SECTION 3
PHYSICS OF RADIATION AND ION-PLASMA TECHNOLOGIES

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USE OF HIGH-VOLTAGE PULSE SUBSTRATE BIAS POTENTIAL IN THE DEPOSITION OF VACUUM-ARC COATINGS (METHOD PIIID)

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The physical foundations of the use of pulsed substrate bias potential (PIIID method) in the processes of coatings deposition from filtered vacuum-arc plasma are considered. The possibilities of controlling the coatings properties when using the PIIID method are discussed. Information is provided on the characteristics of the materials of coatings depending on the parameters of the pulsed potential. The requirements for equipment used in PIIID deposition of vacuum-arc coatings are discussed. A brief review is presented of experimental and theoretical works performed at the NSC KIPT on deposition hard carbon and nitride coatings by PIIID method from filtered vacuum-arc plasma.

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INTRODUCTION

The method of plasma coating deposition under the condition of applying a high-voltage pulsed bias potential to the substrate has been developed since the 1980s [1–4]. By that time, sufficient information had already been accumulated regarding the influence of ion bombardment during deposition on adhesion, microstructure, morphology, internal stresses and film uniformity [5]. A feature of the method is the pulsed treatment of the surface of the coating being deposited with a flow of accelerated ions from the plasma, the energy of which is determined by the potential difference between the plasma and the surface.

In the first publications on the topic, specialized ion sources were used to modify the properties of coatings and the surface of materials. Later, it was proposed to implant ions from plasma, for which a high negative potential was applied to the substrate to accelerate the ions. This approach provides an additional element of process control in plasma technologies PVD and PECVD deposition.

It should be noted that in high-energy, continuous ion flux surface treatment processes, the coating temperature increases rapidly over time and may exceed acceptable limits. To overcome this limitation, it has been proposed to apply a combined substrate bias potential during coating deposition, which consists of a base DC potential and superimposed pulses of higher potential [4]. The amplitude of the potential pulses is at a level that is usually used for etching or sputtering, and the base potential is at the same level as in the usual sputtering process. During the pulse (potential amplitude from 500 V to several kilovolts), positively charged ions of both metal and gas (reactive or inert) are accelerated to the surface, which during this time affect the adhesion, microstructure, morphology, internal stresses and uniformity of the film. In the intervals between pulses, conventional PVD deposition processes take place (base potential from -50 to -300 V). With a rather low (usually 5…10%) relative duration of the high potential (duty cycle value), the deposition temperature does not increase much. As a result, conditions arise for the formation of metastable phases in the coating, and importantly, this process can be controlled by adjusting the pulse repetition frequency or their duration, i.e. by changing the pulse mode parameters.

Currently, this method of plasma immersion ion implantation and deposition (PIIID) widely used to create coatings with improved properties, as evidenced by the large number of published works on this topic. Among them, detailed reviews should be highlighted [6–11].

It should be noted that plasma ion implantation and deposition related to energetic deposition methods known by various names, abbreviations, and trademarks, including the following: plasma source ion implantation (PSII), plasma immersion ion implantation (PIII or PI3), plasma ion implantation (PII or PI2), plasma ion plating (PIP), plasma immersion ion implantation and deposition (PIIID or PIII&D), metal plasma immersion ion implantation and deposition (MePIIID), plasma doping (PLAD), and plasma ion immersion processing (PIIP) [7, 8, 11]. The common features of these methods are the presence of a plasma in which the substrate is immersed and the application of a high negative bias potential to it, usually in a pulsed mode.

1. PHYSICAL BASICS OF USING PULSED SUBSTRATE BIAS POTENTIAL (PIIID METHOD)

The process of coating deposition by the PIIID method should be considered from the point of view of the physics of plasma-solid interaction. The properties of the plasma (source of the substance to be deposited as a coating) are of the same importance as the characteristics of the processes in the solid that occur when plasma particles hit it. The surface of the sample in the PIIID method is either flowed by a plasma stream or placed in a region of space occupied by the plasma, and a relatively high potential of negative polarity is applied to it, as a result of which a layer of uncompensated plasma is formed near the surface, i.e. enriched in ions that are accelerated to the substrate. The size (thickness) of the uncompensated layer and the number of ions accumulated in it depend on the plasma density.

Pulsed potential supply allows:

• To avoid arcing on the substrate;

• To limit the size of the layer of uncompensated plasma when working with high voltage (above 10 kV) and in low-density plasma;

• To regulate the quantity of ions inside the uncompensated plasma layer which would use for the surface treatment;

• To use additional parameters (pulse amplitude and relative duration of the high potential action) which influence the coating property and avoid overheating.

