Indian Journal of Science and Technology, *Vol 8(36), DOI: 10.17485/ijst/2015/v8i36/90573, December 2015* *ISSN (Print) : 0974-6846* *ISSN (Online) : 0974-5645*

Control of Vacuum Arc Macroparticles by High-Frequency Short-Pulsed Negative Bias Application

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Abstract

Background/Objectives: Paper is devoted to a brief review of the investigation of short negative bias pulse application for titanium and aluminum macroparticles control on the substrate immersed in vacuum arc plasma. **Methods/Statistical Analysis:** The vacuum-arc plasma generator with a titanium or aluminum cathode which operated in a DC mode with the arc current 100 A was set on the side flange of the vacuum chamber. 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 μs, pulse repetition rates 10^5 pulse per second (p.p.s.), negative pulse amplitude 0.5–3.5 kV. The MP densities on the substrate surface were studied using optical and electron (Hitachi TM–1000 and Hitachi TM–S 3400 N) microscopes. **Findings:** It was found that the decreasing of MP surface number density on a negatively biased substrate is determined by the pulse amplitude, pulse duration, pulse frequency, plasma density and processing time. A possibility to reduce the macroparticle number density more than 1000 fold has been demonstrated. **Applications/Improvements:** The application of high voltage high-frequency short-pulse negative bias to a substrate immersed in vacuum arc plasma provides the possibility of the multiple decrease of MP number surface density and opens a possibility for realization plasma immersion metal ion implantation technology using DC vacuum arc plasma.

Keywords: Macroparticles, Metal Plasma, Negative High-Frequency Short-Pulsed Bias, Vacuum-Arc

1. Introduction

The vacuum-arc plasma is characterized by the high degree of the ionization of cathode material erosion products[1–3]. This benefit provides the wide practical application of vacuum-arc plasma for different multifunctional coating deposition and particle beam surface modification technologies[4–8]. The main disadvantage of vacuum-arc discharge, which considerably limits its applications, is the presence of significant amount of microdroplets often referred to as MPs. MPs have size 0.1 μm to 100 μm at velocities from 1 m/s to 800 m/s[9,10]. 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[11,12].

The effect of the MPs numberdensity reducing on the substrate immersed in vacuum arc plasma during the coatings formation has been observed in the following works[13–15]. The authors of these papers observed the MPs density reducing effect on the TiN coating surface by 3–4 times with increasing DC negative bias potential on the substrate up to $\varphi = -1000$ V. Subsequent publications have shown that the application of repetitively pulsed negative bias for MP number decreasing is more efficient[16–20].

This paper is devoted to a brief review of the investigation of short negative bias pulse application for titanium and aluminium MP number density control on the substrate immersed in vacuum arc plasmas.

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2. The Experimental Setup and Methodology of Research

The investigation of the influence of high-frequency short-pulse negative substrate biasing on the behavior of vacuum-arc MPs near and on substrate was carried out using the experimental setup described in[21]. The vacuum-arc plasma generator with a titanium or aluminum cathode which operated in a DC mode with the arc current 100 A was set on the side flange of the vacuum chamber.

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 μs, pulse repetition rates 10^5 pulse per second (p.p.s), negative pulse amplitude 0.5–3.5 kV.

The MP densities on the substrate surface were studied using optical and electron (Hitachi TM–1000 and Hitachi TM–S 3400 N) microscopes.

3. Experimental Results with Titanium Vacuum arc Plasma

3.1 Accumulation of Titanium MPs on the Substrate Surface at the Anode Potential

The typical photograph of substrate surfaces after the deposition of titanium plasma without bias potential (φ_b = 0 V) are presented in Figure 1. Figure 1 shows that the majority of MPs on the substrate surface has near-spherical shape. The flux of molten MPs is formed due to the explosion of micro edges in the cathode spot of vacuum arc discharge. The effect of MPs cooling during their transport in vacuum arc plasma is well known and conditioned by the substantial exceeding of energy loss caused by a heat radiation from the MPs surface, the evaporation of MPs materials, thermionic emission over the extra energy obtained from the ions and electrons at the low plasma density.

