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Pulsed Glow Discharge Plasma Nitriding of Commercially Pure Titanium BT1-0

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Surface hardening of commercially pure titanium VT1-0 (Grade 2) by plasma nitriding within the DC glow discharge initiated in the pulsed and stationary modes is investigated. The experiments are carried out in pure nitrogen at a pressure in the reactor of 150 Pa and temperature of 450°C and 550°C with the treatment-duration changes from 3 to 9 hours. In these plasma-nitriding conditions, a pulsed glow discharge provides a more hardening effect with the hardness of the surface layer, which is by 15–25% higher than that obtained in a steady glow discharge and by 2–5 times higher than reference sample. As revealed, an increase in duty cycle from 40 to 80% at a pulse train frequency of 1 kHz during pulsed glow discharge plasma nitriding leads to an increase in the microhardness of a titanium surface by 25–30%.

Key words: plasma nitriding, commercially pure titanium, abnormal DC glow discharge, pulsed glow discharge, surface modification.

Досліджено поверхневе зміцнення технічно чистого титану ВТ1-0

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плазмовим азотуванням у жеврійному розряді постійного струму, що ініціюється як в імпульсному, так і в стаціонарному режимах його горіння. Експерименти проводили в середовищі чистого азоту за тиску в реакторі у 150 Па і температури у 450°C і 550°C зі зміною тривалости оброблення від 3 до 9 годин. Показано, що в цих умовах плазмового азотування імпульсний жеврійний розряд забезпечує більш високий зміцнювальний ефект із твердістю поверхневого шару, яка на 15–25% перевищує твердість, одержану в постійному жеврійному розряді, і в 2–5 разів перевищує твердість поверхні контрольного (необробленого) зразка. Виявлено, що збільшення шпаруватости від 40 до 80% за частоти ланцюга імпульсів у 1 кГц під час плазмового азотування в імпульсному жеврійному розряді приводить до підвищення мікротвердости поверхні титану на 25–30%.

Ключові слова: плазмове азотування, технічно чистий титан, аномальний жеврійний розряд постійного струму, імпульсний жеврійний розряд, модифікування поверхні.

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1. INTRODUCTION

As far as it is known, plasma nitriding is one of the widespread techniques for surface modification of metals. It improves their hardness, tribological properties, corrosion behaviour, *etc.* [1]. The hardening effect of the titanium surface during plasma nitriding is achieved mainly by the formation of a compound layer and following diffusion zone with a predominant content of titanium nitrides TiN, Ti_2N, and nitrogen stabilized α-Ti phase [2].

Normally, for plasma generating while nitriding process the stationary abnormal glow discharge with the constant DC power supply is widely used. However, the long-term industrial application of DC glow discharge initiated in a stationary mode for the plasma-activated thermochemical processes, some specific disadvantages have been revealed. Among them, one of the biggest problems is the possibility of a partial or complete loss of glow discharge`s stability related to the presence on a treated surface of different inhomogeneity in the form of narrow slots and gaps [3]. As it has been already reported in Refs. [4, 5], at a certain condition, these can lead to both the appearance of a 'hollow cathode effect' or even a transition to an electric arc as a more stable form of a gas discharge. All of these inevitably lead to the uncontrollable localized overheating of the treated surface due to the spike of electric field strength in the arcing region resulting in the melting of a workpiece surface and violation of the plasma-nitriding process as a whole [6].

To eliminate the above-mentioned issues, the use of pulsed DC glow discharge (pulsed GD) devices while plasma nitriding become one of the

modern trends. As was shown in Ref. [7], during pulsed GD the power isn`t applied constantly but periodically with a certain frequency. This benefits us in many ways. Firstly, adjusting the pulse duration and duty cycle of discharge current allows us to eliminate of an arcing phenomenon on a sample surface with the more precise controlling of treating temperatures without overheating. Secondly, it is believed that the pulsed GD provides a higher hardness of nitrided surfaces because of the denser-plasma formation with a higher concentration of ionized active component near the treated sample in comparison with stationary DC glow discharge [8]. The thing is to ensure the duration of the pause between pulses, which does not lead to a dramatic decrease in the concentration of plasma ions on the surface of the samples.

Thus, the aim of the present work was to compare the effect of plasma nitriding in pulsed and stationary modes of DC glow discharge on the structure, hardness and surface morphology, particularly roughness of the commercially pure titanium BT1-0 (grade 2).