The energy Ei of the accelerated ion that has reached the surface determines the nature and result of its interaction with the solid [12]. If Ei < Ed (displacement energy of surface atoms), the ion remains on the surface and, due to the super-thermal energy, has increased mobility along the surface, where the processes of crystallite nucleation are stimulated. The subsequent growth and merging of crystallites into a continuous coating is accompanied by the appearance of a large number of pores and residual mechanical tensile stresses. The result state of the coating can be presented in the form of a structural zone diagram [13].

When Ei > Ed, there are opportunities for implementing other process variants. First, the ion can displace a surface atom near the point of impact, thereby increasing the mobility of surface atoms, causing changes in the crystal structure (or type of bonds in the case of an amorphous structure). Second, the ion enters the depths of the solid, under the surface (the so-called subplantation occurs). The ion brings with it energy (kinetic) and excess atomic volume into the solid. At moderate energy, the ion motion stops between the atoms of the solid, the kinetic energy is spent on creating a cascade of displacements in the atomic lattice. In this case, mechanical compressive stresses are generated in the coating due to the brought volume [14]. With a higher initial ion energy, due to the energy released in the volume of the displacement cascade, conditions arise for the occurrence of a thermal peak within it, which leads to stress relaxation due to the displacement of excess atomic volume to the surface.

Physical processes in vacuum-arc PIIID deposition occur differently during the pulse durations and in the intervals between them. Depending on the energy of the ions, two types of condensation are realized: the surface plating or sub-surface one. During the action of a high-voltage pulse, most of the accelerated film-forming ions or atoms, the energy of which exceeds the displacement energy Ed, penetrate below the surface region, i.e., sub-plantation occurs. Therefore, during these time intervals, film growth may occur below the surface rather than on the surface. As a result of such process, the dense and hard films are formed, the structure and level of internal compressive stresses of which depend on the energy of the deposited particles. [15]. The maximum stress level for various coating materials occurs at energies from tens of electron volts to several hundred electron volts [14]. At the initial stage of deposition, the effect of mixing the atoms of the film and the substrate occurs, forming strong bonds, which promotes good adhesion. However, if the internal stresses are excessive, this can lead to delamination of the film.

The deposition mode in the time intervals between pulses is determined by the current potential of the substrate, the value of which can be set from an additional DC power source, or you can use the so-called "floating" potential, which depends on the parameters of the plasma surrounding the substrate surface. Accordingly, if the potential is several electron volts, superficial, layer-by-layer film growth occurs. At potential value of tens of volts and higher, sub-plantation occurs, which leads to the formation of dense and hard films with an increased level of internal compressive stresses [16].

The range of ion energies employed in the PIIID compared to other energetic deposition methods is illustrated in Fig. 1.

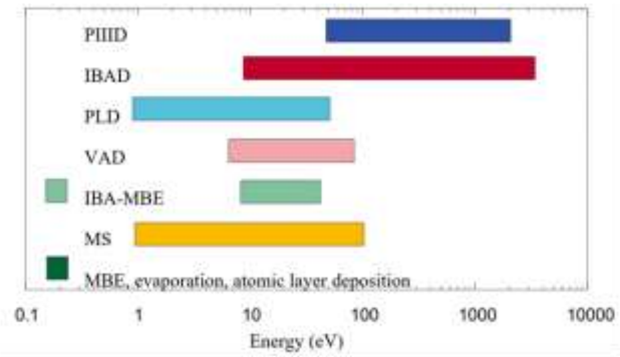

Fig. 1. Typical energy ranges for different PVD processes. PIIID – plasma immersion ion implantation and deposition; IBAD – ion beam assisted deposition; PLD – pulsed laser deposition; VAD – vacuum arc deposition; IBA–MBE – ion beam assisted molecular beam epitaxy; MS – magnetron sputtering; MBE – molecular beam epitaxy [9]

The supply modes and parameters of the high-voltage potential differ somewhat for different methods of creating the plasma from which deposition is carried out (PECVD, FVAD, MS, HIPIMS, PLD). However, in each of them, when implementing PIIID, pulses of negative bias potential are supplied to the substrate, usually with a duration of several microseconds and an amplitude of several hundred to thousands of volts. Unlike high DC bias potential, pulsed supply allows to minimize the effects associated with sputtering of the coating surface and to form metastable structures with reduced stress levels at low substrate temperatures. In this case, the amplitude of the potential pulses must be of sufficient magnitude to accelerate the ions being deposited to an energy that ensures their penetration deep into the substrate [6–8].

It should be taken into account that the potential of the substrate surface may differ from the potential of the substrate holder to which the pulses are applied, as a result of which the energy of the ions accelerated to the surface turns out to be less than that given by the pulse amplitude. The efficiency of applying potential to the surface depends significantly on the electrical conductivity of the substrate material and (in the case of low electrical conductivity) its thickness. The efficiency is best on metals, very low on dielectrics (Fig. 2 [17]). This feature also concerns the coatings made of materials with low electrical conductivity deposited on metal substrates: the thicker the coating becomes during the deposition process, the lower the potential appears on its surface during the action of pulses.

