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# Application of high frequency short-pulsed negative biasing for the decreasing of macroparticle content on substrate immersed in vacuum arc plasma

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#### ABSTRACT

The result of experimental study of macroparticles (MPs) accumulation on negatively biased substrate immersed in DC vacuum arc titanium plasma are presented. It was found that the negative high frequency short pulse biasing of substrate with respect to the adjacent plasma significantly reduces the MPs content on substrate surface. It was shown that the decreasing of MPs surface number density on a negatively biased substrate is determined by the pulse duration, pulse amplitude and processing time. The surface density of MPs with the size <1.5  $\mu$ m was decreased 1500-fold at the bias pulse repetition rate 105 pulse per second (p.p.s.), bias potential -2 kV, pulse duration 8  $\mu$ s and processing time 9–18 min. The total MPs surface density in these cases was decreased 67-fold.

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#### 1. Introduction

The vacuum arc discharge is a simple means of creating a highly ionized metal plasma for ion implantation and coating deposition [1–6]. The main disadvantage of vacuum-arc discharge, which significantly limits its applications, is the presence of the significant amount of microdroplets often referred to as macroparticles (MPs) [7–9]. MPs have size from 0.1  $\mu$ m to 100  $\mu$ m at velocities from 1 m/s to 800 m/s [8,10,11]. In order to produce defect-free coatings for different applications vacuum arc plasma need to be filtered of MPs. Most of research effort on MPs filtering has been focused on development of magnetic and electromagnetic plasma filters [12,13].

Recently, researchers have intensified the search for alternative methods of MPs suppression [14–17]. Some of the very interesting effects of MPs number and surface density reduction exerted on the substrate during the coating formation have been observed experimentally in the following works [18,19]. The authors of these papers observed the MP number density reduction effect on the coating surface 3–4-fold with increasing negative DC bias potential on the substrate immersed in the vacuum arc plasma up to  $\varphi_{\rm b} = -1000$  V. In the study of these results in Refs. [20,21] the explanation of observed effect has been proposed. The authors suggested

http://dx.doi.org/10.1016/j.apsusc.2014.03.113 0169-4332/© 2014 Elsevier B.V. All rights reserved. that it was based on the possibility of the electrostatic repulsion of MPs negatively charged in the vacuum arc plasma by the electric field of sheath near the biased substrate. Ref. [22] describes investigation of the possible enhancement of MPs reflection effect by increasing the temperature of plasma electrons and therefore increasing up to the potential in which MPs were charged in the plasma. A possibility of short-pulse high-frequency negative bias potential applications to decrease the MPs number density on substrate immersed in DC Ti vacuum arc plasma has been presented in papers [23,24].

This paper is devoted to the study of MPs content decreasing on the substrate immersed in a titanium vacuum arc and gas discharge plasma versus the different parameters of high-frequency shortpulse negative biasing.

#### 2. The experimental setup and methodology of research

The investigation of the influence of high-frequency short-pulse negative substrate biasing parameters on the accumulation of vacuum-arc MPs on the substrate surface was carried out using the experimental setup presented in Fig. 1 and described in more details in Refs. [25,26]. A DC vacuum-arc plasma generator with a titanium cathode was located on the side flange of the vacuum chamber and operated at 100 A arc current.

The arc generator with the hot cathode "PINK" [27] provided the argon plasma formation. The application of gaseous plasma source along with the metal plasma generator gave the

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2

## **ARTICLE IN PRESS**

A.I. Ryabchikov et al. / Applied Surface Science xxx (2014) xxx-xxx

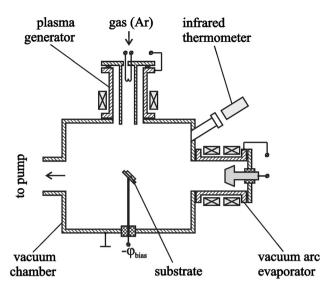


Fig. 1. The scheme of experimental installation.

possibility of plasma density variation near the substrate surface by means of changing its location in the chamber. The Ti ion saturation current density from plasma on substrate was 4.4 mA/cm<sup>2</sup>. The ion saturation current density from argon plasma on substrate was 1.4 mA/cm<sup>2</sup>.

A high-frequency short-pulse negative bias generator was used to carry out the investigations. The parameters of the generator are: pulse duration  $1-9\,\mu s$ , pulse repetition rates  $10^5$  p.p.s., negative pulse amplitude 0.5-3.5 kV.

