Investigation of Tungsten DC Vacuum Arc Characteristics. Technological Application[1]

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Abstract - The paper presents the results of investigation of regularities of generation of W vacuum arc plasma continuous flows and their application for deposition of coatings with high adhesion strength.

The developed construction of the vacuum arc evaporator (VAE) ensures dc mode of W plasma generation in the current range of 350 A – 750 A at arc voltage drop of more than 40 V. It is shown that at discharge current of 750 A W coating deposition rate can achieve 80 µm/h, which offers attractive perspectives for a number of technological applications of W plasma.

We also investigated some regularities of changes in element composition, adhesion strength, hardness, morphology and structure of W coatings formed from plasma of a dc vacuum arc discharge (VAD) depending on regimes of coating deposition. It is shown that use of Ti plasma for generation of a thick transition and damping layer under the regime of plasma immersion ion implantation and coating deposition under intensive ion mixing allows increase adhesion strength of W coatings by several times.

1. Introduction

Application of VAD for generation of metal plasma is widely used for technologies of plasma deposition of protective and strengthening coatings. Presently Ti, Al, Cu, Cr and a number of other metals are very popular for generation of vacuum arc plasma flows [1]. Combination of the above-mentioned materials with other materials (in composite cathodes) or with reaction gases allows obtain nitrides, oxides, carbides, borides, intermetallic compounds, as well as other composite compounds, which expands application fields for PVD coatings.

At the same time a number of practical applications require coatings with a complex of such physico-chemical properties as low chemical activity, element purity, homogeneous structure, high corrosion- and heat resistance. The most attractive way to solve this problem seems to be use of plasma of heat-resistant materials. For a number of reasons generation of vacuum arc plasma of such materials using standard technological equipment especially in a dc mode is regularly difficult (complicated mechanical treatment of cathodes, stabilization of cathode spots on cathode operational surfaces, high currents and high discharge voltages, high level of inner tensions in generated coatings, etc.)

The presented paper aims at creation of technological equipment adapted to operation using tungsten cathodes and investigation of characteristics of generated plasma flows and discharges, and regularitiesof W-based coating deposition.

2. Generation of tungsten plasma continuous flows

Fig. 1 shows schematic of the experimental set-up used for deposition of W coatings, equipped with two VAEs, one with W cathode, another with Ti cathode. VAE with Ti cathode has a filter for cleaning vacuum arc plasma from microparticle fraction [2]. VAE discharge gap consists of a massive water-cooled cathode and hollow cylindrical anode. The cathode is fixed on the holder, which ensures immediate contact between the cathode and cooling liquid and excludes complicated mechanical treatment of material.

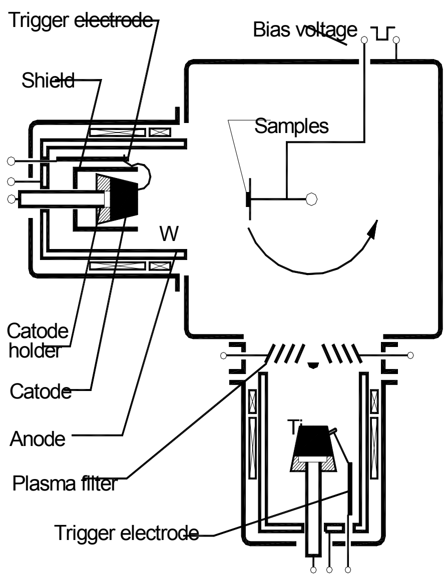


Fig. 1. Schematic of the experimental set-up.

Discharge in VAE with Ti cathode is ignited conventionally, by applying discharge onto the dielectric

[1] The work was supported by awards of CRDF RR2-994, RR2-1030 and award of the Ministry of Education of RF for the Federal Goal Program "Integration" - № Л0059.

surface. The schematic of discharge ignition with W cathode is based on electrical breakdown of the vacuum gap, formed when the triggering electrode is pulled off the cathode. The triggering schematic allows initiate the discharge with the frequency of up to 25 Hz on the front surface of the cathode. Stability of discharge formation is ensured by emission of energy of up to 15 J on the gap.

Stabilization of the cathode spot on the front surface of the cathode is ensured by outer magnetic field of up to 12 mT generated by two coils with current. In order to avoid the shift of the cathode spot onto the cathode lateral surface and holder elements, protective cylindrical screen was used in VAE with W cathode.