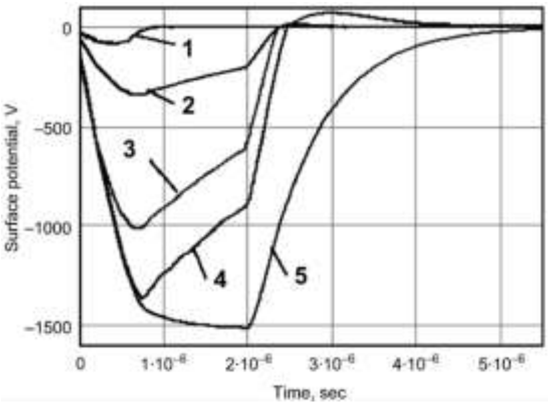

Fig. 2. Shape of the potential pulse on the surface of samples made of different materials (at plasma density): 1 – glass (n = $2\cdot10^{16}$ m^{-3}); 2 – glass (n = $2\cdot10^{15}$ m^{-3}); 3 – glass ceramics (n = $2\cdot10^{16}$ m^{-3}); 4 – glass ceramics (n = $2\cdot10^{15}$ m^{-3}); 5 – metal, initial pulse shape [17]

2. THE CAPABILITIES OF THE PIIID METHOD FOR CONTROLLING COATING PROPERTIES

Among various plasma-based deposition methods, PIIID is a versatile technique that allows multiple operations, such as sequential implantation, coating deposition, and surface etching, to be performed in a single process by combining the advantages of conventional plasma and ion beam technologies [18]. However, the diversity of chemical compounds properties, for example, in nitrides and carbides of some transition metals, does not allow to copy the PIIID regime developed for deposition the coating of specify system in case of another one directly [9]. The PIIID method is most suitable for use in cases of synthesis of materials sensitive to overheating during deposition, such as diamond-like carbon.

The use of PIIID provides the ability to control and manage deposition processes that affect the properties of coatings of various compositions. Among them, the following should be noted:

– Preliminary surface cleaning in a vacuum using a pulsed high-voltage glow discharge in an inert gas (ion bombardment). To accelerate ions, a negative potential is applied to the substrate, i.e. the surface becomes a cathode. Cathodic spots appear on surface defects, especially on contaminated areas, as a result of which the glow discharge changes to a high-current vacuum-arc discharge, the prolonged occurrence of which in the case of high-voltage DC bias leads to surface erosion. In the PIIID method, after the short (microsecond duration) pulses, the arc discharge is interrupted at the initial stage. In such a way, the substrate surface and the samples mounted on it becomes protected from erosion by cathode spots.

– Creating a transitional mixed layer before applying coatings to improve adhesion [19]. An increased level of adhesion to the substrate is achieved when the first (transitional) coating layer is deposited at an increased amplitude of the bias potential pulses. At energies of several keV, subsurface ion implantation occurs with the formation of a thin layer of mixed elemental composition, which provides a smooth transition (adapting) of the elastic properties of the coating and substrate materials [20] unlike a sharp boundary between them in case of absence the transition layer.

– Controlling crystal structure and texture during the deposition of hard ceramic coatings. Such coatings on engineering materials are of great interest due to their ability to ensure wear resistance and long-term performance of products under conditions of increased loads and high temperatures. One of their applications is protective coatings on cutting tools. Among these, nitride coatings are widely used, which are deposited using the PIIID method. Thus, in [21] this method was used to improve the quality of Ti-Al-N coatings deposited from unfiltered vacuum arc plasma. Superhard nanocomposite TiAlSiN coatings with a thickness of 2 μm were deposited on WC-Co (8 wt.%) carbide substrates by the PIIID method from a vacuum arc source plasma with TiAlSi alloy cathodes [22]. Incomplete filtration was achieved by using a plasmaguide in form of the simple bent tube. Ti–Cu–N nanocomposite hard coatings with a thickness of 1.9…2.3 μm were deposited on M2 high-speed steel (HSS) substrates by the vacuum-arc method from unfiltered plasma using a composite TiCu (88:12 at.%) cathode and nitrogen working gas [23].

– Regulation of the level of residual mechanical stresses in the coating. With an amplitude of bias potential pulses of several kV, the stress level is significantly reduced (Fig. 3) [8, 14]. The effect is influenced by the energy of the ions (which is determined by the amplitude of the pulses), the duration of the pulses and their repetition frequency, as well as the degree of ionization of the plasma from which the deposition is carried out. First, the ion energy must be at least 500 eV to create annealing conditions in thermal peaks (i.e., pointwise, in local volumes at the nanoscale level). