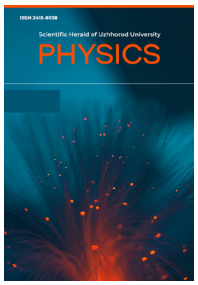
Scientific Herald of Uzhhorod University
Series "Physics"

Journal homepage: https://physics.uz.ua/en
Issue 53, 9-18
Received: 03/02/2023. Revised: 05/01/2023. Accepted: 05/29/2023



UDC 537.525, 621.793.02, 621.793.1
PACS 52.50.Dg, 52.77.Bn, 52.80.Vp, 68.37.Hk, 81.15.Ef, 81.15.Jj, 81.15.Rs, 81.65.Cf
DOI: 10.54919/physics/53.2023.09

Thermionic coating method with preliminary bombardment of the substrate surface with a stream of low energy ions

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Abstract

Relevance. Vacuum-plasma methods of applying functional coatings are widely used to increase the reliability and durability of machine and mechanism parts, cutting tools, and technological equipment, as well as in modern micro- and nanoelectronics for applying conductive and dielectric layers of various thicknesses. The study presents a method of thermionic coating that combines in one technological cycle the processes of surface cleaning by a stream of low-energy ions and plasma electron beam evaporation of a substance under conditions of thermionic plasma formation.

Aim. The research aims to determine the possibility of using the presented methodology to create and process functional coatings without radiation damage to the substrate surface.

Suggested Citation:
Hrechko Ya, Sereda I, Babenko Ie, Azarenkov M. Thermionic coating method with preliminary bombardment of the substrate surface with a stream of low energy ions. *Sci Herald Uzhhorod Univ Ser Phys.* 2023;(53):9-18. doi: 10.54919/physics/53.2023.09.

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Methodology. The experimental studies were carried out in a plasma electron beam system with a primary plasma source based on an arc discharge with a filament cathode. The samples were diagnosed using a scanning electron microscope.

Results. The ion current density from the primary plasma was investigated to determine the efficiency of the surface cleaning mechanism by ion bombardment. The dependences of the ionic current density on the initial parameters of the experiment were determined. The results of the treatment of the (TiZr/TiSi) N sample surface with a plasma ion stream are presented. The obtained scanning electron microscope images showed that such treatment leads to the cleaning of the surface layer from various contaminants without damage and creates a substrate for further thermionic deposition of a coating with a high degree of adhesion. The mechanism of thermionic deposition of a titanium monolayer on a stainless-steel substrate previously cleaned by ion bombardment was studied. The revealed order of magnitude higher values of the ionic current to the substrate during the formation of thermionic plasma indicate a significant increase in the rate of the deposition process and contribute to the formation of a high-quality ion-plasma coating.

Conclusions. The scanning electron microscope images of the sample surface showed that the titanium coating during thermionic deposition was uniformly distributed over the substrate surface, without any droplet phase, which indicates the suitability of this technique for applying functional coatings without radiation damage to the substrate surface

Keywords: vacuum-plasma deposition methods; plasma electron beam evaporation; double electric layer; surface cleaning; functional coatings

Introduction

Among all the approaches currently available to solve problems of increasing the reliability and durability of machine and mechanism parts, cutting tools, and technological equipment, technological methods of surface hardening of structural materials play a key role [1-3]. According to the results of studies [4-6], the use of vacuum-plasma coating methods on structural materials makes it possible to obtain a wide range of functional coatings with predetermined properties. C. Cabral *et al.* noted that it is also important to use these methods in modern micro- and nanoelectronics to apply conductive and dielectric layers of different thicknesses [7].

Vacuum-plasma deposition methods are directly related to PVD methods [6; 8], which have many advantages [9]: coatings have improved physical and technological properties (high hardness, corrosion resistance, high temperature and impact strength, durability); the ability to use almost any type of inorganic materials for coating various groups of substrates and surfaces; low deposition temperatures (usually below 600°C); environmental friendliness. The disadvantages of these methods are the use of sophisticated equipment at a high cost; the velocity of coating is not high enough; the application technique is limited to substrates of complex geometry.

