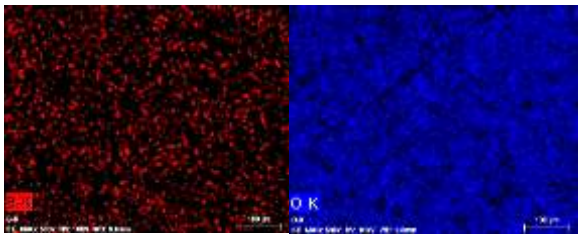

Fig. 2. EDS mapping of selected chemical elements: B (left) and O (right)

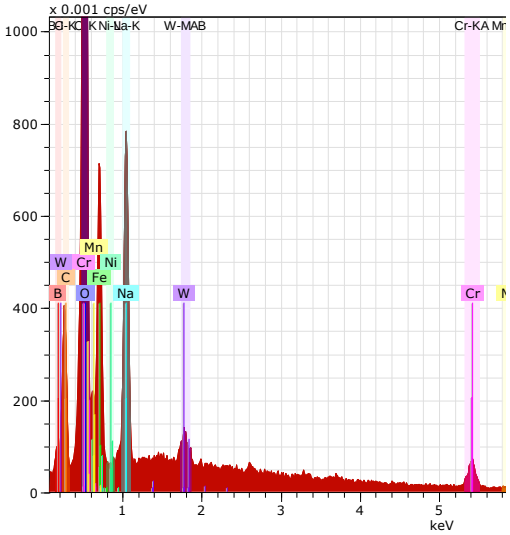

Fig. 3. EDS spectra from EPB on tungsten surface

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]
Boron	K-series	4.40	4.95	10.83
Oxygen	K-series	27.45	30.85	45.64
Sodium	K-series	6.79	7.63	7.86
Iron	K-series	36.14	40.62	17.22
Chromium	K-series	5.91	6.64	3.02
Manganese	K-series	0.00	0.00	0.00
Carbon	K-series	6.84	7.69	15.15
Nickel	L-series	0.22	0.25	0.10
Tungsten	M-series	1.22	1.38	0.18
	Total:	88.96	100.00	100.00

Fig. 4. Chemical composition of tungsten surface after EPB according to EDS

It should be noted that after EPB on a tungsten substrate, the surface became polished. The required exposure for polishing was an order of magnitude less than the saturation exposure. For samples with an area of 8 cm^2 it took 1 min.

In this case, the polishing mode differs from the generally common one for electrolytic-plasma polishing. Electrolytic-plasma polishing usually has a "falling" current-voltage characteristic. In our case, the current-voltage characteristic was linear. During

polishing, the operating point on the current-voltage characteristic did not undergo any significant changes, just a few volts in voltage. The microhardness of the ss samples after saturation with boron was on average 2…2.5 GPa.

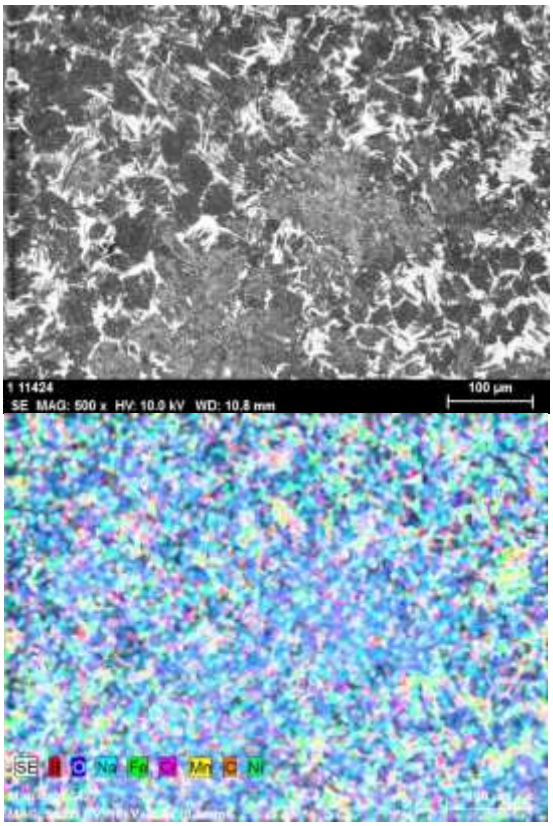

Fig. 5. 12X18H10T SS substrate. EPB in 20% borax ($Na_2B_4O_7$) solution in water. Exposure – 10 min, S = 9 cm², U = 226…222 V, I = 8…9 A

Fig. 5 depicts SEM surface morphology and EDS mapping of 12X18H10T SS surface after EPB in 20% solution of borax Na_2B4O_7 in water. Exposure – 10 min, S = 9 cm², U = 226…222 V, I = 8…9 A.

