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Research

INFLUENCE OF REPETITIVELY PULSED NEGATIVE BIAS PARAMETERS ON MACROPARTICLE SURFACE DENSITY

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ABSTRACT

The results of an experimental study of the influence of a substrate negative bias with various pulse widths and pulse repetition rates ranging from several Hz to 105 Hz on the macroparticle (MP) accumulation on substrate immersed in a DC titanium vacuum arc plasma are presented. It was found that the rate of MP deposition on the substrate surface depends significantly on the bias pulse parameters and the processing time. The influence of the multiple recharging of MPs in the plasma and the sheath on the reflection of these MPs in a sheath electric field is discussed.

INTRODUCTION

The vacuum arc plasma is characterized by the high degree of the ionization of cathode material erosion products (Plyutto, *et al.*, 1965; Lunev, *et al.*, 1976; Kondrat'eva, *et al.*, 1999). This benefit provides the wide practical application of vacuum arc plasma for different multifunctional ion implantation and coating deposition technologies (Sanders, 1989; Boxman, *et al.*, 1995; Randhawa, 1988; Sharkeev, *et al.*, 1995; Nikolaev, 2012). The main disadvantage of vacuum arc discharge, which considerably limits its applications, is the presence of significant amount of microdroplets often referred to as macroparticles (MPs). MPs have size 0.1 µm to 100 µm at velocities from 1 m/s to 800 m/s (Daalder, 1976; Rysanek, et al., 2008). The presence of MPs in the metal plasma leads to the creation of pores, the degradation of coating homogeneity and properties and a significant increase in their roughness.

A number of different MP filtering systems have been proposed and developed in order to obtain MP – free dense metal plasma. Nevertheless, an efficiency of vacuum arc plasma transportation throw the filters are low (Aksenov, *et al.*, 1978; Anders, 1999; Ryabchikov, *et al.*, 2005).

Many different approaches based on vacuum arc properties have been proposed and used to decrease MP number density in the coatings (Swift, 1996; Bizyukov, et al., 2006; Proskurovsky, et al., 2007).

The authors of several works observed the MPs density reducing effect on the TiN coating surface by 3-4 times with increasing DC negative bias potential up to $\varphi = -1$ kV on the substrate immersed in the vacuum arc plasma (Tau, *et al.*, 1990; Keidar, *et al.*, 1999).

Works (Zhanga, *et al.*, 2012; Ryabchikov, *et al.*, 2014; Stepanov, *et al.*, 2016) described investigation of the possible enhancement of the MPs number decreasing effect by using high-frequency short-pulse negative bias with amplitude up to 3.2 kV.

This paper deals with the study of control possibility of titanium MP surface number density depending on processing time and repetitively pulsed bias parameters including bias pulse repetition rate and pulse width.

MATERIALS AND METHODS

The experimental setup scheme is presented in Fig. 1. The vacuum arc and gaseous plasma sources were installed on the side flange of experimental setup.

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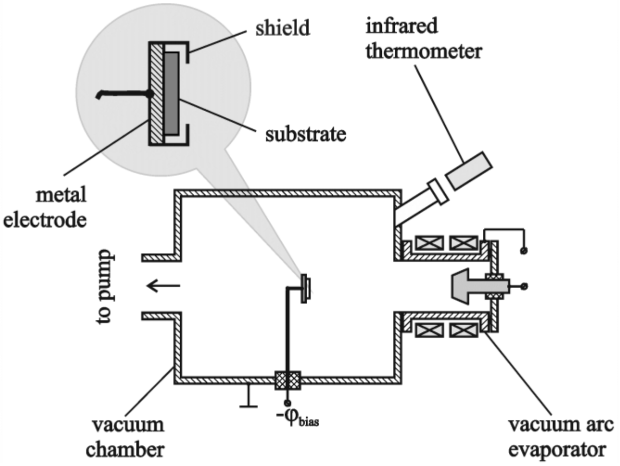

Fig. 1 The experimental installation scheme.

Titanium has been used as a cathode material. The variation of plasma density near the substrate surface was realized by means of the sample-plasma sources distance control.

For carrying out the experimental investigations, two short-pulse negative bias generators were used. The first generator had fixed pulse width of 7 µs. The pulse repetition rate of this generator could be varied from 10 Hz to $2.5 \cdot 10^4$ Hz. The second generator had a fixes pulse frequency of 10^5 Hz with a possibility to control the pulse width from 1 µs to 9 µs. The negative bias pulse amplitude of the generators could be varied from 0.5 kV to 3.5 kV. Stainless steel and titanium have been used as substrates materials. The surface of samples has been polished (R_a=0.035 µm). The substrates surface was investigated using optical and electron microscopes.