The MPs rapidly cool as the distance from the cathode increases. The estimations[22] showed that the MP with radius of 1 μm keep its liquid state for about 3 ms. It will be the distance from cathode of about 3 cm to solidification at the speed of its movement, for example, 10 m/s.

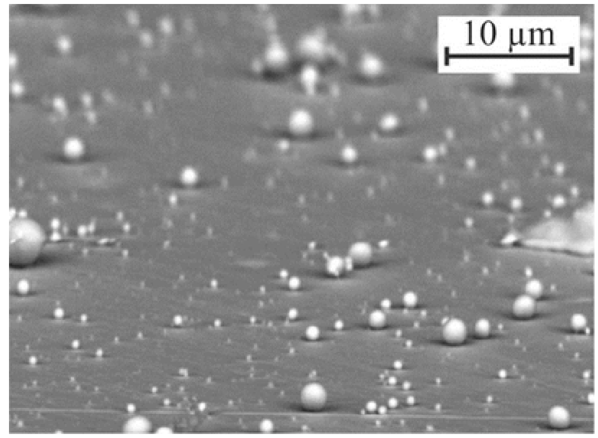

Figure 1. Microphotograph of substrate surface after titanium vacuum-arc plasma and MPs deposition at bias φ_b = 0 V.

To study the dynamics of MPs density accumulation on the sample surface in the course of vacuum arcplasma deposition the series of experiments with various processing time and at small DC bias potential ($\varphi_b \approx -100$ 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 Figure 2.

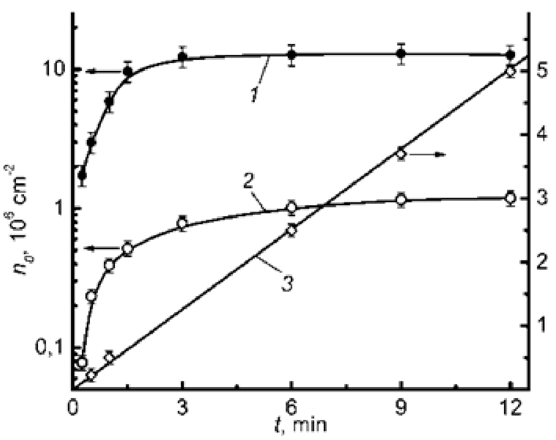

Figure 2. MPs number surface density (n_0) and coating thickness (s) versus vacuum arc plasma deposition time (t): 1 – MP diameter less than 1.5 μm, 2 – MP diameter more than 1.5 μm, 3 – coating thickness.

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 Figure 2, curve 1 for the MPs of small diameter D < 1.5 μm. The maximum of MP number surface density is reached in 3 min after the beginning of plasma deposition. For large MPs (Figure 2, curve 2) the effect of surface density saturation takes place at much bigger time of metal plasma deposition

(t ≥9 min). The measurement of coatings thickness (Figure 2, curve 3) at different times showed that the average rate of plasma deposition was 25 μm/h. Taking into account the rate of plasma deposition and on the basis of Figure 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 μm, and for MPs with D > 1.5 μm at the thickness of coating ~3.5 μm.

3.2 Titanium Vacuum Arc MP Number Density Distribution Versus MP Diameter

For better understanding of what impact on the decrease of MPs surface density can be produced by the MPs with different diameters two substrates were treated for 1 min with $\varphi_b = 0$ V and $\varphi_b = -2$ kV and analyzed in detail using an electron microscope. The corresponding histograms of Ti MP number density distributions versus their sizes are shown in Figure 3.