2. EXPERIMENTAL PROCEDURE

The research was carried out on samples of commercially (technically) pure titanium BT1-0 (grade 2) measuring 30×15×0.8 mm cut from the same ingot. Before nitriding, the samples were mechanically grounded with sandpaper number 4 and 5, followed by polishing to a mirror surface with diamond paste of 1 μm.

Plasma nitriding was carried out both in stationary and pulsed DC glow discharge in pure nitrogen (100% N_2) at gas pressures in the reactor of 150 Pa. The titanium samples were subjected to the plasma nitriding at a temperature of 450°C and 550°C during 3, 6 and 9 hours. To ensure the selected temperature during plasma nitriding in a stationary GD, the discharge current varied in the range of 260–310 mA, while the voltage drop varied from 320 V up to 380 V. The frequency of the rectified current in this case was of 100 Hz. It should be noted that, during stationary mode of glow discharge while treatment (without modulation), GD was powered by full wave rectifier without smoothing filter in the circuit. In this case, in a part of every wave, the discharge current will flow in decaying plasma partially limiting the probability of an electric arc appearance and its subsequent stabilization.

The pulsed GD plasma nitriding was carried out at a pulse train frequency of 1 kHz, and the duty cycle of 40% and 80% (pulse duration of 400 and 800 μs, respectively). In the pulsed mode of GD, the voltage drop on the electrodes was a bit higher and amounted to 370–460 V, while the average discharge current does not exceed 220 mA. Meanwhile, the pulse current increases up to 410–650 mA with the increase of the duty cycle. The generation of current pulses of a

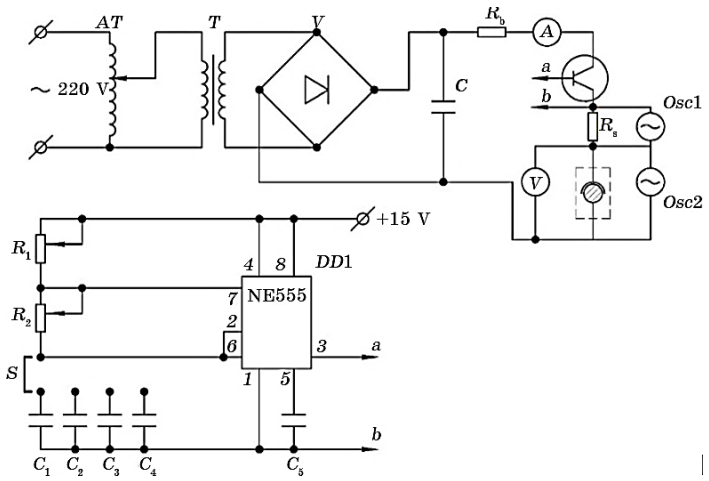

Fig. 1. Glow discharge installation scheme (*a*) and electrical scheme of pulse modulator (*b*): *AT*—autotransformer, *T*—transformer, R_b—ballast resistor, R_s—shunt, *V*—rectifier, *Osc*1, *Osc*2—oscilloscopes, NE555—integrated circuit (timer), *S*—switcher, *DD*1—supply voltage, R_1—pause-duration regulator, R_2—pulse-duration regulator, C_1–C_4—timing capacitors.

given frequency and duration was provided by a corresponding pulse modulator, the electrical scheme of which, along with the GD installation electrical scheme, is shown in Fig. 1.

The main control component is the NE555 integrated circuit, which is used in monostable mode, is timer. Resistor R_1 acts as a pause duration regulator, and R_2 as a pulse duration regulator. To increase of accuracy and prevent the impact of external disturbances, control pin 5 was shunted by capacitor C_5. After applying power, the voltage relative to grounding on pin 3 will be about of 0 V. The timing capacitors C_1–C_4 are completely discharged and the circuit can be in this state until a positive signal arrives at the start pin 2. Its value should be three times less than the supply voltage DD1. After applying power to pin 2, a voltage similar to the supply voltage appears at the output of the circuit. Its time depends on the charge time of C_1–C_4 up to 2/3 from DD1, through resistors R_1 and R_2. As soon as this happens, the output voltage will drop to 0 V and C_1–C_4 will discharge. The generator pulse forms that were used in the experiments are given in Fig. 2.