Experimental data on MP number surface density change are presented in the figures in absolute units and in the form of MP relative number surface density $k=N/N_0=n/n_0$, where $N=n \times S$ is the quantity of MPs located on the chosen area of a substrate (S) at negative biasing; n is the number surface density of MPs at negative biasing; $N_0=n_0 \times S$ is the quantity of MPs on the chosen area of a substrate at anode potential (φ_b=0 V) at the chosen vacuum arc plasma deposition time; n_0 is the number surface density of MPs at anode potential biasing on the substrate at a chosen time of metal plasma deposition; S is chosen for the statistically reliable counting of the MP quantity on substrate surface. For each experimental point total area for MP counting made 6×10^4 µm^2.

RESULTS AND DISCUSSION

A photograph of a typical substrate surface using electron microscope Hitachi TM-1000 after MPs and plasma deposition is presented in Fig. 2.

Fig. 2 shows that a lot of the small size MPs on the sample look like a sphere. At the same time, the MPs with diameter 3 µm and more is partly flattened.

The experiments with pulse repetition rates influence to MP number decreasing were carried out at the amplitude of the negative bias potential of 2 kV and pulse duration of 7 µs. Pulse frequency of the bias potential has been varied from 10 Hz to 10^5 Hz. Stainless steel or titanium samples of 2 cm^3 × 2 cm^3 × 0.3 cm^3 in size were installed at 23 cm from cathode surface of vacuum arc plasma source. In all experiments, the processing time was 30 s. The experimental data of the MP amount on the sample versus pulse frequency are presented in Fig. 3. Increasing the bias pulse frequency from 10 Hz to 10^4 Hz led to a gradual reduction in the MP number acquisition on the target. However, this decrease did not exceed 20%. The character of the curve varies considerably in the bias-pulse frequency range of

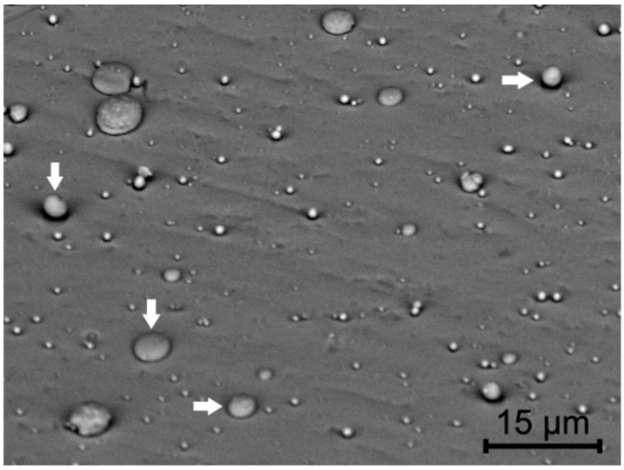

Fig. 2 Microphotograph of a sample with different forms of MPs (titanium plasma, φ_b=0 V, deposition time is 3 min).

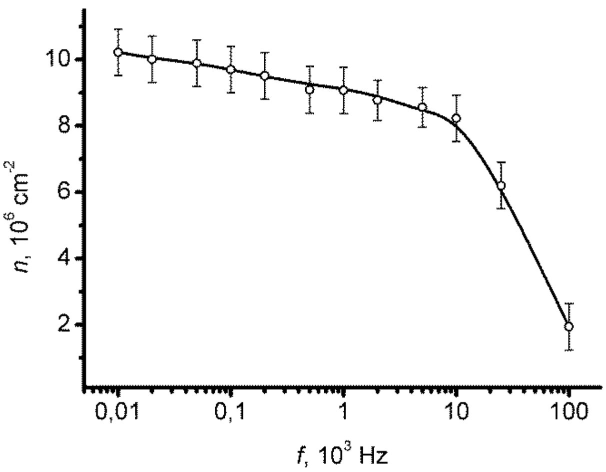

Fig. 3 MP number density (n) versus pulse repetition rate (f) at φ_b=–2 kV, a processing time of 30 s, and a cathode–steel substrate distance of 23 cm (j_i=10 mA/cm^2).

10^4 Hz to 10^5 Hz. In this frequency range of the bias pulses, the surface MP number density decreased 5-fold.

The experimental data concerning the decrease in the Ti MP acquisition versus the bias pulse duration at φ_b=–2 kV after 90 s of sample treatment has been carried out at cathode-substrate distance 38 cm (Ti ion plasma saturation current density j_i=4.4 mA/cm^2, and Ar plasma saturation current density j_i=1.4 mA/cm^2) are presented in Fig. 4. Fig. 4 indicates that the MP accumulation on a substrate strongly depends on the bias pulse width.