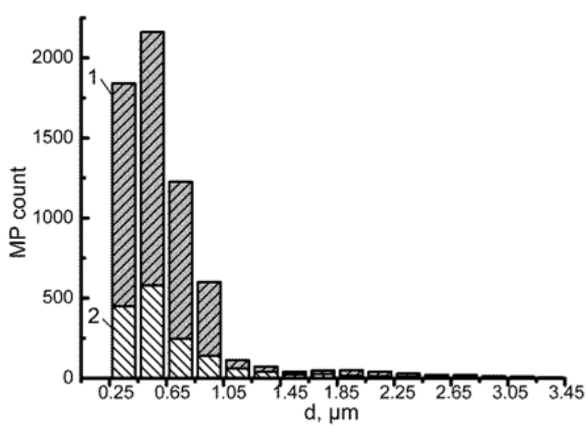

Figure 3. Histogram of MP number density distribution versus MP diameter after 1 min of Ti vacuum arc plasma deposition: 1– substrate bias $\varphi_b = 0$ V; 2– substrate at repetitively-pulsed negative bias with parameters: $\varphi_b = -2000$ V, $\tau = 7$ μs, $f = 10^5$ p.p.s.

The data in Figure 3 shows that the impact of MPs with the diameter smaller than 0.65 μm to the decreasing of MP number density on the biased substrate immersed in vacuum arc plasma was more considerable. Estimation shows that the surface density of small MPs was decreased almost 4 fold. The number of MPs with sizes ranging from 0.65 μm to 1 μm was decreased by 2.5 fold only. The number of MPs with the diameter larger than 1.5 μm at the treatment time of 1 min does not change. The average relative surface density of MPs estimated from the data of histogram is 0.36.

3.3 Bias Pulse Frequency and Duration Influence on MPs Content on Substrate Immersed in Vacuum Arc Plasma

These studies 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 10to 10^5 p.p.s.

Stainless steel or titanium samples with size 2 • 2 • 0.3 cm^3 were installed at a distance of 23 cm from the cathode along axis of vacuumarc plasma generator. In all experiments a processing time was 30s. Experimental dependence of the MP number density on the substrate surface versus pulse frequency is presented in Figure 4.

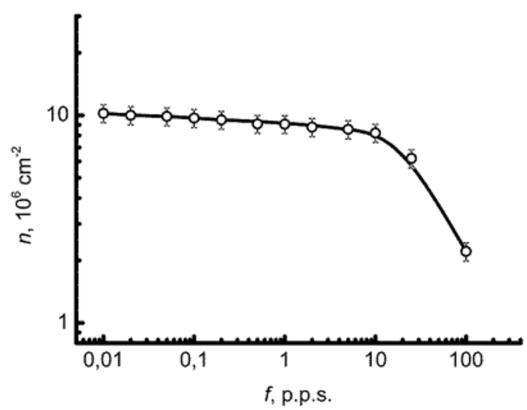

Figure 4. Dependence of the MP number density versus pulse repetition rate at $\varphi_b = -2$ kV, processing time - 30 s, cathode – substrate distance 23 cm.

An increase of the bias pulse frequency from 10 to 10^4 p.p.s. leads to a gradual reduction in MP number density on the substrate surface. However, this decrease does not exceed 20% of the MP number density deposited in the absence of bias. Character of the curve varies considerably in the frequency range 10^4 to 10^5 p.p.s. of the bias pulses. In this frequency range of the bias pulses MP surface number density decreases 4 fold. Compared with the MP number density deposited at $\varphi_b = 0$ V in the case of negative bias potential amplitude of 2 kV, pulse width 7 μs at a pulse repetition frequency 10^5 p.p.s. MP density decreases by more than 5 times.

Experimental data on the Ti MP number surface density decreasing versus bias pulse duration at $\varphi_b = -2$ kV, pulse frequency 10^5 p.p.s. after 1.5 min of processing time (the density of plasma ($j_i = 5.8$ ma/cm^2) are presented in Figure 5.