Microstructural analysis of nitrided samples was carried out using an inverted metallographic microscope PW-1300M, which provides a maximum magnification of up to 900 times with built-in digital screen capture equipment. Microhardness profiles of plasma-nitrided titanium BT1-0 samples were obtained by using PMT-3M microhardness tester in accordance with the ISO 6507 (DIN 50190)

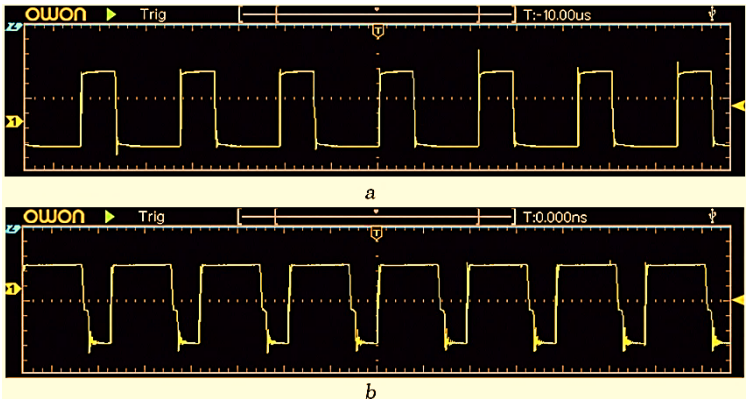

Fig. 2. The oscillogram of the voltage pulses shape of the glow discharge with the frequency of 1 kHz: *a*) 40% of duty cycle, *b*) 80% of duty cycle.

standard. Determination of surface roughness (*R*a) of nitrided samples was carried out by using TR-200 profilometer.

3. RESULTS AND DISCUSSION

Figures 3 and 4 show the microstructures of the c.p. titanium BT1-0 surface after plasma nitriding depending on treatment temperature and time, respectively. Analysing of our microscopic observations, it can be noted that an increase in both the temperature from 450°C to 550°C and nitriding time from 3 to 9 hours leads equally to the growth of α-Ti grains.

It should be noted that pulsed GD plasma nitriding leads to a more significant grain growth of the α-titanium matrix. The latter might be evidence of a more significant surface hardening, since, as is known, the grains growth leads to an increase in surface hardness consequently.

In turn, in the number of works [9–12], analysing the XRD patterns of Grade 2 titanium after plasma nitriding on a similar mode, the authors note the presence in the modified layer of nitride phases mainly ε-Ti_2N and δ-TiN along with grains of titanium matrix. They have also reported the α-Ti grain growth with increasing both treatment temperature and time. The x-ray diffraction pattern of the pulsed GD plasma-nitrided titanium BT1-0 is given in Fig. 5.

Figure 6 illustrates the effect of nitriding temperature and time on the hardness of the titanium BT1-0 surface plasma-nitrided in a stationary and pulsed DC glow discharge.

By analysing the obtained results, it can be concluded that the formation of the above-mentioned nitride phases on the titanium surface during plasma nitriding in a glow discharge leads to