In the experiments stainless steel and titanium substrates were used. The surfaces of substrates were polished to  $R_a = 0.035 \,\mu$ m. The substrates were mounted on a massive holder to decrease the rate of substrate temperature increase. During experiments the temperature of substrate surface was measured by the infra-red thermometer.

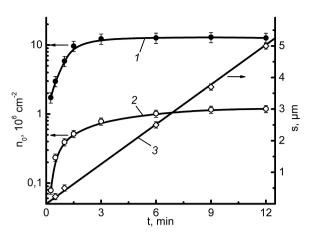
Initially, the surface of substrate was treated by ions using argon plasma and the high-frequency short-pulse bias [25,26]. Thus the samples were preliminarily heated up to temperature  $\sim$ 200 °C.

The MPs densities on the substrate surface were studied using optical and electron (Hitachi TM-1000 and Hitachi TM-S3400 N) microscopes. The systematics of the change of MP number density on the substrate surface were investigated on two groups (MPs with a size to  $1.5 \,\mu\text{m}$  and more than  $1.5 \,\mu\text{m}$ ). Experimental data on MP number surface density change are presented in the figures in absolute units and in the form of MP relative surface density  $k = N/N_0 = n/n_0$ , where N = nS is the quantity of MPs located on the chosen area of substrate (S) at negative biasing; n – number surface density of MP at negative biasing;  $N_0 = n_0 S$  – the quantity of MP on the chosen area of a substrate at anode potential ( $\varphi_{\rm b}$  = 0 V) at the chosen vacuum-arc plasma deposition time;  $n_0$  – number surface density of MPs at anode potential biasing on the substrate at a chosen time of metal plasma deposition; S – chosen for the statistically reliable calculation of the MP quantity on substrate surface. For each experimental point total area for MP calculation made  $6 \times 10^4 \,\mu m^2$ .

#### 3. Experimental results and discussion

3.1. Accumulation of titanium MPs on the substrate surface at the anode potential

To study the dynamics of MPs density accumulation on the sample surface in the course of vacuum arc plasma deposition the series of experiments with various processing time and at small DC bias



**Fig. 2.** MPs number surface density ( $n_0$ ) and coating thickness (s) versus vacuum arc plasma deposition time (t): 1 – MP diameter <1.5  $\mu$ m, 2 – MP diameter more than 1.5  $\mu$ m, 3 – coating thickness.

potential ( $\varphi_b \approx -100 \text{ V}$ ) were carried out. The results of MP number density measurement on the substrate surface at the different times of metal plasma deposition are presented in Fig. 2.

It is visible that the increase in the MPs number surface density occurs in proportion to process time at the beginning of plasma deposition. After a while the MP number surface density reaches saturation and further practically remains constant. It is well visible from Fig. 2, curve 1 for the MPs of small diameter  $D < 1.5 \,\mu$ m. The maximum of MP number surface density is reached in 3 min after the beginning of plasma deposition. For large MPs (Fig. 2, curve 2) the effect of surface density saturation takes place at much bigger time of metal plasma deposition ( $t \ge 9 \,\text{min}$ ). The measurement of coatings thickness (Fig. 2, curve 3) at different times showed that the average rate of plasma deposition and on the basis of Fig. 2 data it is possible to draw a conclusion that for small MPs their surface density reaches a maximum at the thickness of coating ~1.25  $\mu$ m, and for MPs with  $D > 1.5 \,\mu$ m at the thickness of coating ~3.5  $\mu$ m.

## 3.2. Bias pulse parameters influence on the deposited coating thickness

The plasma deposition on a substrate with negative bias is accompanied by the complex of processes including coating formation, ion sputtering and ion implantation. To understand what of processes prevails otherwise experiments on titanium plasma deposition were made at different pulse length and the amplitudes of negative bias potentials. The results of experiments are presented in Fig. 3.

At bias potential  $\varphi_{\rm b}$  = -850 V (Fig. 3, curve 1) for all pulse durations a coating formation takes place. The increase in pulse duration leads to the reduction of coating thickness because of the ion sputtering increasing. Still the bigger contribution of ion sputtering is observed upon increase in negative bias amplitude (Fig. 3, curve 2). At bias  $\varphi_{\rm b}$  = -2 kV (Fig. 3, curve 3) the coating is formed only at bias pulse duration in the range of 2-6 µs. Further increase in pulse duration leads to the coating disappearing because of the ion sputtering.

#### 3.3. Bias pulse parameters influence on MPs content on substrate

Experimental data on the Ti MP number surface density decreasing versus bias pulse duration at  $\varphi_b = -2 \text{ kV}$  after different processing time (the plasma of vacuum arc and gas-discharge plasma) are presented in Fig. 4.

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