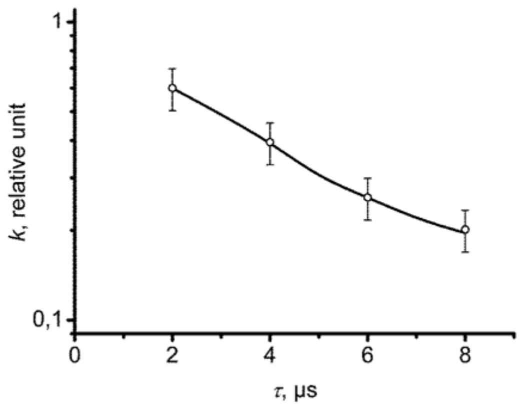

Figure 5. Relative MP number surface density (k) versus negative bias pulse duration (τ) at $\varphi_b = -2$ kV, the processing time – 1.5 min and cathode – substrate distance 38 cm.

Figure 5 shows that the efficiency of MP number surface density decreasing significantly depends on the bias pulse duration. The MP relative number surface density has practically inverse linear relationship in logarithmic scale versus bias pulse length.

3.4 Negative Bias Pulse Amplitude Influence on MPs Content on Substrate

On the accumulation of macroparticles on the substrate immersed in a vacuum arc plasma significantly affects the amplitude of the bias pulse. Figure 6 demonstrates the change in the surface number density of MPs deposited on the substrate surface within 3 min with a bias potential pulse width of 3 μs versus of the negative bias pulse amplitude.

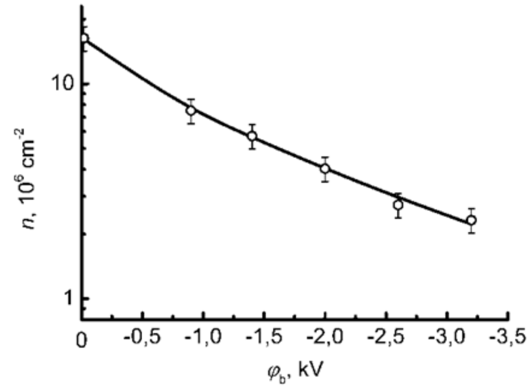

Figure 6. Dependence of the MP density (n) versus negative bias pulse amplitude (φ_b) at f = 10^5p.p.s., τ = 3 μs and processing time t = 3 min.

An increase in the negative bias potential amplitude from 0 to 3.2 kV was followed by an almost 7.7-fold decrease in the MP number density. Study of the substrate surface after their ion - plasma treatment revealed a number of features. At small bias pulse amplitudes (up to 1.5 kV) the majority of MPs on the substrate surface has near-spherical shape like in the case of plasma deposition on a sample with $\varphi_b = 0$ (see Figure1). Further increase in the amplitude of the bias pulses not only leads to decrease in the MP number density on the substrate surface, but also in their forms transformation.

3.5 Plasma Density Influence on MPs Content on Substrate

Therefore, to MP number density decreasing and their form transformation should affect not only the parameters of the bias potential and characteristics of the substrate surface, but the density of vacuum arc plasma. To prove this several experiments with different ion saturation current densities have been carried out. To control the vacuum arc plasma density at substrate surface we have changed a distance from cathode surface of vacuum arc evaporator to substrate immersed in plasma in the range of (23–80) cm. Increasing the distance from 23 cm to 80 cm the ion saturation current in plasma was decreased from 20 mA/cm^2 to 2 mA/cm^2. In these experiment we have used the same bias pulse parameters (φ_b= -2 kV and τ = 7 μs, f = 10^5 p.p.s). The results of MP number density measurement versus of the ion saturation current density in plasma are presented in Figure 7.

Curve 1 Figure 7 shows the MP number density on substrate surface after 30 s of vacuum arc plasma deposition at anode substrate potential. In case of negative bias pulse application during the same processing time (30 s) a significant decreasing of MP number density versus ion saturation current from plasma or plasma density has been found. Data of Figure 7, curve 2 shows that when ion saturation current density at substrate surface was increased to 20 mA/cm2 the MP number density was decreased almost 6 fold after 30s of processing time. On the other hand when ion current density at substrate surface was decreased to 2 mA/cm^2 an effect of MP number density decreasing as well as MPs form transformation practically disappeared. At small plasma density at substrate surface the MP number density decreasing do not exceed several percent, only.