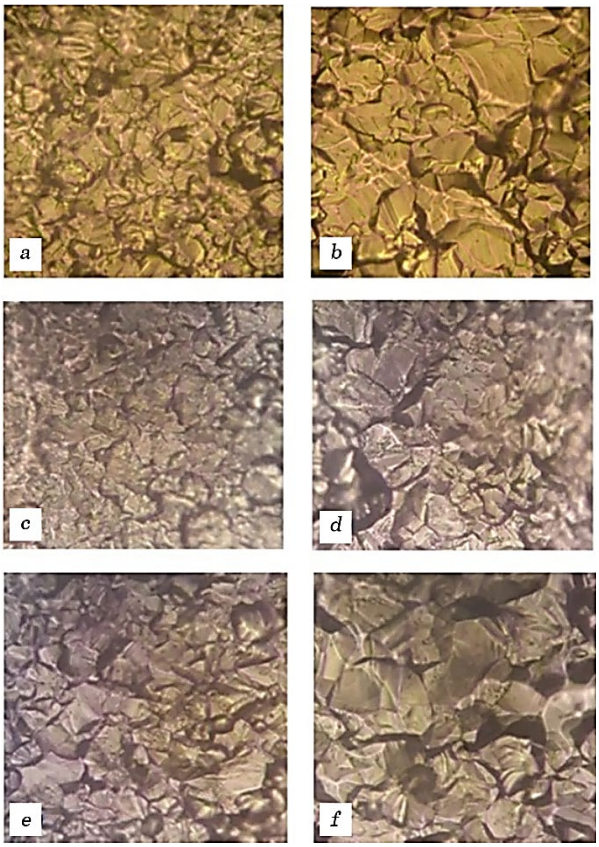

Fig. 3. The surfaces microstructure of titanium BT1-0 samples plasma-nitrided: at a temperature of 450°C (*a*, *c*, *e*) and 550°C (*b*, *d*, *f*), in stationary DC glow discharge (*a*, *b*) and in pulsed DC glow discharge with a pulse frequency $f = 1$ kHz and the duty cycle of 40% (*c*, *d*) and 80% (*e*, *f*). Treatment time is of 9 h. (×600).

significant surface hardening. Thus, in our experiments, increasing in nitriding temperature from 450°C to 550°C during 9 hours led to an increase in the surface hardness from 438 $HV_{0.05}$ to 1010 $HV_{0.05}$ for stationary GD and from 589 $HV_{0.05}$ up to 1190 $HV_{0.05}$ for pulsed GD respectively (Fig. 6, *a*). It should be noted that obtained results of plasma-nitrided BT1-0 samples are 2 to 5 times higher than the microhardness of the reference (untreated) sample (228 $HV_{0.05}$). It was also noticed that the hardness of the samples nitrided in a pulsed GD is 15–25% higher than those, which are nitrided in a glow discharge initiated in a stationary mode.

At the same time, there is an increase in the surface microhardness of titanium samples plasma-nitrided in a pulse GD with a change in the

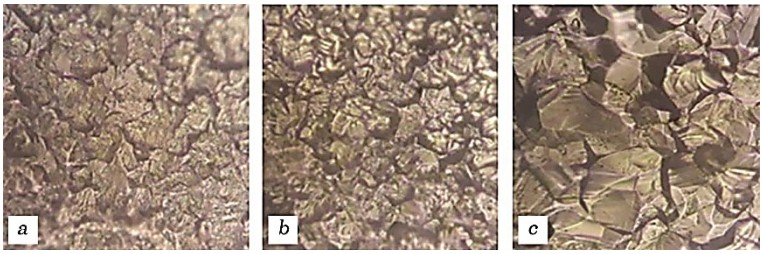

Fig. 4. The surfaces microstructure of titanium BT1-0 samples after nitriding in a pulsed GD at a temperature of 550°C for: *a*) 3 hours, *b*) 6 hours, *c*) 9 hours. (×600).

duty cycle was also observed (Fig. 6, *b*). Thus, in our experiments, increasing the duty cycle from 40% up to 80% at a pulse frequency of 1 kHz while plasma nitriding at a temperature of 550°C resulted in an increase in the surface microhardness by 25–30% for all nitriding durations. It can be explained by an increase in the concentration of nitrogen ions (N_2^+) on the surface of the cathode due to an increase in the number of collisions with the atoms of the active gas or neutrals when the pulse duration goes up.

Thus, the increase in the duty cycle during pulsed GD plasma nitriding provides the rise in the number of N_2^+ bombarding the cathode, thereby enhancing radiation-stimulated diffusion on its surface.

In work [13], it was also reported on the increase in the concentration of ionized active species (N_2 and N_2^+) along with the surface microhardness of AISI H13 steel plasma-nitrided in pulsed GD,

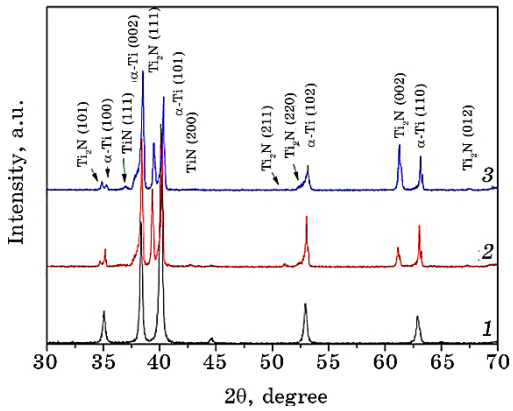

Fig. 5. XRD patterns of plasma-nitrided titanium BT1-0 at 600°C: *1*—reference sample, *2*—sample nitrided at a stationary mode, *3*—sample nitrided at a pulsed mode of GD [9].

