IOP Conf. Series: Journal of Physics: Conf. Series **1115** (2018) 032041 doi:10.1088/1742-6596/1115/3/032041

Modification of stainless steel by low-energy focused nitrogen ion beam

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Abstract. The results of experiments on the modification of SUS 321 stainless steel by a nitrogen ion beam extracted from a gas plasma of the non-self-sustained arc discharge with hot cathode PINK are presented. Extraction and focusing of the beam was carried out through a grid electrode of the definite curvature. When negative electric pulsed bias is applied to the grid electrode and a specimen located under the same potential, a ballistically focused nitrogen ion beam is formed. As a result of the processing, a nitride layer is formed on the surface of stainless steel with an increased hardness compared to the initial one.

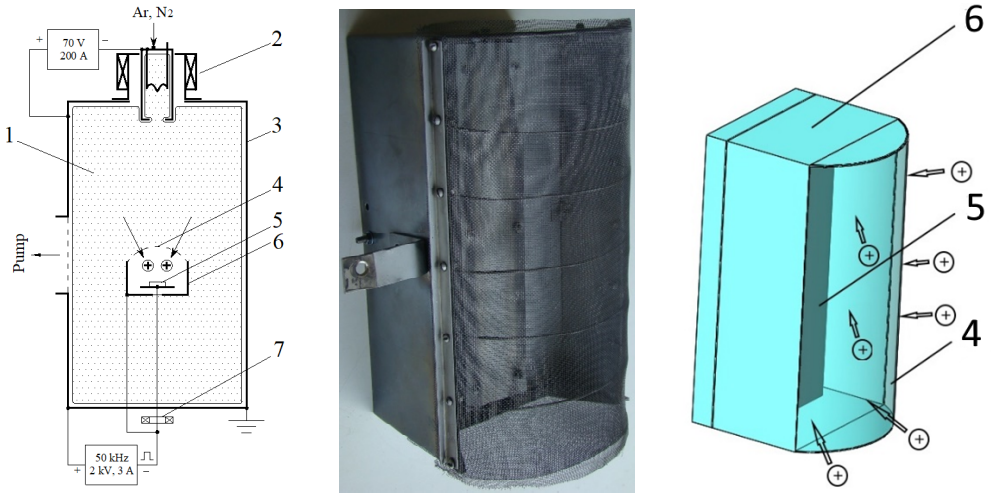
1. Introduction

Increasing the working properties (hardness, wear resistance, etc.) of products made of SUS 321 structural stainless steel, which is widely used in industry, is an important scientific and technical task. Among a wide range of modification methods, ion-plasma modification methods are the most promising and less resource-consuming [1-3]. They are aimed at imparting increased working properties to a thin near-surface layer of the workpiece. One of the most interesting methods is an ion implantation method [4] and, in particular, low-energy high-current plasma immersion implantation method [5-7].

This article presents the results of experiments on low-energy nitrogen ion implantation of SUS 321 stainless steel in the system with ballistic focusing of an ion beam formed from a non-self-sustained arc discharge plasma with a thermionic cathode. This work is a logical continuation of the experiments in the ballistic focusing system of a spherical configuration ion beam [8] with modernization of the electrode system into a cylindrical configuration, which allowed surface processing of a larger area.

2. Material and research technique

All experiments were performed on a plant with a vacuum chamber working volume of ≈0.9 m^3, which was previously evacuated by a turbo-molecular pump with a capacity of 1000 l/s to a pressure of at least 5×10^{-2} Pa. Gas plasma was generated by the PINK source [9] in the axial configuration (figure 1). The power supply of the PINK plasma generator was carried out from a welding transformer with an output voltage of up to 70 V and a current of up to 200 A. Heating of its tungsten thermionic cathode was carried out by a transformer with a thyristor current regulation and a frequency of 50 Hz. In this regard, during the experiments recording of the total current oscillograms of the accelerating voltage source and a collector current, as well as the shapes of the voltage pulses on the Rogowski coils by the LeCroy waveRunner 6050 oscilloscope, their averaging were performed by 200 measurements.



(a) (b) (c)

Figure 1. Scheme of the experiment (a), picture of an external view of the ion source (b) and its schematic 3D image (c). *1* – plasma, *2* – PINK plasma source, *3* – vacuum chamber, *4* – grid electrode, *5* – collector with specimen, *6* – ion beam forming system, *7* – Rogowski coil.

A ballistic focusing system of the ion beam was at the center of the vacuum chamber at a distance of 180 mm from the end of the hollow cathode of the PINK generator. It consisted of three main elements:

- focusing grid electrode which was a part of the cylinder with a radius of 75 mm and a length of 240 mm, covered with a grid with a cell of 0.5×0.5 mm and a transparency of 53%;
- a solid housing with a closed end whose dimensions were 240×120×100 mm, electrically connected to the grid electrode;
- a collector, electrically isolated from the housing, whose dimensions were 45×90 mm, the working plane of which was located in the optical focus of the grid electrode.