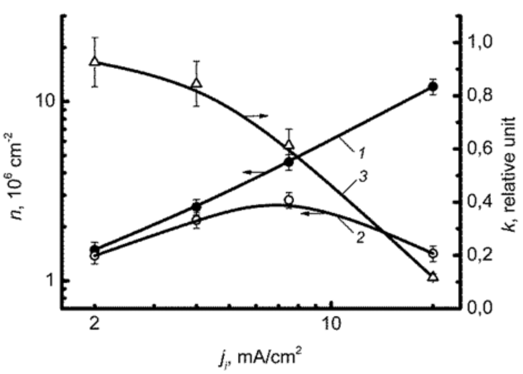

Figure 7. Dependences of MP number surface density on substrate at $\varphi_b = 0$, t = 30 s (curve 1) and at $\varphi_b = -2$ kV, $\tau = 7$ µs, t = 30 s (curve 2) and MP relative number surface density versus ion saturation current from plasma at substrate surface.

3.6 Process Time Influence on MPs Content on Substrate

Significant results on the influence of processing time upon MP number surface density reduction were observed at the amplitude of negative bias potential $\varphi_b = -2$ kV at pulse duration 8 µs. Figure 8 shows the various systematics of MPs behavior with the diameter less and more than 1.5 µm. For MPs of diameter more than 1.5 µm, dependence with some stabilization (Figure 8, a curve 1) takes place when the relative MP number surface density is independent of process time. Finally the tenfold decrease of MPs surface density for large macroparticles with diameter more than 1.5 µm is reached. There is the fast reduction of relative MP number surface density with the process time increasing, for MPs of diameter less than 1.5 µm (Figure 8, a curve 2).

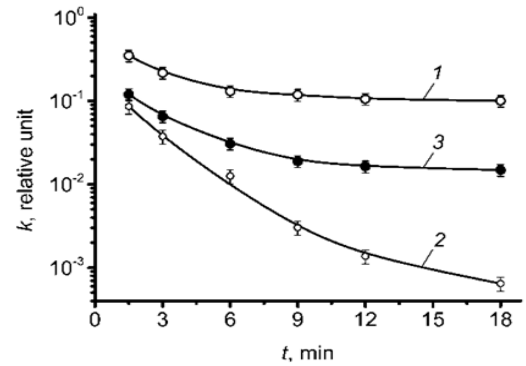

Figure 8. Relative MP number surface density (k) versus the process time (t): 1 – MP diameter more than 1.5 µm, 2 –MP diameter less than 1.5 µm, 3 – for all MPs.

At 18 min of the process time for MPs of diameter less than 1.5 µm their number surface density decreased 1500-fold. Actually only separate MPs were observed on a surface. It is characteristic that at small processing times the relative MPs surface density is determined by small MPs as their initial surface density repeatedly exceeds the surface density of large MPs. After increasing the processing time to 6–9 min the behavior of large MPs starts to play a defining role. In this case their surface density starts to exceed the surface density of small MPs. From data in Figure 5, the curve 3 shows that at processing time more than ~10 min the decreasing of the full number surface density of MPs by 67-fold are reached.

4. Investigation of Physical Mechanisms of Macroparticles Number Density Decreasing on a Substrate Immersed in Vacuum Arc Plasma at Negative High-Frequency Short-Pulsed Biasing

4.1 Ion Sputtering and Repulsion of MPs

To determine the influence of the different physical mechanisms on the total dynamics of Ti MPs number surface density decreasing four experiments with additional tungsten grid placed at 0.8 cm from the substrate were carried out.

In the first case the grid had the anode potential. The substrate was upon negative high-frequency short-pulse biasing (Figure 9(a)).