The electron beam method occupies a special place among the existing vacuum-plasma deposition methods. The studies [10; 11] noted that the essence of the method is that the kinetic energy of the electron beam used to evaporate the substance is converted into thermal energy in the treatment zone. In the classical method, the electron beam is formed by a remote electron gun, and the acceleration and focusing of the beam are achieved by electromagnetic lenses. When the electron beam is focused, this method allows for a high (up to 10^8 W/cm^2) power concentration on the target and high temperature, while ensuring a high rate of evaporation of even the most refractory materials. However, its main disadvantage is the danger of electrical breakdown arising from the transport of a powerful electron beam accelerated to 20 kV and the criticality of the accelerator system to pressure surges.

An alternative to the classical method is the plasma electron beam evaporation method proposed by the authors of [12]. In this method, the electron emitter is plasma, and the beam acceleration occurs in the near-surface layer of the space charge of the evaporated target. In this case, the heating power is controlled by both the relatively low voltage applied to the target (up to 1 kV) and the electron beam current, which depends on the plasma density. Moreover, the sample is heated uniformly from all sides, and the operational risks of electrical breakdown are not critical to pressure surges during evaporation.

An important factor in the coating process is also surface preparation, which includes, in addition to mechanical cleaning, the bombardment of the substrate surface with an intense stream of low-energy ions [13]. This method helps to heat the surface to the required temperature, removes not only all surface impurities but also ensures cleaning of the upper layer of the crystal lattice to a depth of approximately several nanometres without changing its structure. According to the results of V.A. Belous *et al.*, it can be

argued that both of these effects provide a high degree of adhesion of coatings to the substrate surface and a reduction in the internal stress of the coating [14].

The research aims to investigate the possibility of using the presented methodology to create and process functional coatings without radiation damage to the substrate and improve the technological characteristics of materials. To achieve this goal, the following tasks had to be solved: to investigate the ion current density of the primary plasma at the floating potential of the object to be treated and the application of a negative bias potential to it; to study the surface of the sample bombarded with a stream of low-energy ions from the plasma; to investigate the mechanism of thermionic deposition of a monolayer on a substrate previously cleaned by ion bombardment.

Materials and Methods

Experimental studies on thermionic coating were carried out in a plasma electron beam system with a primary plasma source based on an arc discharge with a filament cathode. Figure 1 shows the general scheme of the experiment and the qualitative distribution of the potential in the system.

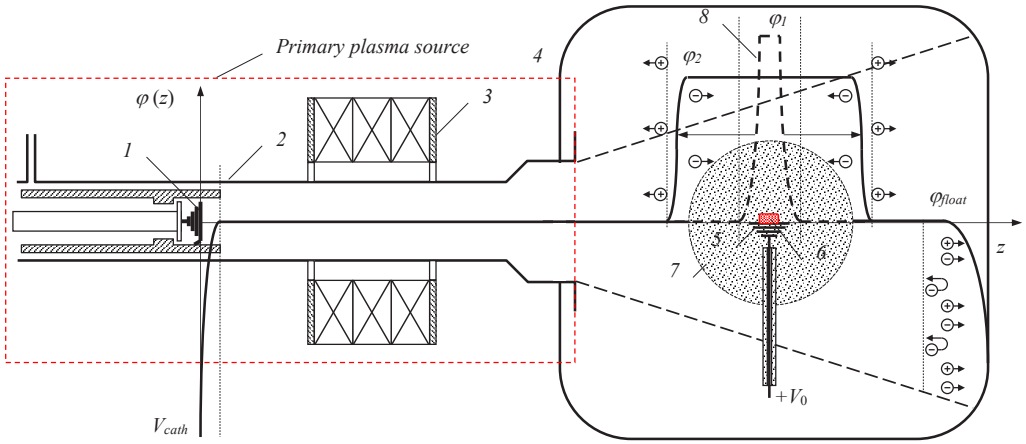


Figure 1. Scheme of a plasma electron beam system with a primary plasma source based on an arc discharge with a thermocathode and qualitative distribution of potential in the system

Note: 1 – thermocathode of the plasma source; 2 – discharge tube of the plasma source; 3 – magnetic field coils; 4 – vacuum chamber; 5 – crucible; 6 – evaporating substance; 7 – thermionic plasma; 8 – distribution of potential at the crucible, V_{cath} – potential drop near the thermocathode; V_0 – the potential supplied to the crucible; ϕ_1 – potential distribution in the bulk charge layer near the crucible; ϕ_2 – potential distribution in a double layer of space charge near the crucible; ϕ_{float} – floating potential