The ion beam was formed in a cathode layer formed in the generator plasma based on a non-self-sustained gas arc discharge with a hot cathode near the grid electrode when a negative voltage pulse was applied to it relative to the discharge anode. Accelerating voltage pulses were supplied from a specially developed source with an output voltage of up to 2 kV and a current of up to 3 A with the possibility of adjusting the frequency to 50 kHz and pulse duty factor from 15 to 80%. The ions, accelerating in the direction of the grid, partly fell on it, and partly flew into the equipotential space formed by the grid and the housing, where they continued to flow rectilinearly. The cylindrical shape of the grid caused the focusing of accelerated ions on the ballistic principle. This ion beam formation method ensures its focusing in proportion to the radius of the cylindrical grid electrode.

The specimens of 43×20×3 mm made of SUS 321 structural stainless steel were pre-wiped with bezine and placed on the collector of the ballistic focusing system, the temperature of which was measured with a chromel-alumel thermocouple. Before low-energy implantation, the specimens were cleaned and heated with 1-keV argon ions for 20 minutes. Implantation at an operating pressure of 0.5 Pa nitrogen for 1 hour was performed. After processing, the specimens in the working chamber to a temperature of not higher than 70°C with a chamber pressure of not higher than 5×10^{-2} Pa were cooled.

The specimen surface relief studies after the processing using a STIL 3D Micromeasure optical profilometer were performed. The surface hardness by a PMT-3M microhardnes tester was measured.

3. Results and discussion

The characteristic oscillograms of currents and voltages obtained in the experiment in figure 2 are shown.

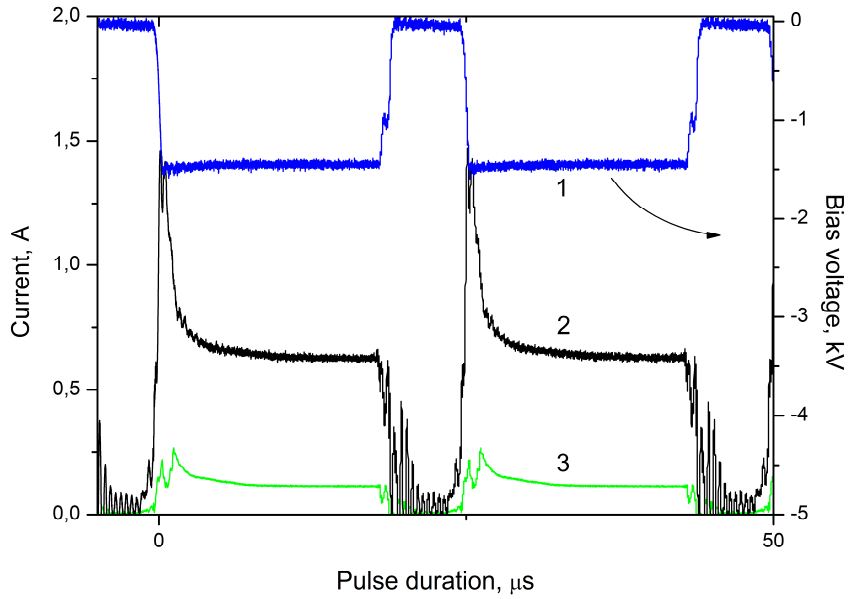


Figure 2. Oscillograms of the bias voltage pulses (1), of current to the ion source (2), of current to the collector (3).

The specimens temperature for the experiments was basic parameter. The temperature of 600°C on the literature data was chosen. The plasma density to maintain the constant specimens temperature, was varied by adjusting the discharge current of the PINK plasma generator and voltage pulse duty factor. Processing modes in table 1 are given. Ion energy, which was set by the pulse bias (1400 V) and repetition rate of the voltage pulses (40 kHz) remained constant. The fixed mean values of the ion current arriving at the collector in the same table are given.

Table 1. The specimen treating regimes.

Specimen number	Duty factor, %	Discharge current, A	Collector average current, мА	Total ion average current, мА
1	40	50	145	830
2	60	28	135	740
3	80	20	95	610

The analysis of the obtained data shows that total ion current and collector current are decrease due to PINK plasma generator discharge current decreasing and simultaneous bias pulses duty factor increasing.

The surface profile studies showed that during processing specimens were subjected to ion etching with different intensities (figure 3). An ion beam focusing improvement can explain this fact. This improvement is due to cathode layer width increasing, caused by plasma density decreasing with discharge current decreasing at constant bias. The etching cavity depth (in the region of the focus line) with increasing pulse duty factor (from 40 to 80%) is increases (from 25 to 50 μm) against the background of the initial specimens curvature.