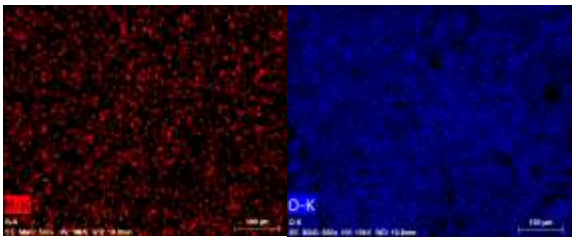

Fig. 6. EDS mapping of selected chemical elements B(left) and O (right)

EDS mapping of selected elements after EPB on SS surface as well as chemical composition are shown in Figs. 6 and 7, respectively. The chemical composition reaches 15.2 at% B and 55.5 at% O after EPB with 20% solution (Fig. 8).

Fig. 9 depicts chemical composition of SS surface after EPB according to EDS (EPB on 12X18H10T. A solution of borax ($Na_2B_4O_7$) in water 15%).

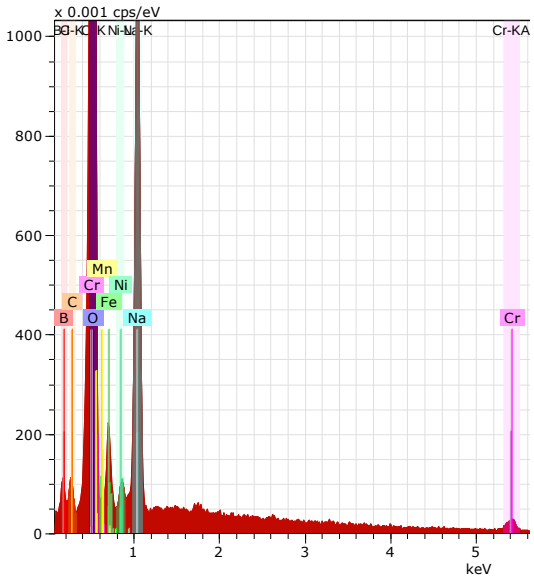

Fig. 7. EDS spectra after EPB on tungsten surface

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]
Boron	K-series	12.27	15.26	24.57
Oxygen	K-series	41.03	51.05	55.54
Sodium	K-series	10.75	13.38	10.13
Iron	K-series	9.12	11.35	3.54
Chromium	K-series	3.00	3.73	1.25
Manganese	K-series	0.20	0.25	0.08
Carbon	K-series	2.38	2.97	4.30
Nickel	L-series	1.62	2.02	0.60
	Total:	80.37	100.00	100.00

Fig. 8. Chemical composition of SS surface after EPB according to EDS

Element	Series	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]
Boron	K-series	8.97	7.84	21.40
Oxygen	K-series	16.69	14.59	26.90
Sodium	K-series	5.02	4.39	5.63
Iron	K-series	57.98	50.70	26.78
Chromium	K-series	14.39	12.58	7.14
Manganese	K-series	0.93	0.81	0.44
Carbon	K-series	4.19	3.66	9.00
Nickel	L-series	6.20	5.42	2.72
	Total:	114.37	100.00	100.00

Fig. 9. Chemical composition of SS surface after EPB according to EDS

Fig.10. depicts SEM surface morphology and EDS mapping of 12X18H10T SS after EPB in 15% solution of borax $Na_2B_4O_7$ in water. Exposure – 10 min, S = 9 cm², U = 226…222 V, I = 8…9 A.

EDS mapping of selected elements after EPB on SS surface as well as chemical composition are shown in Figs. 11 and 12, respectively.

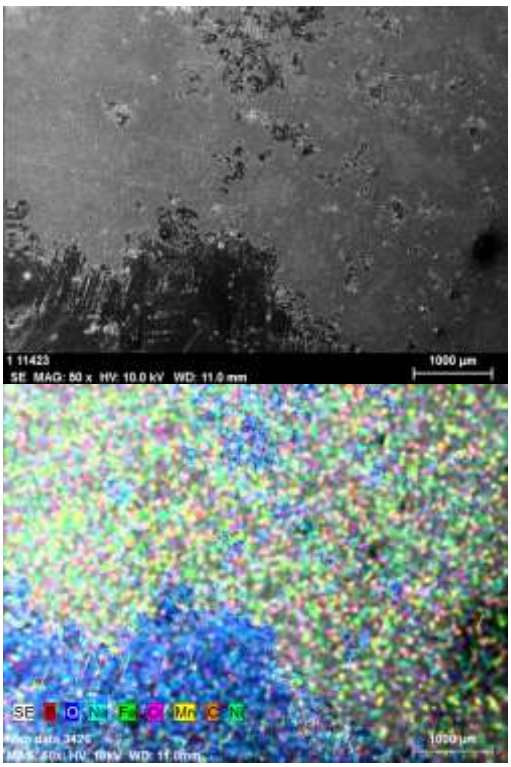

Fig. 10. SEM surface morphology and EDS mapping of 12X18H10T SS after EPB in 15% solution