Source: compiled by the authors

The primary plasma source had the following design. A 2 cm diameter wire spiral tungsten direct filament cathode 1 (thermocathode) was attached to the end of a stainless-steel discharge tube 2 with an inner diameter of 4 cm and a length of 27 cm employing a vacuum electrical input. To prevent the vacuum electric input and the discharge tube from heating up under the influence of the discharge, these elements were cooled by running water passing through an outer casing. At the opposite end, the discharge tube was connected to vacuum chamber 4 employing a flange.

The discharge tube acted as an anode and was grounded, and the thermocathode was supplied with a negative potential of V_{cath}=0-400 V. The axial configuration of the anode was used to allow the charged particles to freely escape from the ionisation zone. To prevent plasma electrons from directly hitting the wall of the discharge tube, the discharge gap was in a longitudinal magnetic field. The configuration of the magnetic field was chosen to form a plasma cord that would not touch the wall of the discharge tube, and the field strength would be sufficient to maintain the plasma. As a result of the calculations, the magnetic field generated by the magnetic coil system 3 had a bell-shaped configuration with a maximum intensity of up to 600 E.

To avoid a powerful parasitic discharge between the thermocathode and the wall of the discharge chamber (under conditions of crossed electric and magnetic fields), the thermocathode was covered from the side with a copper guard, which protruded 1 cm beyond the end of the cathode. The guard spigot was connected to the external terminal of the vacuum electrical input, which provided thermal and electrical contact.

Argon was used as an operating gas. Since the operating pressure of the arc discharge with a thermocathode is more than 10^{-3} mmHg, it was necessary to inject gas into the thermocathode region to reduce the operating pressure in the vacuum chamber to 10^{-4} mmHg. The pressure gradient was formed due to the vacuum resistance of the discharge tube so that the pressure in the vacuum chamber was an order of magnitude lower than in the thermocathode area. At

a pressure of 3×10^{-4} mmHg in the vacuum chamber, the plasma source formed a divergent primary plasma flux with a density near the outlet of $\sim10^{11}$ cm^{-3} at an electron temperature of $T_e\approx2$ eV.

A wire tungsten crucible 5 in the form of a spiral basket with a diameter of 1 cm, with a substance to be evaporated, 6 were located in the middle of the vacuum chamber on the same axis with the plasma source at a distance of ~20 cm from its outlet. The crucible was attached to a high-voltage vacuum input cooled by water. To reduce the surface that collects current from the plasma, the crucible leg and the entire surface of the vacuum input were covered with ceramic insulators.

The mode of electron beam evaporation with a plasma emitter of electrons was realised after applying a positive potential V_0=0-1000 V to the crucible relative to the grounded chamber. A layer of space charge with a potential drop φ_1 was formed near the crucible, in which the electrons of the primary plasma were accelerated, and an electron flux was formed, which heated the crucible. As the crucible heated up, a stream of neutral atoms of the evaporating substance was formed, some of which were ionised by electron impact. The generated ions were accelerated by the electric field of the layer towards the electrons. Under conditions when the rate of ion generation near the crucible surface reached such a value that the number of born particles exceeded the number of particles passing through the layer, a dense (up to 10^{14} cm^{-3}) thermionic plasma was formed between the layer of space charge and the crucible surface [15]. The initial layer of negative space charge φ_1 was transformed into a double electric layer φ_2 at the front of the thermionic plasma. The double layer is an effective mechanism for accelerating charged particles because it concentrates all the voltage applied to the crucible [16]. Due to the gas kinetic pressure, the thermionic plasma expanded, causing the double layer to be squeezed away from the crucible surface, increasing its surface and, accordingly, increasing the current through the layer. This expansion occurred until the conditions for the existence of the double layer were violated [17; 18]. With the disappearance of the layer, the electron beam disappeared, and the crucible heating and the formation of thermionic plasma stopped. After the collapse of the thermionic plasma, a layer of space charge was formed near the crucible again with a drop in the potential φ_1 and the whole process was repeated.