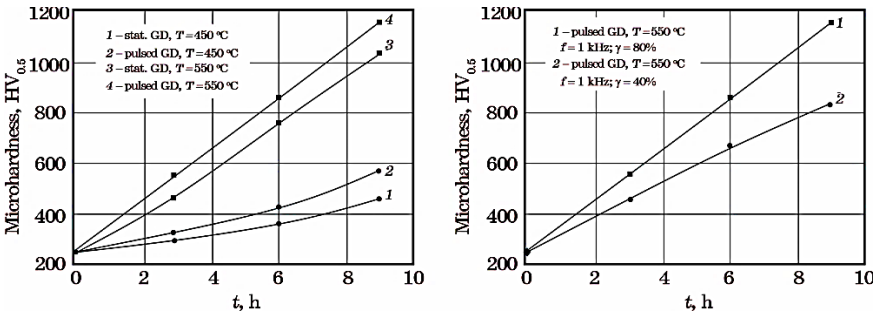

Fig. 6. The dependences of microhardness of titanium BT1-0 samples plasma-nitrided in a stationary and pulsed DC glow discharge on nitriding temperature and time (*a*) and duty cycle at a fixed temperature of 550°C (*b*).

when the duty cycle goes up from 25% to 75%.

The distribution of microhardness over depth of the modified layer of plasma-nitrided titanium samples at different modes is shown in Fig. 7. The obtained microhardness profiles illustrate standard behaviour for the nitriding mechanism with a sharp decrease after the maximum microhardness at the modified surface to the value of the metal matrix. Thus, the maximum thickness of the diffusion zone of the nitrided layer of about 20 μm was obtained after nitriding in plasma of pulsed GD at a temperature of 550°C for 9 hours with a pulse frequency of 1 kHz and a duty cycle of 80%. This correlates with the results obtained in Ref. [14].

Investigating the effect of pulse duration on the microstructure of plasma-nitrided AISI 409 steel, the authors have noted the greatest increase in the thickness of the diffusion zone at a duty cycle of 80–100%.

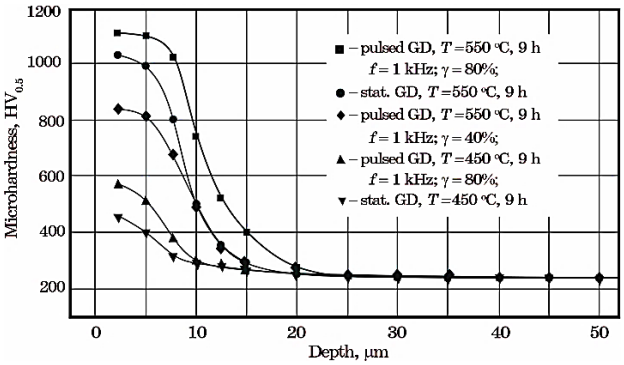

Fig. 7. The microhardness profiles of plasma-nitrided titanium BT1-0 samples at different modes.

In turn, the smoothness of the surface is as important a parameter as its high hardness, specifically in conditions of dry friction. There is a known general tendency to increase in the surface roughness (*Ra* and *Rz*) of titanium and its alloys with increasing nitriding temperature and time [15, 16]. In work [17], it is shown that an increase in the treatment time from 10 to 24 hours at a fixed temperature of 720°C while plasma nitriding of c.p. titanium grade 2 resulted in an increase in roughness of the modified surfaces from $Ra \cong 3.30$ μm (of the reference sample) up to $Ra \cong 4.59$ μm–$Ra \cong 7.26$ μm, respectively. At the same time, the authors of work [18] have reported a sharp increase in the surface roughness of plasma-nitrided Ti–6Al–4V titanium alloy from $Ra \cong 0.1$ μm (of the reference sample) up to $Ra \cong 0.21$–0.5 μm with increasing of nitriding temperature in the range of 600–800°C. Such a significant difference in *Ra* value in the works mentioned above is more likely associated with the difference in the dimensions of the treated workpieces. In this case, to provide of required nitriding temperature for the bigger samples, the energy parameters of a glow discharge (discharge current, current density and voltage drop) should be also increased. The latter results in a more roughening of a treated surface. In turn, there is no information specifically on the impact of the duty cycle on the roughening surface of plasma-nitrided titanium in pulsed GD plasma.

