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Surface hardening of stainless steel by runaway electrons preionized diffuse discharge in air atmosphere

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Abstract. In this paper we present microhardness measurements of stainless steel surface treated by diffuse discharge in air atmosphere. The cleaning from carbon in comparison to the initial sample was observed at a depth exceeding 20 nm. The oxygen concentration was also increased in comparison to that in the initial sample at a depth of up to about 50 nm. Comparative analysis shows that after treatment the microhardness of stainless steel surface increased in 2 times due to interaction of near-surface layers with product of plasma chemical reactions produced in diffuse discharge.

1. Introduction

In recent years, diffuse discharges with runaway electron preionization have attracted much interest because of the possibilities to uniformly generate rather big volume of plasma in different gases (including electronegative [1]) at atmospheric and elevated pressures [2]. This property enables the development of plasma processing technology at ambient pressure and temperature without using of special vacuum technique, and can be used in a wide range of applications, e.g. in medicine and surface modification. This type of discharge was realized in non-uniform electric field by applying of high voltage pulse of 2 ns full width at half maximum to cathode with small curvature radius. As a plane anode, stainless steel foil of 200 µm thickness was used. In a runaway electrons preionized diffuse discharge (REP DD), the anode experiences complex action of several factors:

- dense nanosecond discharge plasma with a specific power up to 1 GW/cm^3 in a pulse mode [3];
- a shock wave which was recorded with a calorimeter [4];
- UV, VUV radiation, as well as X-rays from the discharge plasma [5];
- a supershort avalanche electron beam (SAEB) with a wide energy range [4].

In the present study, REP DD was employed for surface treatment of stainless steel in air atmosphere. The aim of the work to study changes in microhardness of near-surface layer, its roughness characteristics, as well as surface element composition which was occurred under the action of REP DD.

2. Experimental setup and methods of measurements

High voltage pulsed generator RADAN-220 provided negative voltage pulses with amplitude up to 280 kV (in the open-circuit regime) and pulse front of 0.5 ns (on a matching load) was used for REP DD formation. At such experimental conditions, the specific input power in air plasma was about 100

MW/cm^3 per pulse. Diffuse discharge was formed between cathode and anode in ambient air of atmospheric pressure. The distance between high voltage tube cathode of diameter 6 mm and plane anode was 16 mm.

Before the experiments, plane samples of 150-μm-thick stainless steel foil was cleaned twice in ultrasonic unit Elmasonic S 10H (Elma-Hans Schmidbauer GmbH & Co.) with distilled water and alcohol at 65 °C. The pulse plasma processing of the steel surface was performed with frequency of 1 Hz.

Microhardness was measured by Berkovich diamond indenter on the system of NanoTest 600 (Micro Materials Ltd., UK) by Oliver-Pharr method [6] at loads of 0.5, 1 and 2 mN. After REP DD treatment, concentration changes of the main chemical elements within the surface layers were studied by Auger electron spectroscopy (AES) with the use of STIL-2 Auger-spectrometer [7]. The surface roughness of treated samples was studied with using of a 3D contactless profilometer Micro Measure 3D Station (STIL).

3. Results and discussion

It has been found that samples modified by REP DD have microhardness almost 2 times higher than untreated ones, and its values increase with increasing of number of pulses (see fig. 1). So microhardness of the initial stainless steel sample was around 5.5 GPa, whereas after the exposure to 3000 discharge pulses it increased ~1.5 times, and the value of the sample treated by 6100 pulses increased ~2 times.

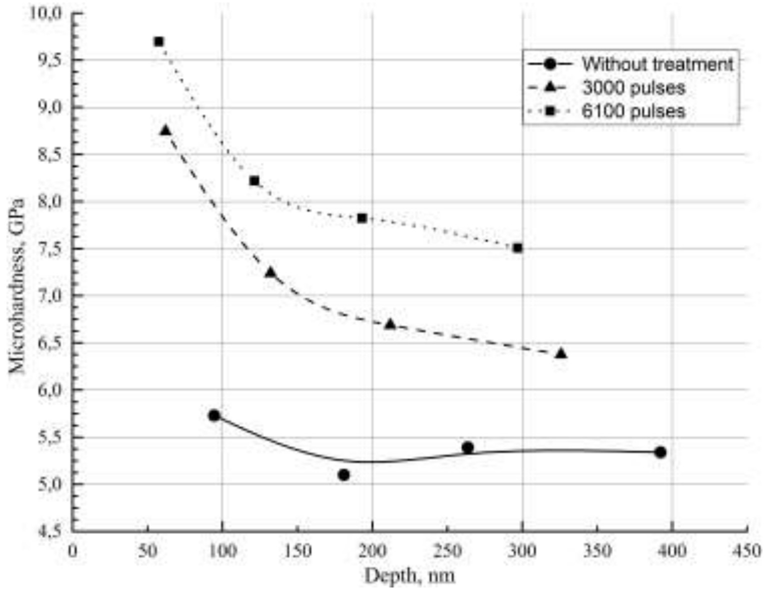

Figure 1. Dependences of surface microhardness versus its depth for initial stainless steel samples, and samples treated by REP DD.