The samples to be coated were placed on a sample holder, which was a circular copper table with a diameter of 10 cm. The sample holder was located at a distance of 10 cm from the surface of the crucible. The holder was either electrically isolated from the conductive elements of the chamber and received a floating potential φ_{float}, or a negative bias potential φ_{bias}=0-1000 V was applied to it from a high-voltage power supply. A Linnik microinterferometer was used to determine the thickness of the obtained coatings. The surface microstructure of the samples was studied using a Tescan Vega 3 LMH scanning electron microscope.

Results and Discussion

The study of the mechanism of near-surface formation of an intense low-energy ion flux for preliminary surface cleaning before coating requires the study of the ion current density from the plasma created by a source based on an arc discharge with a filament cathode. The paper [19] presents the main results of this study and describes in detail the process of ion bombardment of the surface for its cleaning. Figure 2 shows the dependence of the ionic current density j_+ supplied to the sample holder on the negative bias potential of the holder φ_{bias} for different values of the discharge current of the primary plasma source i_d. The dependence was obtained at a vacuum chamber operating pressure of $p_c\sim4\times10^{-4}$ mmHg. The ion current densities at the floating potential of the holder φ_{float} ~150 V are marked with separate points on the graph. The Figure 2 shows that the application of an additional negative bias potential to the holder can significantly increase the ion current density of the primary plasma compared to the ion current density at the floating holder potential. The dependence demonstrates a linear increase in the ion current density with an increase in the negative bias potential. The ion current density also increases with an increase in the discharge current of the primary plasma source, which indicates that the current balance in the system is maintained. However, this increase is not significant at the floating potential of the holder.

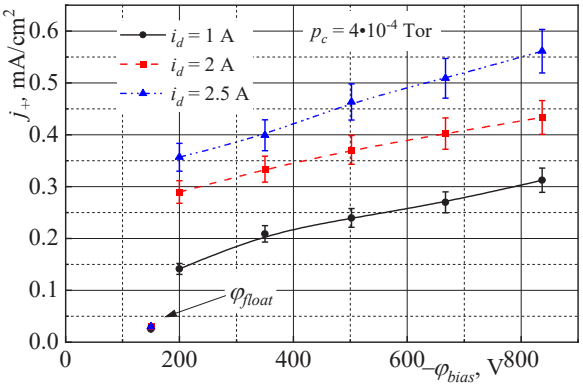


Figure 2. Dependence of the ion current density j_+ supplied to the sample holder on the negative bias potential of the holder φ_{bias} for different values of the discharge current of the primary plasma source i_d

Note: j_+ – ionic current density supplied to the sample holder; φ_{bias} – negative bias potential applied to the sample holder; i_d – discharge current of the primary plasma source; p_c – operating pressure in the vacuum chamber; φ_{float} – floating potential

Source: [19]

The operating pressure in the vacuum chamber is an important parameter in vacuum plasma coating, therefore, the effect of changing the operating pressure on the ion current density from the primary plasma was additionally studied. Figure 3 shows the dependence of the ion current density j_+ supplied to the sample holder on the negative bias potential of the holder φ_{bias} for different values of the vacuum chamber operating pressure p_c. Figure 3a corresponds to the case when the sample holder was centrally located on the same axis as the plasma source, Figure 3b – the sample holder was located at a distance of ~10 cm from the centre. It can be seen from the figures that a decrease in the working pressure in the vacuum chamber leads to a decrease in the ion current density going to the holder. The ion current density also decreases significantly with distance from the discharge axis of the plasma source. For example, when the holder is moved by 10 cm, the ion current density flowing to the holder decreases by an average factor of 4 at an operating pressure of p_c~4×10^{-4} mmHg. This indicates that when cleaning the surface of the substrate with an ion flux from the primary plasma, the sample should be located as close as possible to the discharge axis of the plasma source to obtain the highest ion current density on its surface.