Fig. 2 (curve 1) shows the dependence of change in VAD life on current. The presented data show that the discharge dc regime is realized at the current of more than 350 A for a given VAE construction. For this current value characteristic voltage drop at the arc is about 40 V (curve 2).

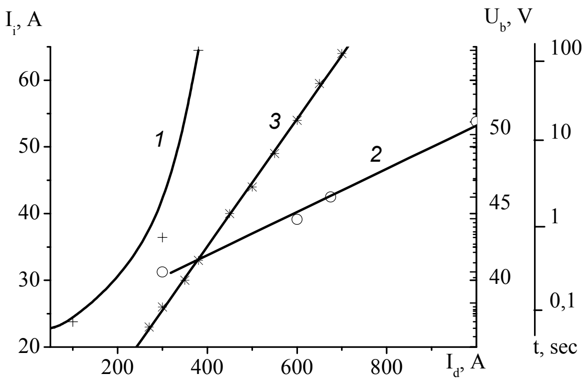
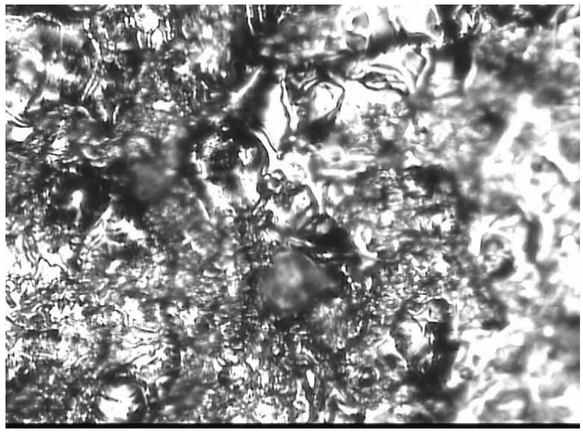


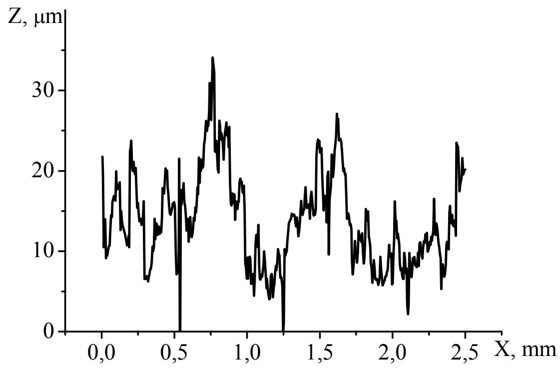
Fig. 2. Parameters of a dc VAD with W cathode:
1 – Dependence of discharge glow time on VAD current;
2 – Volt-ampere characteristics of the VAD; 3 – Saturation ion current from plasma depending on VAD current.

Changes in ion current from I_i plasma depending on discharge current I_d were measured at pressure in the vacuum chamber equal to $4{\cdot}10^{-4}$ Pa and outer magnetic field equal to 10 mT. Collector with negative potential was located at the source output. The dependence presented at Fig. 2 (curve 3) shows that for W plasma we have $I_i \cong (0{,}08 \div 0{,}09)\ I_d$, and plasma flow is generated with pronounced increase in ion current density j_i near its axis. This phenomenon can be explained by the fact that we employed some means to compulsorily stabilize the cathode spot on cathode front surface, as well as by the proper magnetic field generated at discharge currents equal to several hundreds of amperes.

Fig. 3 shows the pictures and profile of W cathode surface. The data confirm that after many hours of operation the cathode surface has low roughness, which means low coefficient of cathode erosion. We see that under the given experimental conditions W erosion coefficient does not exceed $40{\cdot}10^{-9}$ kg/C.



a)



b)

Fig. 3. Pictures (a) and profiles (b) of W cathode surface after 8 hours of operation.

Absence of sharp fringes on the cathode surface means that during operation the temperature of the cathode surface was near the melting temperature. Paper [3] shows that a number of droplets produced by W arc cathode spot per one second is $3{\cdot}10^3$, which is one order of magnitude less than for Al.

Despite high melting temperature of W, the largest portion of microparticle fraction observed in our experiments had spherical droplet shape, which shows that it was formed from the liquid phase. Maximum size of observed droplets didn't exceed 30 μm. At the same time we discovered impurities with the size of 50 μm with sharp fringes at the surface of the analyzed sample. Shape of these particles is characteristic of the microparticle fraction formed from the solid phase. As a rule, the mechanism of such microparticle fraction formation can be explained by appearance of high mechanical tensions in cathode material. It was noticed that probability of microparticle fraction formation in solid aggregative state decreases with the increase in VAD glow duration.

3. Deposition of tungsten coatings and investigation of their properties

Tungsten coatings were deposited onto steel 3 samples with the diameter of 50 mm. Before deposition the sample surface was mechanically polished and burnished using abrasive pastes ACH 40/28, 14/10 up to the roughness Rz ≈ 1,28 μm and chemically cleaned in the ultrasonic bath.

Ti plasma cleaned from microparticle fraction was used to prepare the surface for W coating deposition. We executed the following stages: ion etching, formation of a surface layer with large amount of active sorption centers, heating of samples, formation of a substrate-coating transition layer, and deposition of a damping layer.

High intensity ion treatment of the surface was realized under high frequency generation of short negative voltage pulses on samples [4]. Pulsed generation of a negative potential allows avoid formation of erosion centers on a sample surface and ensures stable operation of the power supply system. Various regimes of ion-beam and ion-plasma material treatment are realized at the change of the amplitude and pulse duty factor in the range (0,3 ÷ 2) kV and 0,1 ÷ 0,66, respectively. The coating was deposited when we switched on the VAE with W cathode facing sample operational surface. The pressure in the vacuum volume was ensured by a diffusion pump and was equal to 10^{-3} Pa. The temperature was (400 ÷ 450) ^{0}C.

Fig. 4 shows dependencies of W coating condensation velocity changes along plasma flow cross section. The data were obtained by measuring the thickness of the obtained coating using the method of "spherical section" [5]. The presented data show that when the samples were located at the distance of 450 mm from the source, coating deposition rate could reach 80 μm/h. Since the plasma flow expands quickly, homogeneity of formed coating thickness distribution reaches 82 %, which together with W coating formation rate offers attractive perspectives for a number of technological applications.

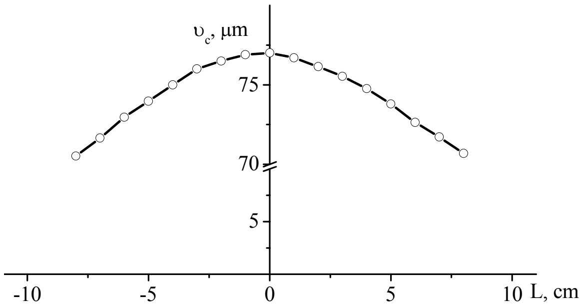


Fig. 4 Change in coating deposition rate across plasma flow cross-section: I_d = 650 A U_b = 500 V.

Fig. 5 shows the results of morphological investigation of W coating surface using the Micro Measure 3D Station (STIL) [5]. The presented data show that roughness of the formed coating is 4,12 μm along Rz. Increase in coating roughness compared to sample initial state is related to presence of a small amount of microparticle fraction in plasma flow and formation of phases with a large size of grains (up to 12 μm). More homogeneous structure and decrease in surface roughness up to 2,88 μm along Rz were obtained for coatings generated at lower discharge current and increased bias potential (Fig. 5b).

