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ELECTRON BEAM DEPOSITION OF BORON-BASED COATINGS UNDER VACUUM PRESSURE AND EXPERIMENTAL RESULTS OF NITROGENATION IN ELECTRON BEAM PLASMA

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Abstract: The article presents the study results of electron beam deposition of boron-based coatings under vacuum pressure and nitriding in electron beam plasma, measurement results are presented, and the mass-charge composition of the beam plasma is determined.

Keywords: vacuum, plasma-cathode, electron sources, electron beams, boron coating, mass charge, plasma nitriding.

Introduction. The process of impregnation with nitrogen in order to make the surface layer of the parts very hard is called nitriding. The surface layer of the part, saturated with nitrogen, is very hard and resistant to corrosion, as well as corrosion, because nitrides are formed in the surface layer. The process of nitriding is based on the release of active nitrogen atoms during the dissociation (decomposition) of ammonia: Nitrogen in the form of separated atoms diffuses to the surface layer of the detail and forms nitrides [1].

The nitriding process takes 25-60 hours at a temperature of 500-600°C, depending on the thickness of the layer to be nitrided. (a 0.1 mm thick layer is nitrogenized every 10 hours). However, nitriding has a number of advantages over cementation: the heating temperature is relatively low, so there is no need to redefine the details after nitriding. Therefore, the nitrification method is widely used.

Fig. 1. Coating setup (left), nitriding setup (right).

Nitriding and coatings were carried out in two devices equipped with vacuum plasma electron sources based on independent light discharge with a hollow cathode.

Experiments on vaporization of targets with pure boron (boron content 99.9%) were carried out in an experimental device using a forevacuum plasma electron source based on a hollow cathode glow discharge operating in continuous mode, construction and parameters. It is detailed in [2]. The electronic source ensured the creation of an electron beam with a current of up to 150 mA and an energy of up to 15 kV. Electron beams are focused by the magnetic field of the focusing system on a target with a diameter of up to 5 mm. On the way to the boron target, the electron beam was transported through the cavity of the vacuum chamber filled with working gas (He) at a pressure of 5-15 Pa, creating a dense beam plasma. Under the influence of electron beam, vaporized boron atoms are heated, vaporized and ionized. Boron vaporized from the surface of the target is deposited on a sample mounted on a rotating device (Figure 2, when applying coatings, the product is placed 2 cm from the target). Chamber vacuum is provided by an ISP-500C spiral mechanical oil-free front vacuum pump. The duration of the whole process was 3 minutes. The surface temperature of the samples was monitored by a Raytek high-speed optical pyrometer.

Results: Monitoring of the plasma composition during the deposition of boron coatings was carried out using a modified RGA-300 quadrupole residual atmospheric mass analyzer [3]. The high vacuum in the ion transport, separation and recording area inside the RGA-300 was provided by a Pfeiffer HiCube 80 Eco stationary high vacuum pumping station. Figure 3 shows the mass-charge spectra of the plasma at all stages of the target heating process: boron vaporization during heating, melting, and plating. Observation of the mass-charge composition showed that when the boron target is heated, gas ions are recorded in the plasma, which are desorbed from the surface of the target, the crucible and the vacuum chamber when heated. At this stage of the process, the sample was uniformly heated to prevent explosive destruction due to thermal stresses, as well as to transfer the boron target material to a conductive state. A further increase in the light power density ensures the melting of the surface layers of the sample with partial vaporization of its material, which leads to the appearance of boron isotope ions with a low signal amplitude in the spectrum. When the power density of the electron beam reaches 2.5 kW/cm^2, only target ions are recorded in the spectrum. In this case, ions of two stable boron isotopes are recorded in a ratio corresponding to their distribution in nature.

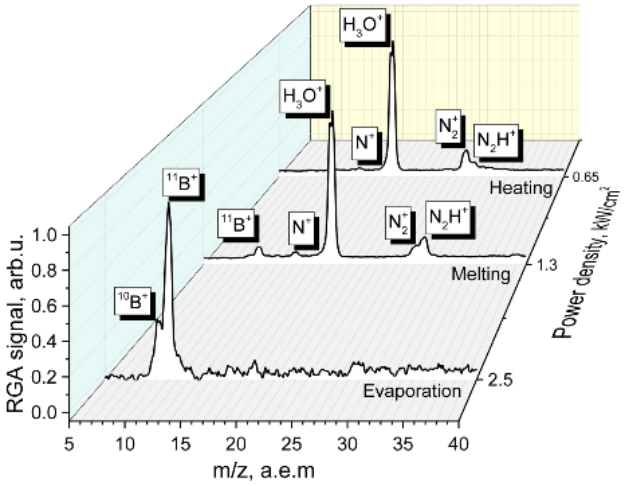


Fig. 3 Mass-charge spectrum of plasma during the application of boron-based coatings at different stages of the heating of a crystalline boron target.