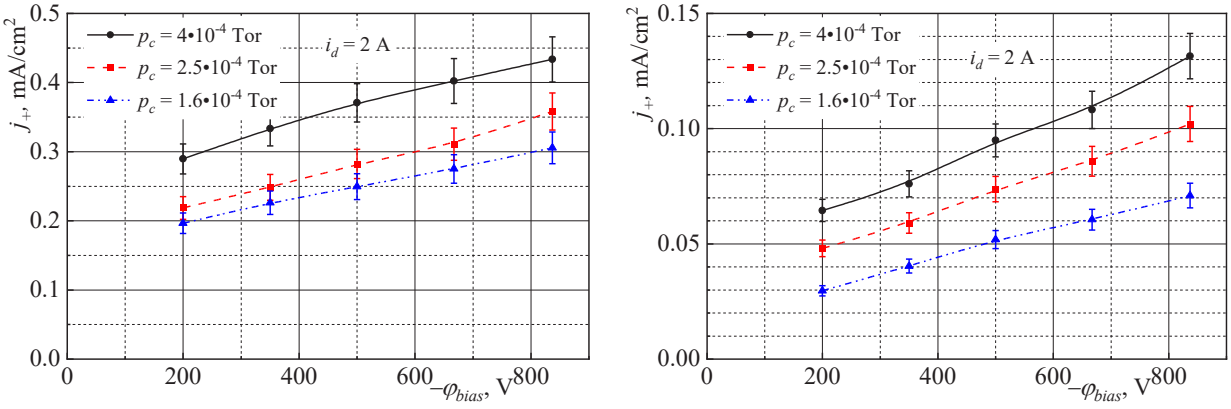


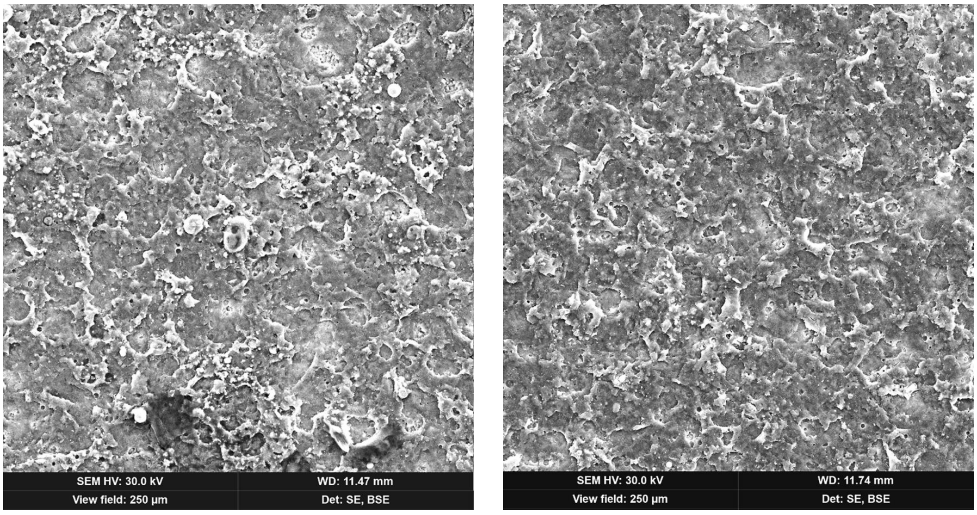
Figure 3. Dependence of the ion current density j_+ supplied to the sample holder on the negative bias potential of the holder φ_{bias} for different values of the operating pressure in the vacuum chamber p_c

Note: a – The sample holder, located centrally on the same axis as the plasma source; b – The sample holder, placed at a distance of ~10 cm from the centre; j_+ – ionic current density supplied to the sample holder; φ_{bias} – negative bias potential applied to the sample holder; p_c – operating pressure in the vacuum chamber; i_d – discharge current of the primary plasma source

Source: compiled by the authors

In the system under consideration, to implement a technique that combines the ionic cleaning process and the subsequent thermionic coating in one technological cycle, the crucible with the substance must be located on the same axis as the plasma source so that the power of the electron beam is sufficient to create a thermionic plasma. It should also be noted that during thermionic deposition, the substrate should be at a certain optimal distance from the crucible surface so that the substrate surface does not overheat, and the resulting coating is uniformly distributed over the substrate surface with a given thickness. One of the technical solutions to effectively combine the process of substrate cleaning and subsequent coating is to use a movable holder, which, when cleaning the surface, takes a position closer to the axis of the plasma source, and when applying the coating, returns to the determined optimal distance.