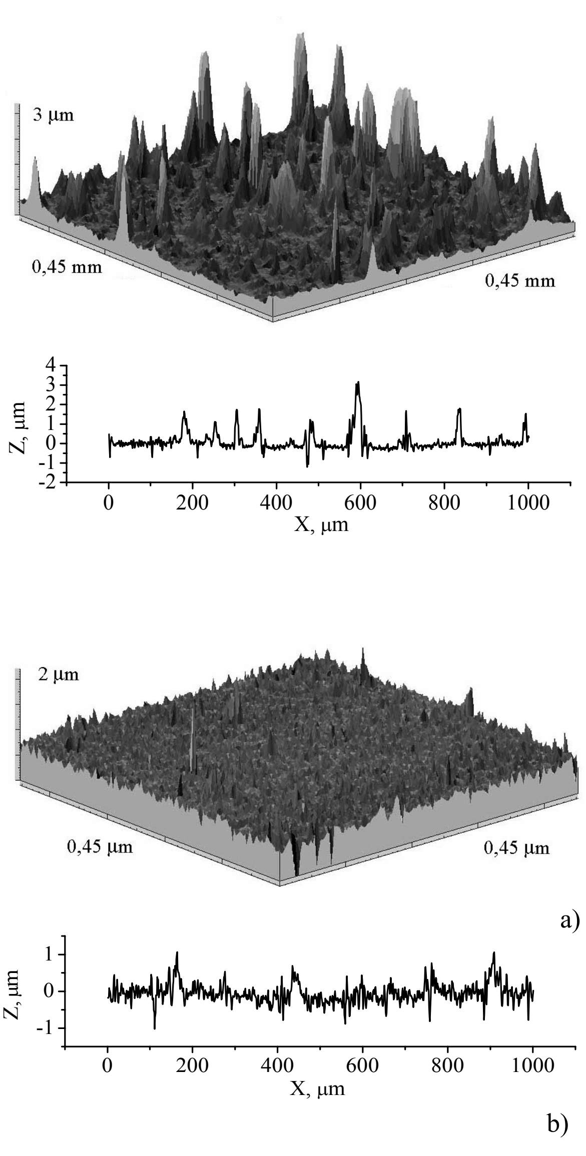


Fig. 5. 3D image and profile of W coating surface with the thickness of 4 μm: a) I_d = 750 A, U_b =500 V, b) I_d = 550 A, U_b =1000 V.

It was experimentally shown that W condensation velocity and bias potential on samples influence both change in coating surface morphology and coating surface structure. In particular, porosity of coatings formed under *1* - I_d = 750 A, U_b = 500 V and *2* - I_d = 500 A, U_b = 1000 V was changed. We calculated porosity as ratio of coating density *1*- $14,3 \cdot 10^3$ kg/m^3 and *2*- $16,9 \cdot 10^3$ kg/m^3 and W polycrystal density - $18,8 \cdot 10^3$ kg/m^3. In first case coating porosity was 76 %, in second – 90 %.

Coating hardness was measured using the Nano Hardness Tester (CSM) [5]. Hardness of the coating formed under the first coating deposition regime (Fig. 6, curve 2) was HU = 3227 MPa. Coating hardness increased up to HU = 4703 MPa with decrease in discharge current and increase in bias voltage. Curve 1 shows change in conditions of indenter loading and unloading.

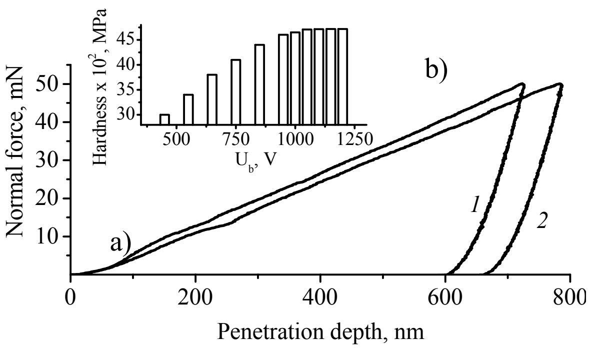


Fig. 6. a) Indenter loading depending on its penetration: *1*-, I_d = 550 A, U_b = 1000 V, *2* - I_d = 750 A, U_b =500 V. b) Dependence of change in coating hardness on sample potential.

Fig. 6b shows dependence of increase in W coating hardness on increase in bias potential. This effect can be explained by the regime of coating formation under intensive ion mixing accompanied by changes in phase-formation conditions, intensive diffusion processes, and surface ion sputtering processes.

Coatings from materials with high melting temperatures are characterized by high inner tensions formed both at the stage of coating deposition in the field of temperature gradients and during coating exploitation because of difference between the coefficients of thermal expansion of the coating and substrate [6].

In order to decrease inner tensions of W coating, a gradient structure between the substrate and the coating was formed. The structure was composed of a thick transition layer formed along the depth of the model sample and thin damping layer at its surface. Fig. 7 shows distribution of element composition along the depth of the formed coating and model sample surface layer.

The presented data show that the transition Ti layer with the thickness of ~ 700 nm was formed along the depth of the sample surface layer. The layer was formed under the regime of high intensity short-pulsed high frequency plasma immersion ion implantation, which stimulated diffusion of titanium deeper into the layer [4].

In order to compensate inner tensions in W layer, Ti layer with the thickness of ~ 500 nm was deposited onto the model sample surface under the regime of ion assistment.

