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Titanium macroparticles density decreasing on the sample, immersed in plasma, at repetitively pulsed biasing

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1. Introduction

A DC vacuum-arc discharge is an attractive source of highly ionised metal plasma. However, vacuum-arc plasma contains a significant amount of MPs emerging at the micro-edges of the explosion on the working spot of a cathode [1–3]. MPs travel at velocities ranging from 1 to 800 m/s and deposit on the substrate in the form of micro-droplets ranging from 0.1 to 100 μ m in size [2,4–6]. The presence of MPs in the metal plasma can degrade the quality of coatings [1–3]. A number of different MP filtering systems have been proposed and developed in order to obtain MP-free dense metal plasma [7–9]. Nevertheless, the efficiency of vacuum arc plasma transportation through such filters is low [10,11].

Many different approaches have been proposed and used to decrease MP number density in the coatings [12,13]. It was demonstrated also that an application of negative DC biasing to a substrate immersed in vacuum arc plasma significantly reduces the MP content in coatings [14–16]. The authors of these papers observed that the MP surface density on the TiN coating was reduced three- to four-fold when the negative bias potential on the substrate, which was immersed in the vacuum-arc plasma, was increased up to $\varphi_b = -1000 \text{ V}$. The paper [15] investigated the possible enhancement of the MP reflection effect by increasing the temperature of the plasma electrons and therefore increasing the potential

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ABSTRACT

We have studied the features of the Ti macroparticles (MPs) accumulation on a substrate immersed in a DC vacuum arc plasma with a high frequency short pulse negative bias. The influence of several factors including processing time, the parameters of bias potential, and substrate characteristics was defined. It was found that the MP accumulation on a repetitively biased substrate was uneven over time. A deposition of MPs to substrate were significantly decreased after 1 min of processing at bias parameters $f = 10^5$ p.p.s., $\varphi_b = -2$ kV and $\tau = 7 \mu s$. It was experimentally shown that DC vacuum arc plasma without pre-filtering of MPs can be used for high frequency short pulse plasma immersion metal ion implantation.

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to a point at which the MPs were charged in the plasma. They used conventional vacuum-arc plasma, as well as the gas-discharge plasma of a low-pressure external arc with a hot cathode. This work observed a five-fold decrease in the MP surface density when the DC bias potential was increased to -1000 V.

A high-frequency short-pulsed negative bias potential was also used in previous works [16,17]. Using short pulse duration of the bias potential $(1-9\,\mu s)$ allowed an increase in the negative bias potential amplitude up to 2–3 kV. The number density decreasing by several orders has been demonstrated [18]. This paper addresses the study of different bias pulse and substrate parameters to decreasing of Ti MP number decreasing.

2. Experimental setup

The influence of high-frequency, short-pulsed negative substrate biasing on the behaviour of vacuum-arc plasma Ti MPs near and on a potential sample surface was investigated using the complex setup that has previously been described in details [19,20]. The vacuum-arc plasma generator with a titanium cathode, which operated in DC mode with an arc current of 100 A, was set on the side flange of the vacuum chamber.

A high-frequency short-pulsed negative bias generator was used to carry out the investigation. The parameters of the generator were set as follows: pulse duration $1-9\,\mu$ s, pulse repetition rate 10^5 pulse per second (p.p.s.), negative bias pulse amplitude 0.25–2 kV. Typical oscillograms of sheath current and bias pulses of the generator are shown in Fig. 1.

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2 D.O. Sivin et al. / Applied Surface Science xxx (2014) xxx-xxx (a) 200 /, mA 100 0 M 5 10 15 20 t, µs (b) t, µs 0,5 5 10 15 20 11111 0,0 -0,5 -1,0 U, k∨ -1.5 -2.0 -2,5

Fig. 1. Oscillograms of sheath current (a) and bias (b) pulses of the high frequency short pulse generator.

Stainless steel and titanium substrates were used in the experiments. The surfaces of substrates were polished to $R_a = 0.035 \,\mu$ m. The substrates were mounted on a massive holder to decrease their heating in the process of substrate treatment by ions. The substrates were vertically mounted near the axis of the metal plasma flux at a distance of 38 cm from the cathode of the vacuum-arc evaporator. A measured ion current density on substrate was 4.4 mA/cm².

Initially, the surface of the substrate was treated by ions using an argon plasma and high-frequency short-pulse bias [16,17].

The surface density of MPs on the samples as well as the changes in the MP form and size were studied using high-resolution optical and scanning electron microscopes Hitachi S-3400 N. The energy dispersion attachment (EDS) Bruker XFlash 4010 incorporated in scanning electron microscope was used for the X-ray spectrum analysis of substrate elemental composition. The experimental data for the MP surface density change are presented in the figures in



Fig. 2. Dependence of the MP surface density (*n*) on the substrate versus deposition time (*t*) at anode potential $\varphi_b = 0$ V (curve 1) and repetitively pulsed potential ($f = 10^5$ p.p.s., $\varphi_b = -2$ kV) for different pulse lengths: $2 - \tau = 3 \mu s$, $3 - \tau = 5 \mu s$, $4 - \tau = 7 \mu s$.

absolute units. The MP numbers were calculated over a surface area of $270 \times 210 \ \mu m^2$ for each experimental point.

3. Experimental results and discussion

The MP quantity and density change on the substrate immersed in vacuum-arc titanium plasma are presented in Fig. 2 as a function of the negative bias pulse width and processing time.

The dynamics of MP accumulation over time on the substrate with an anode potential are given in Fig. 2, curve 1. Increasing the time of the process from 0.25 to 3 min increases the MP number density on the substrate surface by more than 5-fold. Over time, the curve approaches saturation. This behaviour of the MP surface number density is connected to the thickness of the deposited coating, which increases as the plasma deposition time increases. Thus, the previously deposited MPs are immured in the coating. First, small particles are concealed; as the coating thickness increases, the larger particles are also concealed. Dynamic equilibrium will be reached for long process times. Specifically, the number of arriving MPs per unit time on the substrate surface equalled the number of MPs simultaneously immured in the coating. The MP density on the substrate surface remained constant thereafter.

Fig. 3 demonstrates the Ti MP number growth on substrate with vacuum arc plasma deposition time increasing from 1.5 to 6 min.

The appearance of large MPs with radius more than $1.5 \,\mu$ m on the microphotographs obtained with a scanning electron microscope agrees with the assumption that the MPs were molten metal drops when they impacted the surface. The vast majority of such MPs on the surface are partly flattened. The analysis of microphotographs (Fig. 3) shows that the number of MPs with



Fig. 3. Microphotograph of substrate surface after different titanium vacuum-arc plasma and MPs deposition time at $\varphi_b = 0$ V: (a) 1.5 min; (b) 3 min; (c) 6 min.

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