This paper provides the comparison results of the effect of a pulse frequency and the duty cycle on the surface roughening of nitrided titanium BT1-0 in plasma of a glow discharge, initiated both in steady and pulsed mode. The surface profiles of c.p. titanium were investigated after plasma nitriding at a temperature of 550°C for 9 hours. The research was carried out with a contact profilometer TR-200 at a distance of 15 mm. The corresponding surface profiles are given in Fig. 8.

Analysing the obtained surface profiles of c.p. titanium BT1-0 plasma-nitrided in a stationary GD at a frequency of 100 Hz, discharge current of 310 mA and voltage drop on electrodes about 380 V allowed us to classify this treatment as one that least distorts the surface morphology. Under these conditions, the surface roughness increases from $Ra = 1.2$ μm for the reference sample up to $Ra = 2.1$ μm. Instead, nitriding in a pulsed GD at a current frequency of $f = 1$ kHz leads to a greater distortion of the titanium surface microrelief. It should be noted that the surface roughness increases up to $Ra = 2.8$ μm and $Ra = 3.9$ μm with the decrease in the duty cycle from 80% to 40%, respectively (Fig. 8). Such an effect can be described with the fact that pulsed GD operating at higher voltages compared with the stationary one at the same discharge power. Thus, while the constant current the reduction of the duty cycle leads to increasing of a cathode voltage to maintain the current.

As far as it is known, the discharge voltage, specifically, cathode

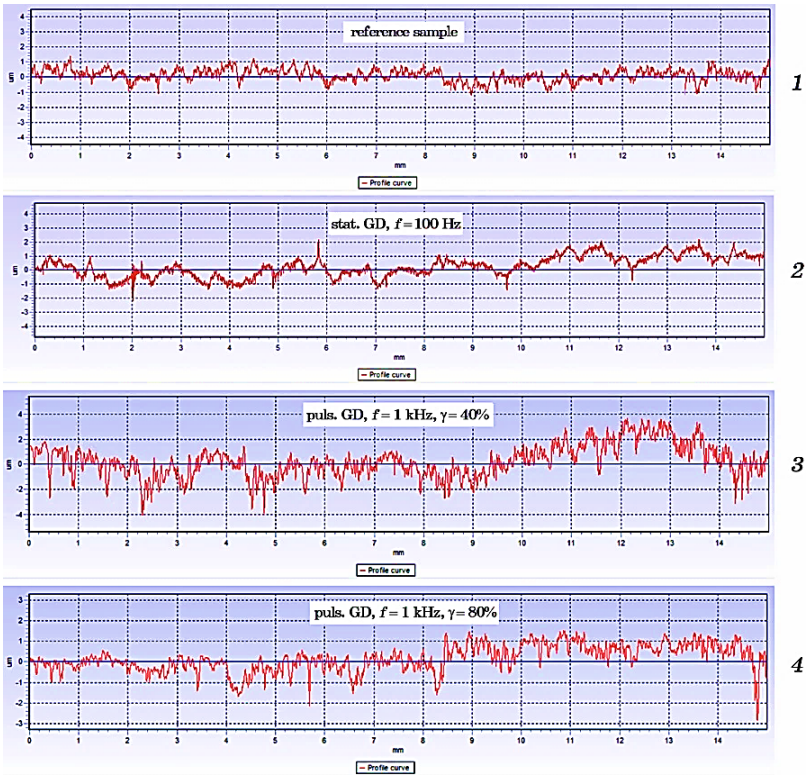

Fig. 8. Surface profiles registered with the contact profilometer of commercially pure titanium BT1-0 plasma-nitrided in a glow discharge both in steady and pulsed mode at a temperature of 550°C for 9 hours.

voltage drop (U_c), determines the ions' energy while the discharge current determines their amount in a plasma, given the fact that the pulsed GD during plasma nitriding burns at a lower average current. However, at the same time, the pulse current is 2 or even 3 times higher than the average one and those, provided by the stationary GD. Additionally, as it was mentioned above, the glow discharge voltages in a pulsed mode are higher rather than in a steady one.

