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TITANIUM MACROPARTICLE FRACTION SUPPRESSION WITH STEERED ARC AND NEGATIVE PULSE BIASING

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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. The influence of normal and tangential to cathode surface magnetic field and high-frequency short-pulsed negative bias has been investigated. It was shown that the application of tangential to cathode surface magnetic field instead of normal one allows to reduce titanium MP surface density 2 fold. Applying of short-pulsed bias leads to decreasing of total MPs surface density after 3 minutes of ion-plasma treatment 30 fold.

INTRODUCTION

A vacuum arc discharge is well-known method to obtain highly ionized plasma of any conductive material (Plyutto et al., 1965; Lunev et al., 1976; Kondrat'eva et al., 1999). Cathode arc found its application for variety of deposition technics of thin coatings and ion implantation technology. Despite its excellence the main disadvantage of a vacuum arc is macroparticle (MP) contamination which roughly limited its application in areas where high qualities of coatings are demand. As solution of MPs problem many plasma filtering systems (PF) are developed (Aksenov et al., 1978; Anders, 1999; Ryabchikov et al., 2005). PFs usually allow to reduce MPs up to 2-4 orders but also provide a very limited possibilities of plasma transportation through their magnetic and electromagnetic systems. Some approaches propose to suppress generation of MPs based on vacuum arc discharge properties (decreasing of cathode temperature, pulsed discharge, etc.) (Bizyukov et al., 2006; Proskurovsky et al., 2007). P.D. Swift showed that using tangential to cathode surface magnetic field noticeably increase velocity of cathode spot motion which leads to decreasing of local temperature of cathode and as following generation of MPs (Swift, 1996). Studying of MPs size distribution showed that most significant effect acquired for micron-sized MPs.

experimentally in works (Tau et al., 1990). The authors of these papers applied negative bias up to φ = - 1000 V to substrate immersed in vacuum arc and observed general decreasing of MPs in 3-4 times after 10 minutes of TiN deposition. Further development of approach leads authors of papers (Guoqiang et al., 2004; Zhang et al., 2012; Stepanov et al., 2016) to using short-pulsed high frequently negative bias which allows to use potentials up to φ = - 3.2 kV. They observed significant reduction of titanium and aluminum droplets on a substrate immersed in vacuum arc. For MPs with diameter less than 1 µm and treatment time 18 minutes in titanium plasma total reduction of MPs was about 1500 times. Total reduction for titanium MPs of all sizes after 18 minutes of plasma deposition was about 67 times.

Taking into account high efficiency of decreasing small MPs with short-pulsed high frequency negative bias and reduction of generation micron-sized MPs with steered arc brings to idea of combining both methods to receive droplets-free substrate surface after ion-plasma treatment. This paper deals with studying influence of normal and tangential magnetic field to cathode surface and repetitively pulsed negative bias applied to substrate immersed in titanium plasma on MPs accumulation.

MATERIALS AND METHODS

An effect of MPs reduction has been observed

Investigation of magnetic field influence on MPs

generation was carried out with two types of vacuum arc evaporators. Cathodes of both evaporators made of titanium. The first is traditional cathode with normal to surface magnetic field. The second is evaporator with tangential to cathode surface magnetic field or so called steered vacuum arc generator. Both evaporators are shown in Fig. 1.

Short-pulsed negative bias generator with following parameters has been used in experiments: pulse duration τ = 7 µs, pulse repetition rates f = 10⁵ pulse per second (p.p.s), negative bias pulse amplitude U_b = 2 kV.

Polished to $R_a = 0.035 \mu m$ stainless steel substrates are used. The substrates were mounted on a massive holder. The distance from samples to the cathode of

the vacuum arc evaporator was 24 cm. The surface of each substrates was pretreated with ions using argon plasma and the high-frequency short-pulsed bias.

The MP densities on the substrate surface were studied using electron (Hitachi TM–S 3400N) microscopes. Experimental data on changes found in MP number surface density are presented in the fig in absolute units. For each experimental point, total area for MP calculation made $60,000 \ \mu m^2$.

RESULTS

Influence of Normal to Cathode Surface Magnetic Field

In case of normal to cathode magnetic field a deposition of titanium plasma on substrate at anode



Fig. 1 The experimental installation scheme.