Let's consider the differences between DC and repetitively pulsed negative substrate biasing. Two processes occur when negatively charged plasma MPs enter the sheath. On the one hand, the velocity of the MPs decreases because of the interaction of negatively charged MPs with the sheath electric field. On the other hand, after entering the sheath, the MPs will be gradually recharged, both by the ions that interact with them and because of the ion–electron emission and thermionic emission from the surfaces of the MPs. In the sheath ions extracted from plasma boundary have rather high directed energy and, consequently, a directed velocity (~1.5 × 10^6 cm/s) that is two orders of magnitude higher than the velocity of the fastest MPs. Therefore, the positive charge stored on the MPs will depend on the sheath ion current density and the MPs cross section. If only the recharging of the MPs caused by the ion flux is taken into consideration, then as a first approximation, the time required for negative charge compensation on the MPs in the sheath can be estimated as follows:

$$\tau_{re} = \frac{Q}{z \cdot e \cdot n \cdot \upsilon_\partial \cdot (\pi \cdot R^2)} \quad (1)$$

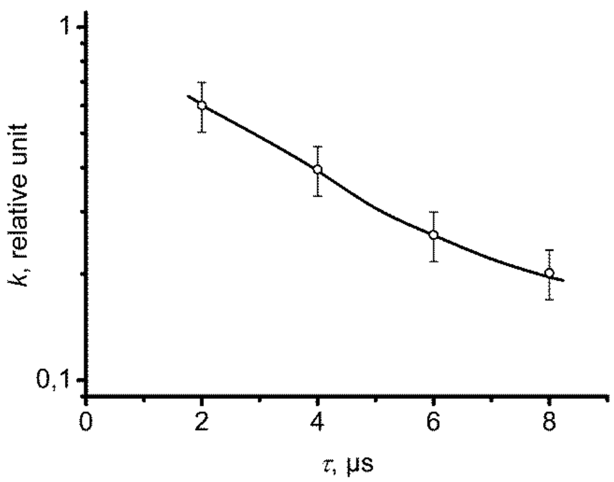

Fig. 4 Reduction in the MP number acquisition on the target versus bias pulse length (τ) for φ_b=–2 kV, a processing time of 1.5 min and a cathode–titanium substrate distance of 38 cm.

where z is the average charge state of the ions, e is the electron charge, n is the plasma density, υ_p is the plasma jet velocity, $Q \approx 4\pi\varepsilon_o R\varphi_{MP}$ is the negative charge accumulated by an MP in the plasma, R is the radius of an MP, φ_{MP} is the MP potential, and ε_o is the dielectric permeability.

According to Eq. 1, the recharge time for MPs in the sheath is approximately 10 μs at a plasma density of ~10^{10} cm^{-3}. If an MP has not yet left the sheath before its negative charge is entirely lost, then it begins to gain a positive charge in the sheath and accelerates toward the dc-biased substrate surface. A thickness of the sheath s can be estimated from Child low of space-charge-limited current in a plane diode:

$$s = \left(\frac{4}{9} \frac{\varepsilon_0}{j_i} \left(\frac{2ze}{m_i} \right)^{\frac{1}{2}} U^{\frac{3}{2}} \right)^{\frac{1}{2}} \quad (2)$$

where m_i is the ion mass, U is the bias amplitude.

If an MP has an initial velocity of ~4 m/s, then it will penetrate a sheath with a thickness of ~4 mm within approximately 10^{-3} s. According to Eq. 1, during this time, the MPs can accumulate a positive charge with an absolute value that is two orders of magnitude greater than the negative charge accumulated in the plasma (during the same time). Thus, the MPs can be significantly accelerated as it approaches the substrate surface.

A different situation can arise in the case of the repetitively pulsed biasing. In this case, the MP, while approaching the substrate surface, will alternate between being located in the sheath (during the bias pulses) and in the plasma (in the intervals between pulses). In the sheath, the MP loses its negative charge as estimated by Eq. 1 for approximately 10 μs, which is comparable to the bias pulse duration in our experiments (7 μs). After the bias pulse cut off, the MP will again be located in the plasma, where it can get an additional negative charge. The droplet charging time in plasma is defined by the precise equality of the electron and ion currents to MP. For micron-sized droplets in a titanium arc plasma with a density of 10^{10} cm^{-3} this time will be several microseconds. It is evident that the MP recharging time in the plasma is comparable to the minimum interval between pulses (3 μs) at f=10^5 Hz in our experiments. Therefore, at a bias pulse repetition rate of 10^5 Hz, an MP with the initial velocity of 4 m/s penetrating a sheath with the thickness 4 mm will be subjected to approximately 100 recharging cycles in the sheath and in the plasma. Thus, upon the application of the repetitively pulsed biasing, repeated exposure of the sheath electric field to a negatively charged MP is possible.