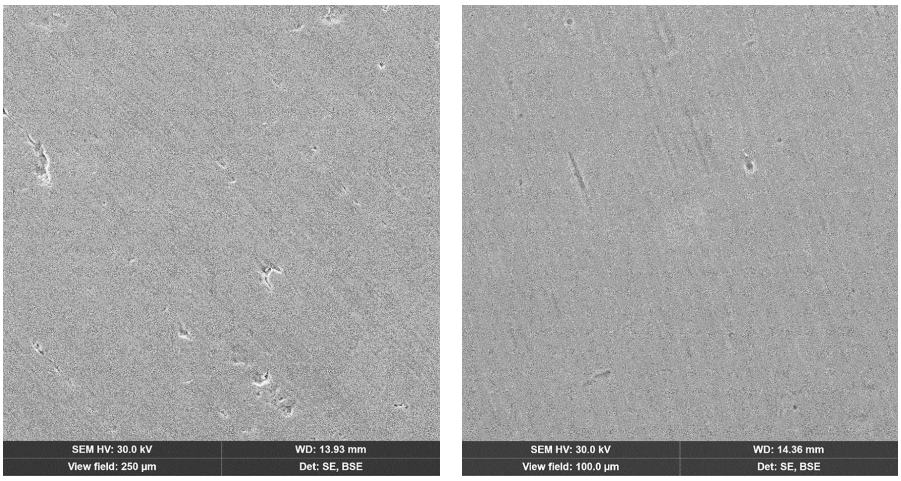
Figure 4 shows scanning electron microscope (SEM) images of the surface of the (TiZr/TiSi) N sample before (*a*) and after (*b*) surface treatment with a stream of ions from the primary plasma. For more efficient surface treatment, the sample was placed on a holder located on the same axis as the plasma source. A negative bias potential of φ_{bias}=800 V was applied to the holder. The processing time was ~5 minutes. The images obtained show that the surface treatment with a stream of ions from the primary plasma leads to the cleaning of the surface layer of the sample from various impurities and contaminants without damaging its structure and can be used for further coating of the sample.



a) b)

Figure 4. SEM image of the (TiZr/TiSi) N sample surface
Note a – before surface cleaning; b – after surface cleaning by ion bombardment
Source: [19]

The next step was to study the mechanism of thermionic deposition of the monolayer on a substrate previously cleaned by ion bombardment in one technological cycle. Titanium was chosen as the evaporated material, which was deposited on a stainless-steel substrate. For this purpose, the sample was placed on a holder at a distance of 10 cm from the discharge axis of the plasma source. Pretreatment was performed by applying a negative bias potential of φ_{bias}=800 V to the holder. The processing time was ~5 minutes. After cleaning by ion bombardment, the negative bias potential was stopped and the process of thermionic deposition of the titanium monolayer began. The volume of titanium evaporated was 10 mm^3. Figure 5 shows SEM images of the surface of the stainless-steel substrate pretreated by ion bombardment (Fig. 5a) and the titanium monolayer deposited by the thermionic method (Fig. 5b). The thickness of the titanium monolayer is ~270 nm, which was determined by the interferometric method. From the images obtained, it can be seen that the titanium coating during thermionic deposition is evenly distributed over the surface of the substrate, without any droplet phase, which indicates the possibility of using this technique for the application of functional coatings with predetermined properties.



a b

Figure 5. SEM image of the surface of stainless-steel substrate and Ti monolayer deposited by a thermionic method
Note: a – surface of the substrate, pre-cleaned by ion bombardment; b – Ti monolayer deposited by the thermionic method
Source: compiled by the authors

The studies revealed an order of magnitude higher ionic current to the substrate during the formation of thermionic plasma, which indicates a significant increase in the rate of the deposition process. Figure 6 shows an oscillogram of the ionic current flowing to the sample holder. The red colour of the curve corresponds to the ionic current without thermionic plasma generation, and the black colour corresponds to the ionic current under thermionic plasma generation. The oscillogram was obtained at the discharge current of the primary plasma source i_d=2 A, the working pressure in the vacuum chamber p_c~2.5×10^{-4} mmHg. Figure 6 shows that the level of ion current flowing to the holder in the case of thermionic plasma generation is an order of magnitude higher than the ion current without thermionic plasma generation. This is since in the case of thermionic plasma generation, a double layer of space charge is formed in a self-consistent manner at the edge of the primary and thermionic plasma with different concentrations and provides an effective transfer of energy from the external electric field to the kinetic energy of the bipolar motion of charged particles [18]. The created ions are accelerated in the electric field of the double layer from the crucible surface to the vacuum chamber wall, creating an additional ion beam to the substrate surface.

