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Tangential cathode magnetic field and substrate bias influence on copper vacuum arc macroparticle content decreasing

Igor B. Stepanov ^{*}, Alexander I. Ryabchikov, Peter S. Ananin, Denis O. Sivin, Alexey E. Shevelev, Sergei G. Zhelomsky

National Research Tomsk Polytechnic University, 634050, Lenina 2, bdg. 4, Tomsk, Russia

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ABSTRACT

The paper presents the results of an experimental study of accumulation of copper macroparticles (MP) on a negatively biased substrate immersed in DC vacuum arc plasma. The influence of normal and tangential magnetic fields and high-frequency short-pulsed negative bias was investigated. The joint application of a tangential magnetic field and high frequency short-pulsed negative bias provides an effect of MP multifold suppression. With a tangential magnetic field strength of 175 Gs and repetitively pulsed bias (7 μ s, 10⁵ p.p.s., -2 kV), total suppression efficiency is 250-fold after 6 min of ion-plasma treatment. For macroparticles with the diameter of less or >1 μ m, the efficiency is 3000-fold or 70-fold, respectively. In comparison with an axisymmetric vacuum-arc plasma source, the application of a steered arc ensures a 10-fold reduction in MP density on a substrate immersed in copper vacuum-arc plasma. The possibility of ion implantation using low-energy high-frequency short-pulse plasma immersion by implementing DC vacuum arc plasma is discussed.

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1. Introduction

Vacuum-arc discharge is a well-known method to obtain highly ionised plasma of any conductive material [1–4]. Vacuum arcs have found application in different techniques of thin coating deposition and ion-implantation. Despite these advantages, the main drawback of DC vacuum arcs is macroparticles (MP) in a plasma flow, which noticeably limits their application in areas where increased quality of ionmodified surfaces or deposited coatings is required. As a solution to the MP contamination problem, many plasma filtering (PF) systems were developed, which allow reducing the MP fraction in plasma up to 2–4 orders of magnitude [5–7]. However, a substantial plasma loss in the electromagnetic system of PFs reduces the production rate 3–4fold and, obviously, increases the costs.

A partial solution to decreasing the MP number can be based on an effect, which was found experimentally in Ref. [8]. The authors of this paper applied DC negative bias up to $\varphi = -1000$ V to a substrate immersed in vacuum arc plasma and observed generally 3–4-fold decrease in MP number after 10 min of TiN deposition. Papers [9–11,12] present a detailed study of the influence of a cathode material and plasma electron temperature on MP density reduction in metal and TiN coatings using DC (up to 1000 V) and repetitively pulsed (up to 300 V) negative bias. Significant increase in MP suppression efficiency was achieved in Ref. [13]. The authors applied a short-pulsed negative bias (pulse

* Corresponding author. *E-mail address:* stepanovib@tpu.ru (I.B. Stepanov).

http://dx.doi.org/10.1016/j.surfcoat.2016.04.003 0257-8972/© 2016 Elsevier B.V. All rights reserved. duration $\tau = 1-9 \,\mu$ s) to the substrate immersed in vacuum arc plasma, which allowed increasing the negative bias amplitude up to 3200 V without arcing. The high pulse repetition rate (100 kHz) provided high duty factor. The possibility of decreasing the total number density of titanium MPs by up to 2 orders of magnitude was demonstrated. The surface density of small MPs with the diameter <1.5 μ m was reduced by >3 orders of magnitude. The amount of large MPs was decreased only 10-fold. Low efficiency of the suppression of large MPs was also observed in those papers, where DC negative bias was used [9–11].

Thus, there is an interest in using the properties of vacuum arc discharge for decreasing the total amount of MPs and especially large ones. In the case of an axisymmetric magnetic field, the motion of the cathode spot on the cathode surface is chaotic [14]. Low velocity of the cathode spot motion leads to local overheating of the cathode surface and generation of a considerable amount of MPs [15]. Application of a tangential magnetic field significantly increases the cathode surface and, hence, the production of MPs [16–18].

This paper investigates the multifold suppression of copper MPs using high-frequency short-pulsed bias and tangential magnetic field.

2. Experimental setup

The experimental setup is shown in Fig. 1 and is described in more details in Ref. [19]. Two types of evaporators with tangential and normal magnetic fields were used in the experiments. DC vacuum-arc plasma generators with copper cathodes were located on the side flanges of

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Fig. 1. Experimental setup scheme.

the vacuum chamber. Argon plasma was formed using PINK arc generator with hot cathode [20]. The argon plasma ion saturation current density on the substrate was 1.4 mA/cm².

Short-pulse generator of the negative bias was used in experiments for biasing the substrate during its argon-plasma pretreatment and during its ion-plasma treatment by copper plasma. The parameters of the generator were fixed: pulse duration $\tau = 7 \,\mu$ s, pulse repetition rate f = 10⁵ pulse per second (p.p.s.), negative bias pulse amplitude ϕ = 2000 V. The mean copper ion charge state in vacuum-arc plasma is about 2 [21]; thus, the mean energy of the ions interacting with the substrate surface is about 4 keV. At such ion energy, the self-sputtering coefficient of copper coating is about 3 [22]. Taking into account the high duty factor of bias pulses, it is obvious that during ion-plasma substrate treatment the coating cannot be formed, and only the regime of low-energy plasma immersion ion implantation using DC vacuum-arc plasma is possible.