Studies of the microhardness on the surface for each specimen, carried out at the center of the etching cavity and at a periphery of 6 mm from it at an indentation load of 50 g (table 2) showed that the hardness increased both at the center of the etching well and at its periphery. The highest hardness was observed on specimen No. 2. The lower hardness at the center of the etching well for all modes can be associated with a faster rate of the solid near-surface nitride layer etching formed during processing.

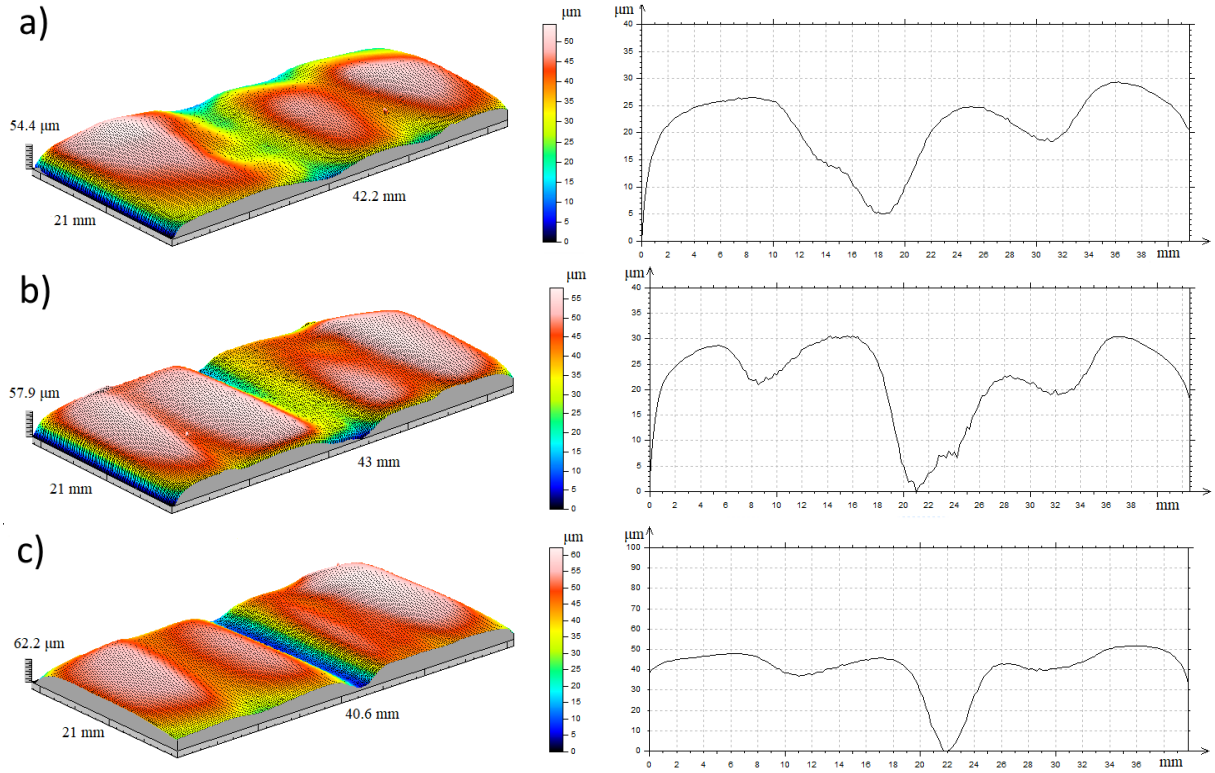


Figure 3. Specimen surface profiles: a) – specimen №1; b) – specimen №2; c) – specimen №3.

Table 2. The specimen surface microhardness (initial hardness 3 GPa).

Specimen number	Hardness, GPa	
	Center	Periphery
1	8.0	8.3
2	8.4	8.7
3	7.6	8.6

We cannot exclude effect of local overheating of the specimens in the focusing region, since the temperature was measured by a thermocouple mounted on the back of the specimen in the middle. In this situation, improving the ion beam focusing increases power density directly opposite the thermocouple. In this case, total power brought by the ion beam to the entire surface of the specimen decreases. This should cause an increase in the temperature gradient from the center of the specimen to its edges. However, more research is needed to confirm it.

Estimates of the modified layer depth at different indentation loads indicate that the thickness of the modified layer is not large and the penetration depth of the indenter does not exceed 10% of it. Thus, the thickness of the modified layer does not exceed 30 µm, or it significantly decreases in depth.

4. Conclusion

Processing of SUS 321 structural stainless steel was performed using the ion-beam low-energy nitrogen implantation method. It was shown that microhardness on the surface increased more than 2.5 times for one hour, and the surface underwent intensive ion etching with the formation of the well.

Maximum hardness on the surface was achieved not at the center of the etching well but at its periphery, which could be due to the factors including both ion beam local density and local surface temperature of the specimen.

Acknowledgments
The work was supported by the Russian Science Foundation (Grant No. 17-19-01169).

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