In the second experiment, on the contrary, the grid had the negative high-frequency short-pulse bias, but the substrate was under anode potential (Figure 9(b)). It is necessary to mention that the use of grid with cell sizes much smaller than the width of the sheath provides the opportunity to create an electric field near this electrode which is almost the same as the one of a solid electrode. Non uniform electric field in case of grid will take place on distances in some cell sizes only. Thus, the negatively charged MP, entering from plasma into a sheath, should be repulsed in the same way as in the case with the solid electrode. Ion sputtering of substrate and MPs should not take place in the second case, because after the acceleration of ions in the sheath before the grid, they will be decelerated in the electric field between the grid and the substrate.

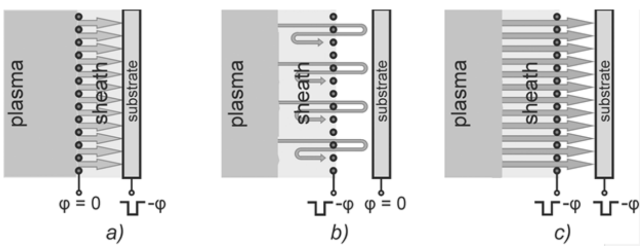

Figure 9. Set of experiments with the additional grid electrode.

In the third case both the grid and the substrate had the same negative high-frequency short-pulse bias (Figure 9(c)). When the grid and the sample are under negative bias, the strong electric field exists before the grid, only. Between the biased grid and the substrate the electric field is insignificant and defined by a space charge of ions in this gap. This means that in this case, there is the strong electric field near the grid for the electrostatic repulsion of the charged MPs. At the same time, in this case an ion sputtering of MPs should take place.

In the fourth experiment both the grid and the substrate had anode potential, and the usual deposition of metal plasma and MPs took place.

The results of all experiments are presented in Figure 10. The dynamics of MP number surface density increasing versus vacuum arc deposition time on substrate when the grid and the substrate had anode potential is shown in Figure 10, curve 1.

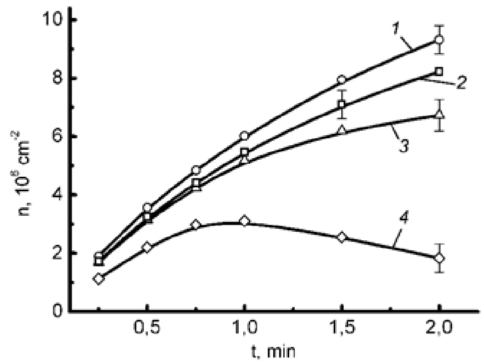

Figure 10. MP surface number density at different grid and substrate potentials versus processing time: 1 – $\varphi_{grid} = \varphi_{sub} = 0$ V; 2 – $\varphi_{grid} = -2$ kV, $\varphi_{sub} = 0$ V; 3 – $\varphi_{grid} = \varphi_{sub} = -2$ kV; 4 – $\varphi_{grid} = 0$ V, $\varphi_{sub} = -2$ kV.

Experiments have shown that in the case when negative high-frequency short-pulse bias has been applied only to a substrate, the surface density of MPs decreased depending on processing time as it is shown in Figure 10, curve 4. MP surface density is decreased 5 fold after 2 min of processing time.

The results of the experiments with a biased grid (Figure 10(b)) were unexpected. The decrease of MP number surface density using biasing –2 kV does not exceed 10% (Figure 10, curve 2). It means that not more than 10% of the MPs negatively charged in plasma were repulsed in electric field near negatively biased substrate.

The third series of experiments (see Figure 10(c)) showed that the number surface density of MPs decreased by 30% after 2 min of processing time at bias on a grid and a target –2 kV (Figure10, curve 3). Actually, it means that ion sputtering reduces the MPs surface density by 20%, only. But if one pays attention to Figure 10, curve 4, it is evident that at processing time of 2 min the 5 fold decrease of MPs surface density was reached. This means that in addition to the MPs repulsion after its interaction with the electric field in the sheath and ion sputtering, there are few other mechanisms reducing of MP number surface density.