Moreover, study of the sample's surface topology has shown that under the action of REP DD, the planarization of the surface has been occurred. Thus roughness parameter R_a (roughness average, μm) of the initial sample was equal to 0.0315 μm, after treatment by 500 discharge pulses – 0.0234 μm, and for the sample treated with 6100 pulses it decreased in 3 times and was equal to 0.0104 μm. The 2D-profilogram, 3D- topography and statistics data of the geometrical properties of stainless steel surface upon the exposure to 6100 discharge pulses of REP DD are shown on fig. 2.

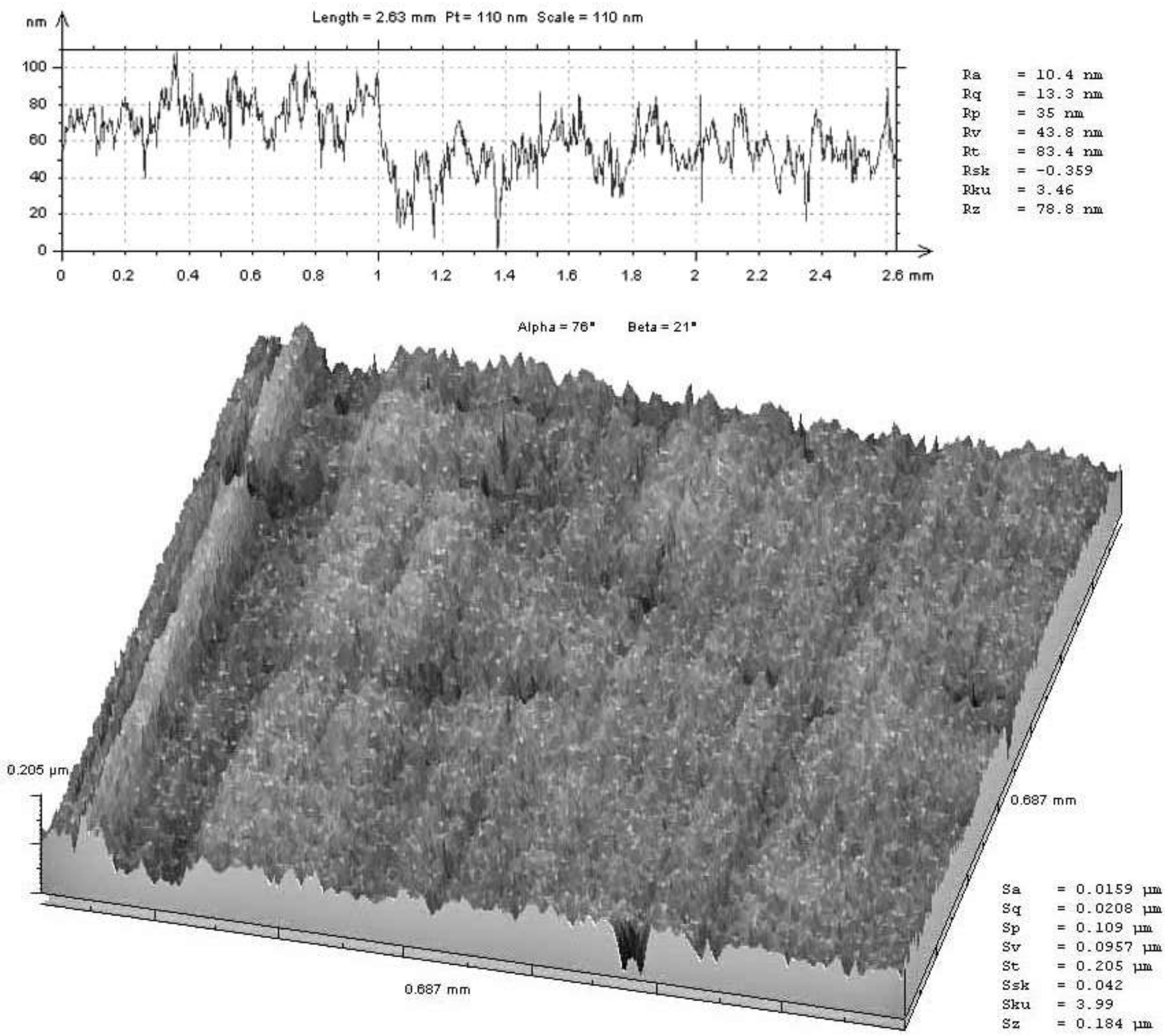

Figure 2. 2D-profilogram, 3D-topography and statistics data of the geometrical properties of stainless steel surface upon the exposure to 6100 discharge pulses of REP DD.

The effect of the diffuse discharge plasma on the examined specimen surface was estimated from the degree of variation in its composition. For this purpose, surface layers of thickness 100–200 nm before and after treatment were analyzed by Auger spectrometry. Fig. 3 shows the carbon and oxygen concentrations in the stainless steel surface layer before and after treatment. It is seen from fig. 3a that the most substantial changes in the carbon concentration of treated samples occurs at a depth of up to 10 nm and decreases with increasing of number of discharge pulses. Thus, after exposure to 1200 pulses the carbon concentration decreases ~2 times, and after exposure to 6100 pulses, it decreases almost 5 times.
Moreover, Auger spectroscopy also showed that under the action of REP DD, the formation of oxide layer on the sample's surface took place. The oxygen concentration and oxide depth in the threated samples increases with increasing of number of discharge pulses. It is seen from fig. 3b that the oxygen concentration and oxide depth for the stainless steel sample treated by 3000 pulses was 10% higher and 1.5 times deeper than in the initial one.