In the study, stainless steel substrates polished to $R_a = 0.035 \,\mu m$ were used. The substrates were mounted on a massive holder. The distance from the samples to the cathode surface of the vacuum-arc evaporator was 24 cm. The surface of each substrate was pretreated with ions using argon plasma and a high-frequency short-pulsed bias. Measured plasma ion saturation current density for the evaporator with normal magnetic field was 8 mA/cm² for the discharge current of 100 A. In the case of steered arc source, the discharge current was increased up to 150 A, to obtain approximately the same ion current density.

The MP densities on the substrate surface were studied using electron microscopes (Hitachi TM–1000 and Hitachi TM–S 3400N). Experimental data on the change in the surface number density of MPs are presented in the figures in absolute units. Taking into account the appreciable inhomogeneity of MP distribution on the substrate surface for each experimental point, the total area for MP quantification amounted to $60,000 \,\mu\text{m}^2$.

3. Influence of normal magnetic field

A microphotography of Cu-coating formed using axisymmetric evaporator after 6 min of plasma deposition on the substrate with anode potential is shown in Fig. 2a. The dependencies of the surface number density of macroparticles versus deposition time are presented in Fig. 3, curve 1 for small (diameter d < 1 μ m,) and curve 2 for large (d > 1 μ m) MPs. Total surface number density of MPs sharply increases during the first 0.5 min of plasma deposition exceeding 3 \cdot 10⁶ particles/cm². Further increment of processing time shows gradual increase in the number density of MPs. After 6 min of plasma deposition, there is





Fig. 2. SEM images of the substrate surface after 6 min of ion-plasma treatment (normal field): a – substrate under anode potential; b – substrate under repetitively pulsed negative bias.

an obvious trend towards saturation, related to the burying of small MPs under the growing coating. Scanning electron microscope (SEM) images show that the majority of MPs are <1 μ m. The minimal size of counted MPs with given microscope resolution is about 100 nm. The number of large MPs is negligible, but their role in surface contamination is prevailing. Most MPs with diameters of up to dozens of micrometers are flattened. This means that in the moment of interaction with the substrate surface, such MPs were in a liquid state.



Fig. 3. Surface density of MP number versus processing time in the case of normal magnetic field: 1, 2 – anode potential; 3, 4 – repetitively pulsed negative bias.

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Fig. 3 (curves 3 and 4) presents the dependencies of the surface number density of MPs versus processing time at repetitively pulsed negative bias. Application of high-frequency short-pulsed negative bias to the substrate significantly impacts the dynamics of MP accumulation on the substrate surface during ion-plasma treatment. By increasing ion-plasma treatment time from 0.5 to 6 min, the efficiency of total MP suppression grows from 3-fold to 40-fold, as compared to plasma deposition with anode potential. MP surface density for macroparticles with the diameter larger than 1 μ m grows from 7 \cdot 10⁴ up to 5 \cdot 10⁵ particles/cm² after increasing ion-plasma treatment time from 0.5 to 6 min (Fig. 3, curve 3). Fig. 3 (curve 4) demonstrates high efficiency of elimination of small MPs during the ion-plasma treatment. SEM image in Fig. 2b illustrates not only flattened MPs, but also many different marks from MPs that have the same microstructure as the surrounding surface.

Accumulation of large MPs during processing practically excludes the possibility of using a DC copper vacuum-arc with normal magnetic field for high-frequency short-pulsed plasma immersion ion implantation. For the purpose of general reduction in the amount of MPs (especially large ones), experiments with a magnetic field tangential to the cathode of the vacuum arc plasma source were carried out.

4. Influence of tangential magnetic field

Our experiments were carried out with tangential magnetic field strength from 60 to 250 Gs with the arc discharge current of 150 A.

The dependence of the ion plasma saturation current density versus magnetic field for the distance from the substrate to the cathode surface of 24 cm is presented in Fig. 5. Ion current density linearly decreases from 10 to 4.5 mA/cm², while the magnetic field strength increases from 60 to 250 Gs. Increasing magnetic field strength leads to the magnetisation of plasma electrons and, as a result, to a decrease in the ion plasma saturation current density.

As shown in Fig. 6, the cathode spot motion velocity also depends on magnetic field strength magnitude. After magnetic field strength rises from 60 to 250 Gs, the cathode spot motion velocity increases from 4 to 15 m/s. According to the plot, there is an obvious trend of the cathode spot motion velocity to saturation, when the magnetic field strength exceeds 180 Gs. Curve 2 in Fig. 6 demonstrates that the change in the surface density of MPs depends on the tangential magnetic field strength. Visual comparison of curves 1 and 2 leads to the conclusion that generated MP flux is inversely proportional to the cathode spot motion velocity. After increasing the magnetic field strength from 60 to 150 Gs, according to the data from Fig. 6, MP surface density decreases 4-fold. Further increase in magnetic field strength from 150 to 250 Gs hardly affects the MP density on the substrate surface. It is important to note that despite increased discharge current, the observed quantity of droplets is less than in the experiment with normal magnetic field. Visual comparison of Figs. 2a and 4a demonstrates the reduction of the maximal size of MPs.