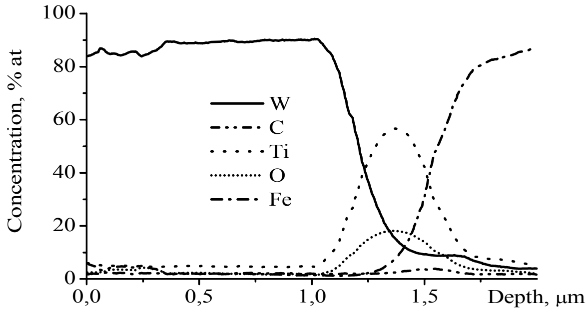


Fig. 7. Distribution of element composition along the depth of W coating and sample surface layer.

Formation of the transition layer between Ti and W was executed at simultaneous treatment of the sample by Ti and W plasma flows. Formation of the second transition layer was also executed under high-intensity ion mixing.

Adhesion strength of coatings was measured using Scratch testing methods and the Micro-Scratch Tester (CSM) [5]. W-coated samples with the thickness of 8 μm formed at U_b = 500 V and T = 400 ^{0}C were analyzed. Before testing the samples were heated and cooled down in the temperature range of 20 ^{0}C - 400 ^{0}C during several hours. Fig. 8 and 9 show the results of investigations in the form of the output signal from acoustic emission sensor and pictures of the indenter track.

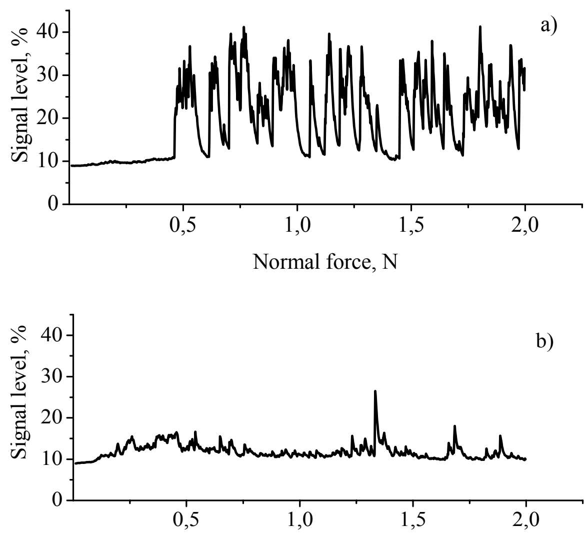
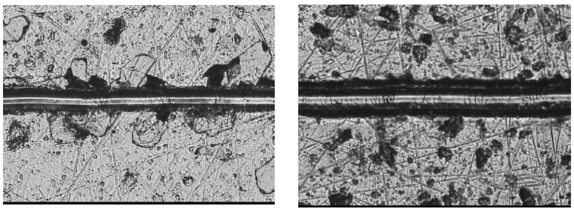


Fig. 8. Level of acoustic emission from loading on indenter.

Fig. 8a shows the processes of destruction of W coating formed in the absence of damping Ti layer. The results show that the process of film destruction began at the loading of ~ 0,5 N. Fig. 9a shows the areas of the delaminated coating formed at critical indenter loading. Destruction characteristics reveal high inner tensions in the material.



a) b)

Fig. 9. Image of W coating surface near the indenter track a) in absence of damping Ti layer; b) with damping Ti layer.

We obtained different data for W coating with a damping Ti layer. Changes in Fig. 8b show that along the whole track from the indenter we can observe low level of acoustic emission, which means increase in coating adhesion strength. Coating destruction starts at loading equal to 1,35 N, which is confirmed by the image at Fig. 9b. For this case coating destructions characteristic of high level inner tensions are absent near the indenter track as well. The obtained data confirm positive influence of the formed Ti layer on compensation of inner tensions arising at thermocyclic coating loadings.

During investigation of regularities of W coating formation using plasma of dc VAD, it was found out that increase in intensities of ion treatment causes increase in coating adhesion strength. Increase of sample temperatures in the range of 300 ^{0}C - 450 ^{0}C does not cause decrease in adhesion strength of formed coatings. At the same time at higher temperatures of coating deposition one can expect decrease in coating adhesion strength due to increase in temperature gradients during formation of coatings.

It was experimentally found out that increase in coating generation rate decreases coating adhesion strength. However, to investigate regularities of the process in details, an additional research is required.

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