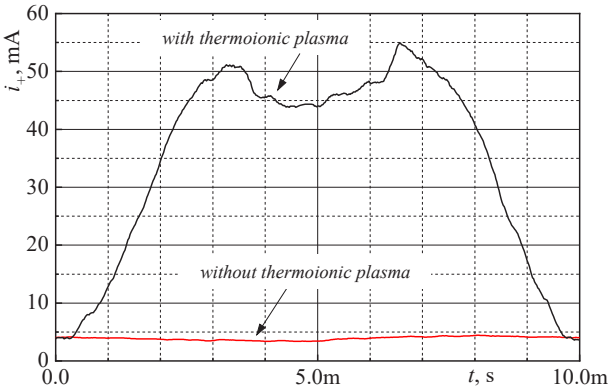


Figure 6. Oscillogram of the ionic current i_+ flowing to the sample holder at the moment of thermionic plasma generation
Source: compiled by the authors

In addition, the presence of an additional ion beam on the substrate surface confirms previous studies in this direction [12], namely, the presence of an additional mechanism for generating ions throughout the chamber volume under the conditions of development of a non-self-sustained glow discharge. The fact is that during the generation of thermionic plasma, the current to the crucible with the evaporating substance can be several times (3-6 times) higher than the discharge current of the primary plasma source. When the current to the crucible is less than the discharge current of the primary plasma source, ions are generated by the primary plasma source. This case corresponds to the red curve in Figure 6. When the current to the crucible is comparable to the discharge current of the primary plasma source, ions are generated near the crucible (formation of thermionic plasma). With a further increase in the current to the crucible, a third mechanism appears – the generation of ions in the entire volume of the chamber under the conditions of the development of a non-self-sustaining glow discharge (black curve in Fig. 6). The cathode is the wall of the vacuum chamber with a cathode potential drop sufficient to increase the generation of ions, and the anode is the crucible with the evaporating substance.

The appearance of an additional ion beam on the surface of the substrate during thermal ion deposition plays a positive role. First, as noted above, it contributes to an increase in the rate of the deposition process. Secondly, the authors of [20; 21] argue that additional ion bombardment during the coating process significantly contributes to the formation of a high-quality ion-plasma coating and significantly affects the physical and chemical properties of the sample surface. This is determined by the bombardment of the target with an additional flux of ions leads to the appearance of point defects, which subsequently become active adsorption centres [20]. The studies reported in [22; 23] were carried out in a vacuum arc evaporation system, one of the advantages of which is the high degree of plasma ionisation and the possibility of the ionised plasma affecting the surface of the samples. This ensures that the processes of cleaning, heating and formation of a metal sublayer to ensure adhesion before the subsequent plasma deposition of the coating are carried out in one technological cycle. However, the main disadvantage of this method is the presence of a droplet phase during surface treatment and coating, which is visible in the SEM images of sample surfaces presented in [22; 23]. By analogy with the vacuum-arc method, the thermionic method presented in this paper provides the creation of a dense plasma with a high degree of ionisation and the generation of an additional ion flux to the surface of the substrate. However, unlike the vacuum-arc method, the thermionic deposition does not have a droplet phase in the coatings, since this approach ensures uniform heating of the sample by electrons of the primary plasma accelerated in the near-surface layer of space charge. The obtained SEM images of the sample surface confirm this well.

Conclusions

This study presents a thermionic coating technique that combines the processes of substrate surface cleaning with a stream of low-energy ions and plasma electron beam evaporation in one technological cycle.

A characteristic feature of this technique is that the formation and acceleration of charged particle flows occur in both cases in the space charge layer.