Figure 4 shows the 3D profiles of the sample before and after coating, which shows that the application of boron-based coating can improve the initial roughness of the sample.

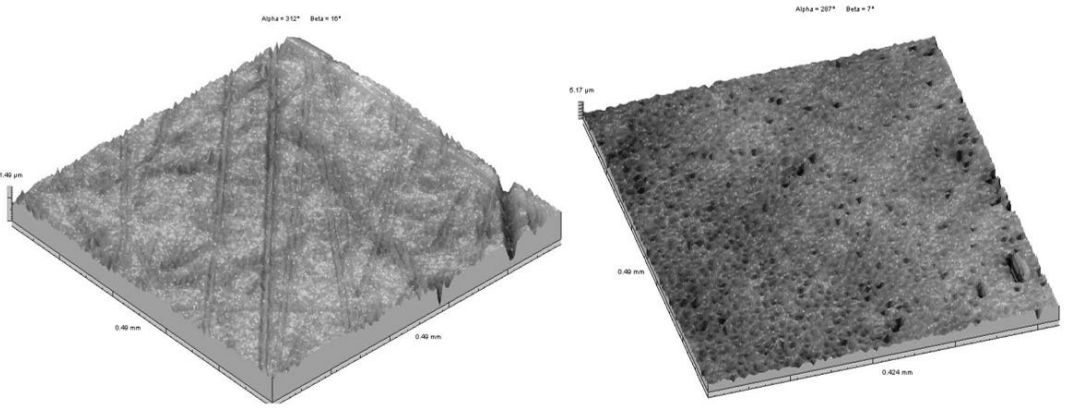
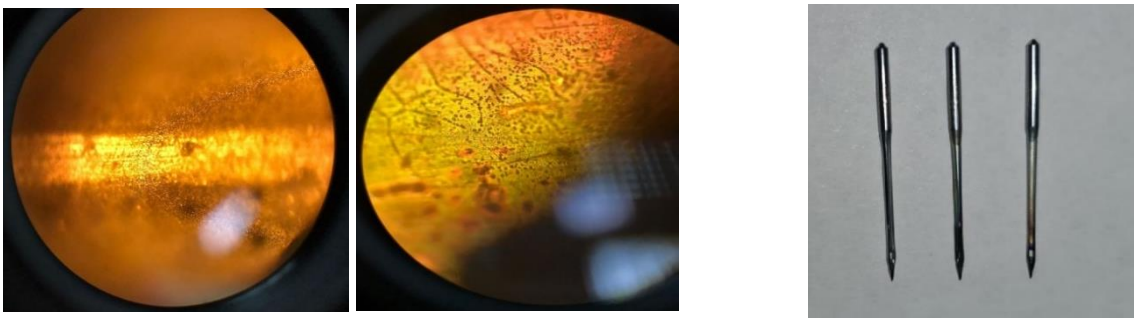


Fig. 4. Sample profiles before coating (left) and after boron coating (right).

Application of boron-based coatings: The coating process took 2 minutes



a) b)

Figure 5. a) Photo taken from a metallographic microscope, b) Photo of boron-based coated needles.

Nitriding. Initial needles (only 2 types of products, conditional (Russian) and (Uzb):

The scheme of nitrification experiments is presented in Fig. 6. A continuous electron beam with an energy of 4-9 keV, a current of 100 mA and a diameter of 20-25 mm was generated by the forevacuum plasma source based on the hollow discharge of the cathode. Nitriding scheme and process are detailed in [4].