4.2 Enhanced Ion Sputtering and Evaporation of MPs

With a significant increase in processing time and temperature of the substrate a three new thermal mechanisms of MP number density decreasing were identified when aluminum vacuum arc plasma was used in the experiments. The measurements using an infrared thermometer showed that the temperature of the substrate was increased to 700 °C after 6 min of processing time in the case of aluminium vacuum-arc plasma. Thus, the substrate temperature after 6 min of treatment was higher than the melting point of aluminum. Figure 11 demonstrates microphotographs of substrate surfaces exposed to ion-plasma processing in aluminum vacuum-arc plasma at anode potential and different plasma deposition time ((a) –1 min, (b) –6 min) and at repetitively-pulsed biasφ_b = –2000 V, τ = 7 μs, f = 10^5p.p.s. ((c) –1 min, (d) –6 min)).

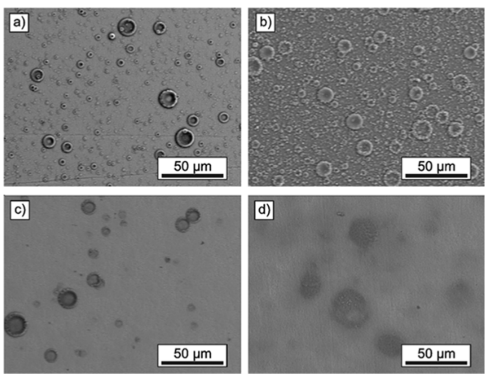

Figure 11. Microphotographs of substrate surfaces exposed to ion-plasma processing in aluminum vacuum-arc plasma at anode potential. (a) –1 min. (b) –6 min. and at repetitively-pulsed bias φ_b = –2000 V, τ = 7 μs, f = 10^5p.p.s. (c) –1 min. (d) –6 min.

The appearance of aluminium MPs 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 the MPs on the surface were flattened to ruses that contained a thin layer of aluminium in the middle. The shapes of MPs after depositing aluminium via pulsed vacuum-arc plasma were similar as reported previously[9].

The analysis of micrographs (Figure 11(a), (b)) shows that the number of MPs with small diameters (less than 2 μm) exceeded the number of large MPs (more than 2 μm) multi-fold. The increase in the aluminium plasma deposition time was followed by an increase in the MP number density on the surface. The MP number density did not increase proportionally to the time of plasma deposition. It tended to saturate due to the immurement of MPs previously deposited as the coating grew. A similar situation was observed in the case of titanium and aluminium plasma deposition process. This tendency can be observed in the micrographs of Figure 11(a), (b) and is demonstrated by the data in Figure 12, curve 1, which were obtained by counting the MP quantity on the substrate surface for plasma deposition times from 0.5 min to 3 min.

The situation is different in the case of a repetitively pulsed bias potential application. The aluminium MP number surface density change on the substrate at φ_b=–2 kV is shown in Figure 12, curve 2 as a function of the process time. These data show that the aluminium MP number density decreased on the repetitively biased substrate by three orders of magnitude after processing for three minutes.

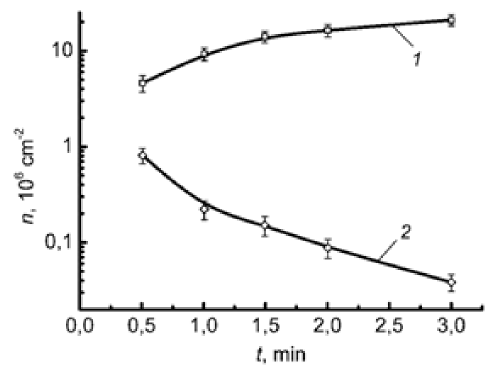

Figure 12. Dependence of aluminium MP number surface density (n) on the substrate on deposition time (t). (a) At anode potential on the substrate φ_b = 0 V. (b) At repetitively-pulsed bias potential f =10^5 p.p.s., φ_b = –2 kV, τ =7 μs.