The investigation of the influence of ion-plasma treatment time on MPs accumulating on the substrate surface was conducted with tangential magnetic field strength 175 Gs to achieve the ion plasma saturation current density similar to that in the experiments with the axisymmetric evaporator. Curves 1 and 2 in Fig. 7 demonstrate the accumulation of MPs on the substrate surface during coating deposition at anode potential. The comparison of deposited MPs at anode potential presented in Figs. 3 and 7 shows that the application of the tangential magnetic field (175 Gs) causes a 5-fold reduction in the MP surface number density after 6 min of vacuum-arc plasma deposition.

High-frequency short-pulsed negative bias differently affects the accumulation of large (>1 μ m) and small (<1 μ m) MPs on the substrate surface. The surface density of small MPs drastically decreases. After 6 min of treatment, the total amount of small MPs drops by nearly 3 orders of magnitude. At the same time, curve 3 in Fig. 7 shows an increase in the surface density of large MPs. After 0.5 min of ion-plasma substrate treatment, the surface density of small MPs is >2 orders of magnitude





Fig. 4. SEM images of the substrate surface after 6 min of ion-plasma treatment (tangential field): a – substrate under anode potential; b – substrate under repetitively pulsed negative bias.

higher than that of the large ones. Further increasing the ion-plasma processing time from 0.5 to 6 min leads to increased surface density of large MPs from $7 \cdot 10^3$ to $8 \cdot 10^4$ particles/cm². SEM image in Fig. 4b demonstrates the effect of multifold suppression of MPs on a negatively biased substrate as compared to vacuum-arc plasma deposition on the substrate with anode potential after 6 min of processing (Fig. 4a).

Comparison of the data presented in Figs. 3 and 7 allows making a conclusion that joint usage of steered arc and high-frequency shortpulsed negative bias provides a multifold suppression of MPs. The application of a tangential magnetic field instead of normal one reduces the



Fig. 5. Plasma ion saturation current density versus tangential magnetic field strength for cathode surface-to-substrate distance L = 24 cm.

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Fig. 6. Cathode spot velocity (1), surface density of MP number after 0.5 min of exposure under anode potential (2) and repetitively pulsed negative bias (3) versus magnetic field strength.

total amount of MPs by up to one order of magnitude and, what is especially important, the amount of the large MPs. As a result, after 6 min of ion-plasma treatment, the surface density of small MPs is reduced down to $7 \cdot 10^3$ particles/cm² (Fig. 7, curve 4), which is 10-fold lower than in the case of normal magnetic field (Fig. 3, curve 4). The surface density of large MPs in the case of steered arc and repetitively pulsed negative bias is also one order of magnitude lower than when evaporator with normal magnetic field is used. Total suppression efficiency is 250-fold after 6 min of ion-plasma treatment. For macroparticles with the diameter less or >1 µm, the achieved efficiency is 3000-fold or 70-fold, respectively.

The achieved efficiency is comparable to the effect of MP filtering by the majority of PFs [5–7]. However, in this case, metal plasma flux near negatively biased substrate remains, as opposed to plasma losses in electro-magnetic filtering systems. The results allow considering the possible implementation of this approach for developing 3dimensional low-energy plasma-immersion metal ion implantation using DC vacuum-arc plasma for modifying complex-shape parts. The application of high-frequency short-pulse negative bias with corresponding bias pulse duty factor can solve the problem of the ion sputtering of surface through its compensation by deposition of plasma. The authors plan to further investigate the dynamics and specificity of accumulation and diffusion of an implanted dopant in the case of three-dimensional low-energy plasma-immersion implantation of metal ions.



Fig. 7. Surface density of MP number versus processing time in the case of tangential magnetic field: 1, 2 – anode potential; 3, 4 – repetitively pulsed negative bias.

5. Conclusions

Joint application of tangential magnetic field and high-frequency short-pulsed negative bias provides a synergistic effect of MP suppression. Implementing tangential magnetic field with strength of 175 Gs and repetitively pulsed bias (7 μ s, 10⁵ p.p.s., -2 kV) allowed increasing the total suppression efficiency 250-fold after 6 min of ion-plasma treatment. For macroparticles with the diameter less or >1 μ m, the efficiency is 3000-fold or 70-fold, respectively. As compared to the axisymmetric vacuum-arc plasma source, the use of a steered arc ensures a 10-fold reduction in MP density on a substrate immersed in copper vacuum-arc plasma. The present data suggest the potential application of DC vacuum-arc for plasma-immersion ion implantation with considerable purification of the substrate surface without decreasing the ion current density.

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