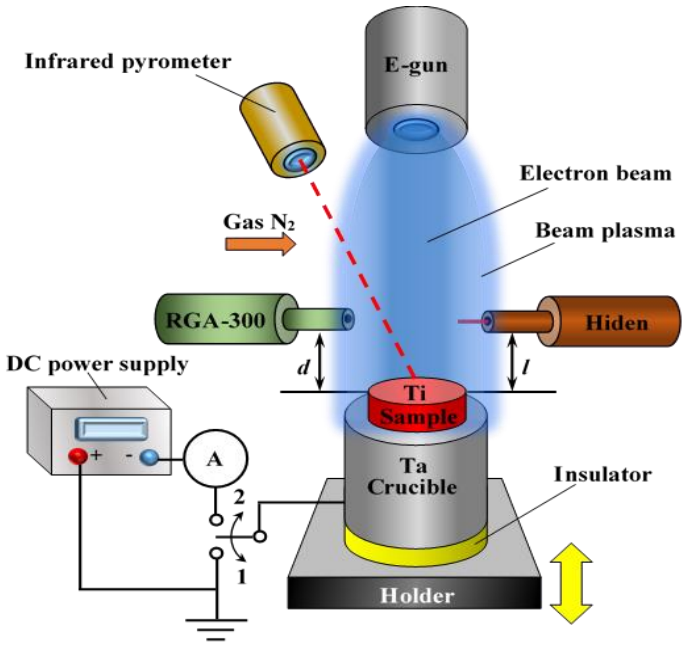
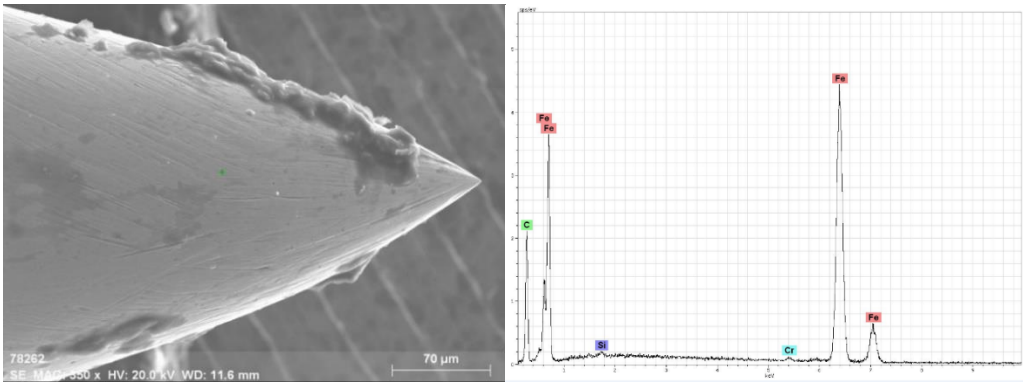


Figure 6 – Scheme of experiments.

The generated electron beam was transported to a titanium target (sample) and used to heat it. The sample temperature was monitored by a non-contact optical pyrometer from Raytek.

Using a nEXT300D turbomolecular pump with a pumping speed of 300 l/s, the working chamber was pre-evacuated to a pressure of 0.03 Pa, after which nitrogen was released into the chamber to the required working pressure of 1 - 7 Pa. Nitriding parameters: Pressure: 5 Pa; Accelerating voltage: 4.5 kV; Light current: 100mA; Temperature: 450 degrees Celsius; Nitriding time: 3 hours.



Element	AN	series	Net	[wt.%]	[norm. wt.%]	[norm. at.%]	wt.% (1 Sigma)
Carbon	6	K-series	6357	34,50917877	30,29628118	66,82859361	5,207204855
Silicon	14	K-series	388	0,226788161	0,19910175	0,187821404	0,042470911
Chromium	24	K-series	414	0,324663238	0,28502819	0,145234584	0,044461202
Iron	26	K-series	51915	78,84502895	69,21958888	32,8383504	2,157584947
			Sum:	113,9056591	100	100	

Figure 8. - The first photo of the needle (Uzb), its elemental composition.

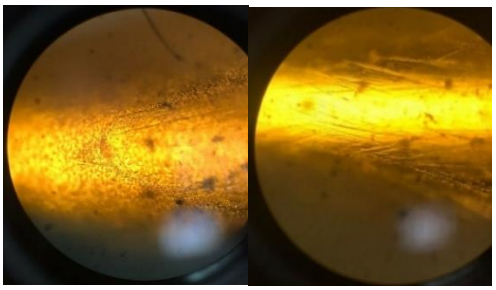


Figure 9. – Needle (Uzb) before nitriding (left) and after nitriding (right).

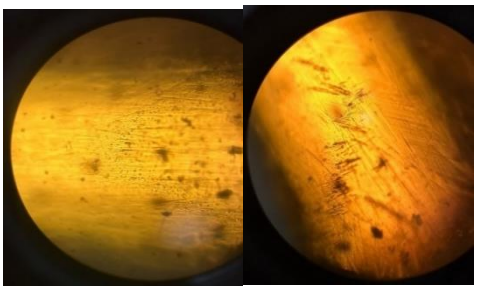


Figure 10. Needle (Rus) before nitriding (left) and after nitriding (right).

Figure 11. – Photo after nitriding.

Conclusion: When using optimal modes, energy consumption is (170-190) W / cm^2. It matches most of the properties of electrolytes for anodic heating.

It was found that the following structures are formed in boron coating: oxide zone (Fe_3O_4, Fe_2O_3, FeO) and nitride-martensite layer (Fe_3N, martensite and retained austenite).

The method of backscattering protons from the nucleus made the concentration of boron in the layer 13% and nitrogen - 11%.

After anodizing the needles, their microhardness increases from HB=54-60 to HB=135-150, that is, the microhardness of their work surface increases 2.5-3 times.

The increase in the microhardness of the needles leads to a decrease in the cost of replacing them with a new one, as a result of which they are slightly (2-3 times) less bent and broken during the sewing process.

After anodic boron-nitriding of the needles, their working surfaces increase the service life of the needles by at least 3 times.

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