With the purpose to confirm this theory, the calculation of average ion energy E_i, bombarding the cathode surface while plasma nitriding in abnormal DC glow discharge both in pulsed and steady modes was conducted. The calculations were carried out by certain transformations of the model proposed by Davis and Vanderslice [19]:

$$\frac{dN}{dE_i} = \frac{N_i}{2}\frac{L}{\lambda}\left[1-\frac{E}{U_c}\right]^{1/2} \exp\left[-\frac{L}{\lambda}+\frac{L}{\lambda}\left(1-\frac{E}{U_c}\right)^{1/2}\right], \qquad (1)$$

where N_i is the number of ions entering the cathode dark space, E_i—ion energy, U_c—cathodic potential drop, λ—the free-path length of the ions, L—the width of the cathode dark space.

Then, the average energy of ions bombarding the cathode surface can be defined as:

$$E_i = U_c \left[2\frac{\lambda}{L} - 2\left(\frac{\lambda}{L}\right)^2 + 2\left(\frac{\lambda}{L}\right)^2 \exp\left(-\frac{\lambda}{L}\right) \right]. \quad (2)$$

In turn, the dark cathode-space length is determined by the Child–Langmuir equation [20]:

$$L = \left[\frac{4\varepsilon_0}{9J}\right]^{1/2} \left[\frac{4q}{M}\right]^{1/4} U_c^{3/4}, \quad (3)$$

where J is discharge current density, q—electron charge ($1.6 \cdot 10^{-19}$ C), M—ion mass (for nitrogen ions, $M = 4.66 \cdot 10^{-26}$ kg), ε_0—vacuum permittivity ($8.85 \cdot 10^{-12}$ F·m^{-1}).

At the same time, the free-path length of ions in the gas medium will depend on the gas pressure in the reactor and is defined as follows:

$$\lambda = kT/(P\sigma), \quad (4)$$

where k is Boltzmann constant ($1.3 \cdot 10^{-23}$ J·K^{-1}), P—gas pressure (150 Pa or 1.125 Torr), σ—ionization cross-section ($2.5 \cdot 10^{-17}$ cm^2), T—cathode temperature (723 K).

The calculations showed that the plasma nitriding in a stationary GD provides the average energy of ions (N_2^+), which bombard the cathode surface, at the level of 47 eV with the accelerating voltage on the cathode (U_c) of about 280 V. Meanwhile, during plasma nitriding in a pulsed GD, the average ions energy at a level of 97 eV for 40% duty cycle ($U_c = 400$ V), which is two times higher than E_i value, obtained in stationary GD and about 62 eV for a 80% duty cycle ($U_c = 340$ V).

In spite of the fact that the pulsed GD plasma nitriding with the frequency of 1 kHz and the 40% of duty cycle provides a higher ions' energy, it releases a more dramatic invasion on a titanium surface. Instead, pulsed plasma nitriding while 80% of the duty cycle resulted in a higher level of titanium surface hardness is mainly because of the longer acting time of glow discharge. The latter provides a higher ionization rate at the higher average discharge current.

4. CONCLUSIONS

The plasma-nitriding process of c.p. titanium BT1-0 in abnormal DC glow discharge, initiated in a stationary and pulsed mode was

investigated. It was established that increasing in nitriding temperature from 450°C to 550°C and time from 3 up to 9 hours resulted in a dramatic increase in surface hardness up to 1010 $HV_{0.05}$ and 1190 $HV_{0.05}$ for stationary and pulsed glow discharge respectively in comparison with the reference sample (228 $HV_{0.05}$).

It was also established that an increase in a duty cycle from 40% down to 80% while plasma nitriding in pulsed glow discharge at a fixed temperature of 550°C with the pulse frequency of 1 kHz provides an increase in a surface hardness by 25–30%.

The pulsed glow discharge of 1 kHz train frequency with the 80% of duty cycle provides a maximum depth of diffusion zone of plasma-nitrided c.p. titanium BT1-0 of about 20 μm. Instead, because of higher pulse voltage while pulsed GD plasma nitriding with the 40% of duty cycle, such a treatment releases as more invasive on a titanium surface with a surface roughness about $Ra \cong 3.9$ μm that is two times higher rather than in stationary mode ($Ra \cong 2.1$ μm).

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