The dependences of the ion current density from the plasma at the floating potential of the holder and the application of a negative bias potential to it are determined. It is determined that the application of an additional negative bias potential to the sample holder can significantly increase the ion current density of the primary plasma compared to the ion current density at the floating potential of the holder. It is shown that the ion current density increases linearly with the negative bias potential and also increases with the discharge current of the primary plasma source. The obtained microscopic images of the sample surface after ion bombardment demonstrate that this treatment leads to the cleaning of the surface layer of the sample from various impurities and contaminants without damaging its structure, and can be used for further coating of the sample.

The mechanism of thermionic deposition of a monolayer on a substrate previously cleaned by ion bombardment in one technological cycle has been studied. It was found that when forming a thermionic plasma, the value of the ionic current on the substrate is significantly (by an order of magnitude) higher than without a thermionic plasma, which indicates a significant increase in the rate of the deposition process. The reason for this is the formation of a double electric layer of space charge at the front of the thermionic plasma, and the emergence of an additional mechanism for generating ions throughout the chamber under the conditions of the development of a non-self-sustaining glow discharge, which was found in previous studies. The microscopic images of the sample surface showed that the titanium coating during thermionic deposition was uniformly distributed over the substrate surface, without any droplet phase. This indicates the suitability of this technique for applying functional coatings without radiation damage to the substrate surface, which will further improve the technological characteristics of structural materials.

Further research in this area involves determining the possibility of deposition of multicomponent coatings, including those with a multilayer structure, by the thermionic method on a substrate surface previously cleaned by ion bombardment. In addition, it is planned to carry out comprehensive diagnostics of the obtained coatings, namely, the study of the surface structure and cross-section, elemental composition, structural and phase state, mechanical properties and tribotechnical characteristics of the coatings.

Conflict of Interest

The authors declare no conflict of interest.

Acknowledgements

The study was carried out within the framework of the grant support project of the National Research Foundation of Ukraine “Modification of the surface of a solid under the influence of plasma and charged particle beams” (project registration number 2020.02/0234).

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Методика термоіонного нанесення покриттів з попереднім бомбардуванням поверхні підкладки потоком низькоенергетичних іонів

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Анотація

Актуальність. Вакуумно-плазмові методи нанесення функціональних покриттів широко використовуються для підвищення надійності та довговічності деталей машин і механізмів, різального інструменту, технологічного оснащення, а також в сучасній мікро- та наноелектрониці для нанесення струмопровідних та діелектричних шарів різної товщини. В роботі представлена методика термоіонного нанесення покриттів, яка поєднує в одному технологічному циклі процеси очищення поверхні потоком низькоенергетичних іонів та плазмового електронно-променевого випаровування речовини в умовах формування термоіонної плазми.

Мета. Метою дослідження було визначення можливості використання представленої методики для створення та обробки функціональних покриттів без радіаційних пошкоджень поверхні підкладки.

Методологія. Експериментальні дослідження проводилися у плазмовій електронно-променевій системі з джерелом первинної плазми на основі дугового розряду з катодом розжарення. Діагностика зразків проводилась за допомогою скануючого електронного мікроскопа.

Результати. Була досліджена густина іонного струму з первинної плазми з метою визначення ефективності механізму очищення поверхні іонним бомбардуванням. Визначені залежності густини іонного струму від початкових параметрів експерименту. Наведені результати обробки поверхні зразка (TiZr/TiSi) N потоком іонів із плазми. Отримані зображення зі скануючого електронного мікроскопа показали, що така обробка призводить до очищення поверхневого шару від різних забруднень без пошкоджень і створює підкладку для подальшого термоіонного осадження покриття з високим ступенем адгезії. Проведені дослідження механізму термоіонного осадження моношару титану на попередньо очищену іонним бомбардуванням підкладку з нержавіючої сталі. Виявлені на порядок вищі значення іонного струму на підкладку при формування термоіонної плазми свідчать про суттєве підвищення швидкості процесу нанесення та сприяють формуванню якісного іонно-плазмового покриття.

Висновки. Отримані зображення поверхні зразка зі скануючого електронного мікроскопа показали, що покриття титану при термоіонному нанесенні рівномірно розподілене по поверхні підкладки, без наявної крапельної фази, що свідчить про придатність даної методики для нанесення функціональних покриттів без радіаційних пошкоджень поверхні підкладки

Ключові слова: вакуумно-плазмові методи нанесення; плазмове електронно-променеве випаровування; подвійний електричний шар; очищення поверхні; функціональні покриття