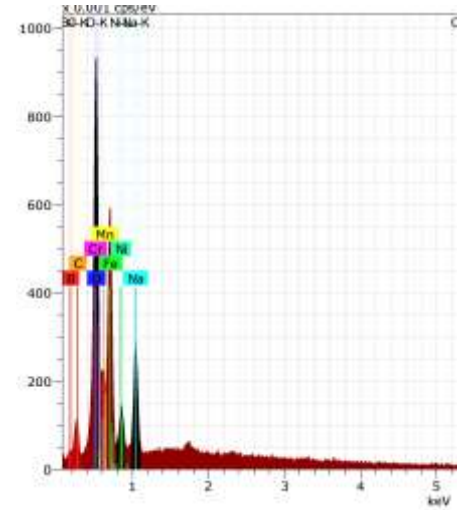

Fig. 11. EDS spectra after EPB on SS surface

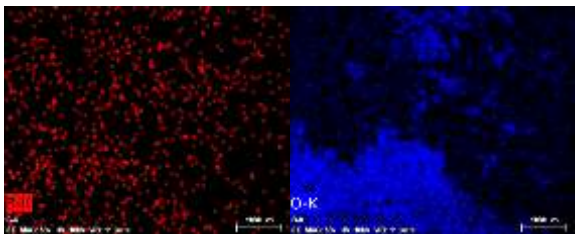

Fig. 12. EDS mapping of selected chemical elements B (left) and O(right) after EPB of SS

CONCLUSIONS

The results of diffusion saturation of stainless steel and tungsten surfaces with boron under conditions of electrolytic-plasma boriding EPB (anodic process) have been considered. After EPB on SS and tungsten substrates, the surface became polished. The microhardness of the ss samples after saturation with boron was on average 2…2.5 GPa. The microhardness of the W samples after EPB increased from initial 200 to 300 kg/mm^2.

REFERENCES

1. Eric J. Mittemeijer, Marcel A.J. Somers. Thermochemical Surface Engineering of Steels // *Woodhead Publishing*. 2015, p. 769-792.

2. H. Kunst et. al. // *Metals, Surface Treatment Ullmann's Encyclopedia of Industrial Chemistry*. 2012, p. 782-783.

3. P. Taheri et. al. Evaluation of nanocrystalline microstructure, abrasion, and corrosion properties of carbon steeltreated by plasma electrolytic boriding // *Plasma Process. Polym.* 2007, v. 4, p. 711-716.

4. S.H. Alavi, C. Dehghanian, P. Taheri. Investigation of corrosion behaviour of carbon steel coated by pulsed plasma electrolytic boronising technique in 3.5 wt.% NaCl aqueous solution // *Surf. Eng.* 2011, v. 27(7), p. 509-514.

5. P. Taheri et. al. Corrosion properties of plasma electro-lytic coated samples // *Anti-Corros. Method. M.* 2007, v. 54(3), p. 148-154.

6. I.K. Tarasov et al. Electrolytic-plasma processing and boriding of various surfaces // *Problems of Atomic Science and Technology.* 2024, N 6(154), p. 100-103.

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МОРФОЛОГІЯ ПОВЕРХНІ ВОЛЬФРАМУ ТА НЕРЖАВІЮЧОЇ СТАЛІ ПІСЛЯ ЕЛЕКТРОЛІТНО-ПЛАЗМОВОГО БОРУВАННЯ

І.К. Тарасов, А.В. Таран, А.М. Тарасов, С.П. Романюк

Розглянуто результати дифузійного насичення бором поверхонь сталі та вольфраму в умовах електролітно-плазмового борування ЕПБ (анодного процесу). Наведено режими обробки та склади електролітів, структурні особливості модифікованих шарів та їх мікротвердість. Зроблено висновок про перспективність електролітно-плазмового борування, обговорено переваги та недоліки методу. Морфологію поверхні та хімічний склад зразків досліджували методами скануючої електронної мікроскопії (СЕМ) та енергодисперсійної спектроскопії (ЕДС).