Fig. 2 SEM photographs of substrate surface after 3 min of a treatment (normal field): a) anode potential, b) negative pulsed bias (τ =7 µs, f=10⁵ p.p.s, U=- 2 kV).

potential reveals large amount of MPs. Fig. 2a shows the substrate surface after 3 minutes of plasma deposition. Scanning electron microscope (SEM) images show that the majority of MPs have diameter less than 1 μ m. Dependence of MP surface number density on processing time at anode potential is shown in Fig. 3 (curve 1). Evidently without negative bias total number of MPs on substrate surface increases gradually. After 3 minutes of plasma deposition one may observe trend to saturation related to burying of MPs under growing coating.

Applying of high-frequency short-pulsed negative bias significantly affects on MP surface density and dynamic of their assembling on the substrate during the treatment time. Dependence of MP surface density on processing time at high-frequency short-pulsed negative bias is shown in Fig. 3 (curve 3). Surface density of titanium droplets gradually decreases from 7.105 to 5.105 particle/cm2 with ionplasma substrate processing time increasing from 30 seconds to 3 minutes. Total reduction of MPs after application of high-frequency short-pulsed negative bias in compare with plasma deposition at anode potential increased from 3 fold after 30 second to almost 15 fold after 3 minutes of a substrate treatment. Variation of high-frequency short-pulsed negative bias's duty factor allows to achieve compensation of ion-sputtering by plasma deposition and as a result exclude real sputtering of sample surface (Ryabchikov et al., 1991; Sharkeev et al., 1995).



Fig. 3 MP surface number density versus processing time at anode and negative bias potential (7 μ s, 10⁵ p.p.s, 2 kV): 1-anode potential, normal field; 2-negative bias, normal field; 3-anode potential, tangential field ; 4-negative bias, tangential field.

Influence of Tangential to Cathode Surface Magnetic Field

In case of tangential to cathode magnetic field a deposition of titanium plasma on substrate at anode potential reveals reduced amount of MPs. This reduction binds with increasing velocity of cathode spot motion which leads to decreasing of

local cathode surface temperature. Cathode spot motion velocity depends on tangential magnetic field strength. However, increasing of magnetic field strength of vacuum arc evaporator with tangential field leads to electron magnetization, decreasing of ion current density and increasing of discharge voltage drop. In our experiments influence of magnetic field strength from 50 Gs to 200 Gs was investigated. Dependences of ion current density and cathode spot velocity versus magnetic field strength measured with cathode to sample distance L = 24 cm are presented in fig.4. Ion current density linearly decreases from 10 to 5.5 mA/cm² when magnetic field strength changes from 50 to 200 Gs (Fig. 4, curve 1). Cathode spot velocity changes from 12.5 m/s (50 Gs) to 22.5 m/s (200 Gs).



Fig. 4 Ion current density (1) and cathode spot velocity (2) versus tangential magnetic field strength on cathode-tosubstrate distance L=24 cm.

Dependence of MP surface density versus magnetic field strength is presented on Fig. 5. Increasing of magnetic field strength from 50 to approximately 200 Gs leads to gradual reduction of MPs generation. MPs surface density decreases up to 4.5 fold.



Fig. 5 MP surface number density versus magnetic field strength after 30 sec of coating deposition.

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Fig. 6 SEM photographs of substrate surface after 3 min of a treatment (tangential field, 190 Gs): a) anode potential, b) negative pulsed bias (τ =7 µs, f=10⁵ p.p.s, U=- 2 kV).

Investigation of processing time influence on MPs assembling dynamics on a target was carried out with tangential magnetic field strength 190 Gs. Curve 2 of Fig. 3 demonstrates MP surface density changes on a sample with anode potential during plasma deposition. Comparison of curves 1 and 2 (Fig. 3) shows that application of tangential to cathode surface magnetic field provide 2 fold MP surface density reduction after 3 min of vacuum arc plasma deposition.

Using of high-frequency short-pulsed negative bias effects on an assembling of MPs on a substrate. As one may see out Fig. 3 (curve 4) MPs surface density reduced from 5.7·10⁵ to 2.6·10⁵ particles/cm² after increasing ion-plasma treatment time from 30 sec to 3 min. After 3 minutes of ion-plasma treatment amount of MPs on a sample surface decreases 20 fold compare to plasma deposition on a substrate with anode potential. SEM images (Fig. 6a and 6b) shows effect of multiply decreasing of MP density on a sample surface with negative pulsed bias compared to vacuum arc deposition on a substrate with anode potential.