When a processing time of ion-plasma substrate treatment is small (1 – 2 min) and substrate temperature is still a small the MPs have the shape of the torus (see Figure 12(c)).The substrate temperature greatly increases with growth time of ion-plasma treatment at these conditions. Gradually, the sample temperature approaches the melting point of aluminum. In such case, the energy losses of the impacted MPs on heat transfer to the substrate will be significantly reduced.

We hypothesize that due to a combination of MPs velocity and high temperature of MPs and substrate each MP can be flattened to a very thin film. A thickness of flattened MP on substrate surface will be decreased by several times. At the same time the surface area of MPs on the substrate will be increased by several times. This results in enhancement of MP ion sputtering on the substrate surface. A time of MP ion sputtering will be decreased by several times, as well.Microphotograph in Figure 12(d) proves this hypothesis. Footsteps of the Al MPs on substrate at high processing time (6 min) are much greater than at the beginning of substrate treatment (1 min, Figure 12(c)).

An evaporation of aluminum MPs are increased when the substrate temperature exceeds the melting point. However, the target temperature is not so great as to provide a very rapid evaporation of the MPs. Rapid removal of MPs from the substrate surface does not provide an ion sputtering, as well. At the same time we can`t find the MPs which impacted with substrate at a very end of the ion plasma substrate processing (see Figure 12(d)). This means that there is another physical mechanism for MPs removing. In this case the absence of footstep of the small diameter low melting Al MPs on substrate gives a possibility to propose that a significant heating and evaporation of the MPs can take place into sheath, before MP interaction with substrate surface. Heating of the MP during their movement occurs due to the energies of ions and secondary electrons from substrate in the sheath. In this case high substrate temperature partially compensates for the loss of MP energy by the thermal radiation.

4.3 Influence of Substrate Morphology to Repulsion of MPs

The repetitively pulsed mode of negative bias potential formation and substrate morphology can influence the dynamic decrease of the MP number density, also. For example, if the width of the sheath is 1 cm, a MP moving at a velocity of 10 m/sec will pass the sheath in 10^{-3} sec. During this time, a pulse bias potential will be applied to the substrate immersed in plasma ~ 100 times. Consider a MP that closely approaches the substrate surface, at which point the next pulse of a negative bias pulse is applied. The MP is inside the sheath near the surface after the charge exchange in the plasma and has a slightly negative potential (approximately the potential of the anode). This result implies that a notably strong electric field occurs between the substrate surface with a potential equal to -2 kV and a micron-sized particle[23]. MPs and other micro-irregularities on the substrate surface accordingly increase the electric field strength near the surface and between the MP and surface. This electric field strength is several orders of magnitude higher than the strength in the matrix sheath. Thus, increasing the electric field strength should cause either a micro-discharge or a corresponding increase in the field emission of electrons from the surface of the negative substrate and provide the charge of MPs with electrons to the potential, which is approximately the same as the surface potential. In this case the charge of MPs is increased by more than two orders of magnitude in compare with the charge of MPs in the plasma. The electrostatic force of MPs repulsion from the substrate surface increases accordingly. Integrating the equation for MP movement with its negative charge close to the charge defined by substrate potential ($\varphi_{sub}= -2$ kV) showed that MPs on the order of 0.5 μm, which received the charge at a distance of 2.5 μm from the surface even at a velocity of 10 m/s, will stop moving without reaching the substrate.

5. Conclusions

The application of high voltage high-frequency short-pulse negative bias to a substrate immersed in vacuum arc plasma provides the possibility of the multiple decrease of MP number surface density and opens a possibility for realization plasma immersion metal ion implantation technology using DC vacuum arc plasma. The decreasing of MP number density on a negatively biased substrate is defined by several various physical mechanisms including ion sputtering, enhanced ion sputtering, and reflection by a sheath electric field, as well as MPs evaporation on substrate surface.

6. Acknowledgement

This work was performed within the state assignment “Science” of the Ministry of Education and Science of the Russian Federation.

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