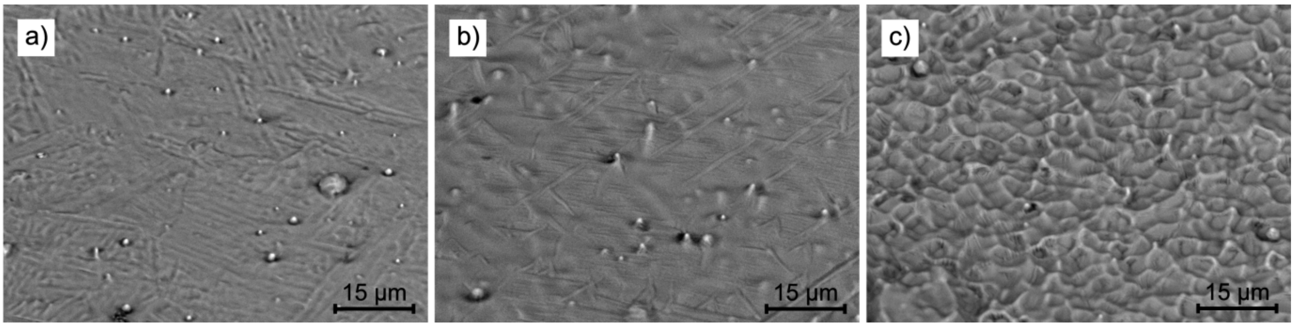

Fig. 5 Microphotographs of the titanium coating on titanium substrate after different time of ion-plasma treatment at bias pulse parameters f=10^5 Hz, φ_b=–2 kV and τ=8 μs: a) t=3 min; b) t=9 min; c) t=12 min.

The influence of the processing time on the decreasing of the surface MP number density was investigated at a negative bias potential amplitude of φ_b=–2 kV and a pulse length of 8 μs when metal vacuum arc and gaseous plasmas were used simultaneously.

The microphotographs in Fig. 5 show the change in MP density on substrate surface at the processing times 3 min, 9 min and 12 min at the high-frequency short-pulsed bias potential with the pulse amplitude φ_b=–2 kV, pulse width τ=8 μs and frequency f=10^5 Hz.

The microphotographs in Fig. 2 and 5a show significant decrease in MPs number after the treatment time of 3 min at a pulsed bias potential in comparison with the MPs number were deposited at φ_b=0 V. The increase in treatment time to 12 min leads to a significant change in the sample surface morphology (Fig. 5c). The main reason of these surface changes can be associated with the increase of sample temperature depending on the treatment time and ion sputtering.

Fig. 6 demonstrates the various trends in MP behavior for MP diameters smaller and larger than 1.5 μm. For MPs of more than 1.5 μm in diameter, some stabilization (Fig. 6, curve 1) occurs such that for sufficiently long processing times, the large size MP amount is independent of the sample treatment time. Ultimately, a tenfold decrease in the MP surface density is reached for large MPs of more than 1.5 μm in diameter. For MPs of less than 1.5 μm in diameter, there is a rapid reduction in the relative surface MP number density with increasing processing time (Fig. 6, curve 2).

For a processing time of 18 min, the surface number density for MPs of less than 1.5 μm in diameter decreased 1500-fold. In fact, only individual MPs were observed on the surface after long time processing. Curve 3 in Fig. 6 indicates that for processing times of more than ~10 min a 67-fold reduction of droplets total number density has been achieved.

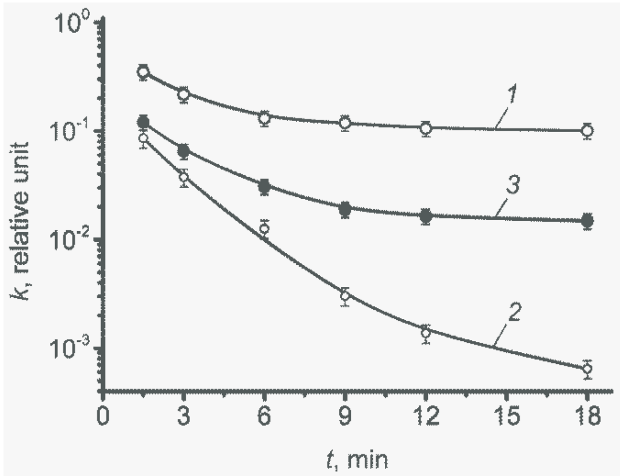

Fig. 6 Reduction in the MP number acquisition on the target versus treatment time (t): 1) droplets diameter D>1.5 μm; 2) droplets diameter D<1.5 μm; 3) all MPs.

CONCLUSION

The possibility of control of MP number density on substrate immersed in vacuum arc and gas discharge plasma has been experimentally demonstrated.

It was found that the increase of the bias pulse repetition rate from 10 Hz to 10^5 Hz allow to change the titanium MP number surface density up to 5 times.

Changing of a bias pulse duration effects on MP number surface density.

The increasing of processing time of ion plasma substrate treatment to 8 min to 18 min allows to decrease the total titanium MP number surface density by 67 times.

ACKNOWLEDGMENTS

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