Earlier in [8] it was shown, that in the pulsed periodic mode of voltage pulses with frequency up to 2 kHz and specific input power of 10 MW/cm^3 per pulse, the hardening effect of steel took place. The reason of the hardening effect is the increasing in scalar dislocation density, the formation of two-phase regions of nanosized structure, a multi-stage process proceeds consisting in fracture (dissolution) of initial Fe_3C particles, carbon redistribution in the grain volume, and repeated precipitation of carbide particles. The structural phase transformations revealed in the steel suggest that treatment with a runaway electron preionized diffuse discharge produces thermomechanical action on the material.

The optical technique based on the relative radiation intensity of rotation structure of electronic-vibrational molecular transitions was used for measurement of gas temperature *Tg*. The values of *Tg* was ~ 80°K at condition under study [9], that was not sufficient for modification due to melting of metal surface layers. Electron beam also could not induce it because of its short time duration and low energy. Most likely microhardness was increased due to change in surface layers chemical composition by mixing the oxide and substrate layers after shock waves.

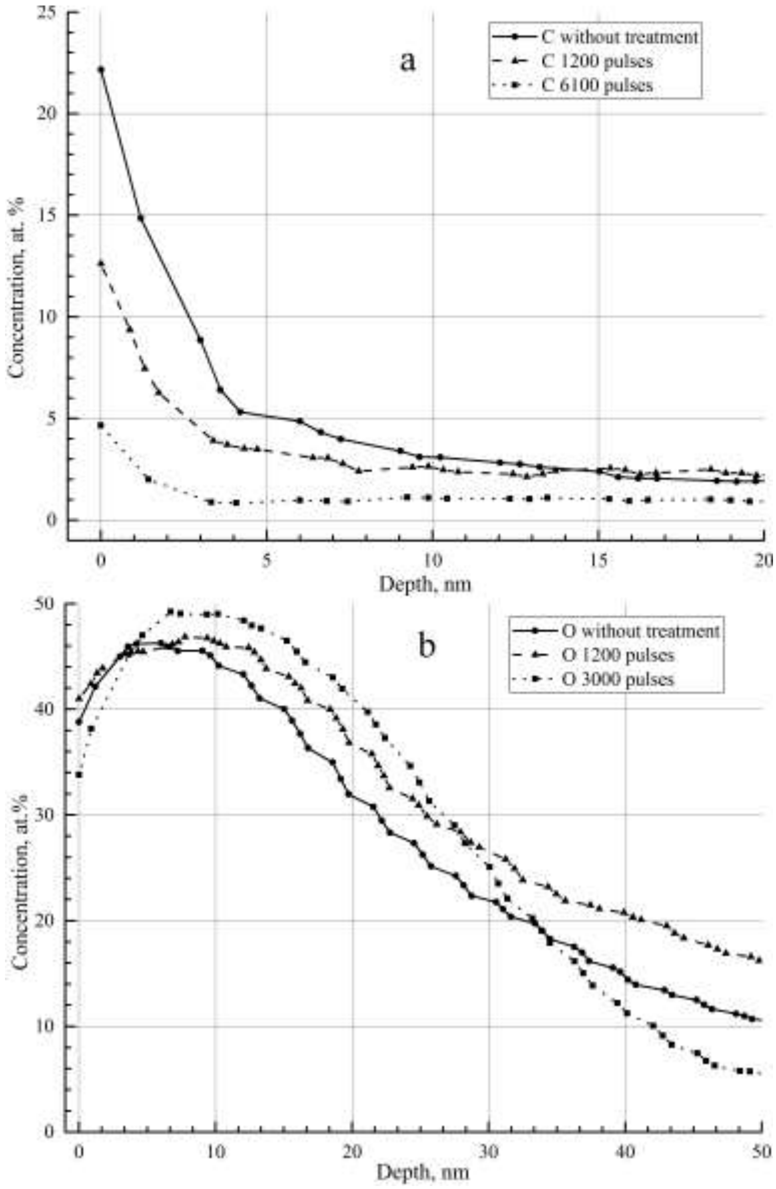

Figure 3. Carbon (a) and oxygen (b) concentration in near-surface layer of stainless steel before treatment and after treatment in air by REP DD with 1200 pulses and 6100 pulses.

4. Conclusion

Thus, experimental results of the action of a runaway electron preionized diffuse discharge show that at atmospheric pressure the planarization of the stainless steel surface, as well as its oxidation and cleaning from carbon took place. It was shown, that mechanical action of shock waves and interaction of near-surface layers with product of plasma chemical reactions produced in diffuse discharge leads to the substructure transformation and may induce hardening effect of stainless steel surface.

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