Comparison of data illustrated on Fig. 3 allows to made conclusion about preference of joint using of tangential to cathode surface magnetic field and high-frequency short-pulsed negative bias in case of titanium vacuum arc plasma. MP surface density after 3 min of plasma deposition with normal to cathode surface magnetic field (Fig. 3, curve 1) surpass similar value in case of tangential magnetic field and negative bias 30 fold (Fig. 3, curve 4). Generally, one may conclude that obtained experimental results show possibility of DC vacuum-arc usage for plasma immersion ion implantation with significant suppressing of MPs on a substrate surface without decreasing of plasma and ion current densities.

CONCLUSION

Joint application of tangential to cathode surface magnetic field and high-frequency short-pulsed negative bias provides significant decreasing of MP density on a sample surface immersed in titanium DC vacuum-arc plasma. Usage of tangential magnetic field decreases generation of MPs 2 fold. In case of normal to cathode surface magnetic field negative bias allows to decrease MP density on a substrate 15 fold in comparison with traditional vacuum arc plasma deposition after 3 min. Applying of tangential magnetic field and negative pulsed bias increases total efficiency of MPs elimination to 30 after 3 min of ion-plasma treatment in comparison with plasma deposition using of axisymmetric vacuum arc evaporator. Given results show possibility of DC vacuum-arc application for plasma immersion ion implantation with significant suppressing of MPs on a substrate surface without decreasing of plasma and ion current densities.

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REFERENCES

- Aksenov I, Belous V, Padalka V, Khoroshikh V. 1978. Apparatus to rid the plasma of vacuum arc of mocroparticles. *Instrum. Exp. Tech.* 21: 1416-1418.
- Anders A . 1999. Approaches to rid cathodic arc plasmas of macro- and nanoparticles: a review. *Surf. Coat. Technol.* 120-121: 319-330.
- Bizyukov A, Sereda K, Kashaba A, Romaschenko E,

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Chibisov A, Ponevchinskiy V, Sleptsov V. 2006. Evaporation of macroparticles in plasma of highcurrent pulsed arc discharge at low pressure. *Problems. Atomic. Sci. technol.* 5: 136.

- Lin G, Zhao Y, Guo H, Wang D, Dong C. 2004. Experiments and theoretical explanation of droplet elimination phenomenon in pulsed-bias arc deposition. J. Vac. Sci. Technol. A. 22: 1218-1223.
- Kondrat'eva N, Koval N, Korolev Yu , Schanin P. 1999. A spectroscopic investigation of the nearcathode regions in a low-pressure arc. *J. Phys. D: Appl. Phys.* 32: 699-704.
- Lunev V, Padalka V, Khoroshikh V. 1976. Plasma properties of a metal vacuum arc. Instrum. Exp. Tech. 19: 1465-1495.
- Plyutto A, Ryzhkov V, Kapin A. 1965. High Speed Plasma Streams in Vacuum Arcs. J. of Exp. Theoret. Phys. 20: 328-337.
- Proskurovsky D, Popov S, Kozyrev A, Pryadko E, Batrakov A and Shishkov A. 2007. Droplets evaporation in vacuum arc plasma. *IEEE Trans. Plasma. Sci.* 35: 980-985.
- Ryabchikov A, Nasyrov R. 1991. Repetitively pulsed, high-concentration implantation. *Nuclear Inst. Methods. Phys. Res*. 61: 48-51.

- Ryabchikov A, Ryabchikov I, Stepanov I, Sivin D. 2005. Complementarity of Xray diffraction and RBS in thin film characterization. *Vacuum*. 78: 445-449.
- Sharkeev Yu, Girsova N, Ryabchikov A, Kozlov E, Perevalova O, Brown I, Yao X. 1995. Dislocation structure in coarse-grained copper after ion implantation. *Nuclear Instrum. Methods. Phys. Res.* 106: 532-537.
- Stepanov I, Ryabchiko A, Ananin P, Shevelev A, Sivin D, Zhelomsky S. 2016. Tangential cathode magnetic field and substrate bias influence on copper vacuum arc macroparticle content decreasing. *Surf. Coat. Technol.*
- Swift P. 1996. Macroparticles in films deposited by steered cathodic arc. *J. Phys. D: Appl. Phys.* 29: 2025-2031.
- Tau C, Koh E, Akari K. 1990. Macroparticles on TiN films prepared by the arc ion plating process. *Surf. Coat. Technol.* 43-44: 324-346.
- Zhang G, Gao G, Wang X, Lv G, Zhou L, Chen H, Pang H, Yang S. 2012. Influence of pulsed substrate bias on the structure and properties of Ti–Al–N films deposited by cathodic vacuum arc. *Appl. Surf. Sci.* 258: 7274-7279.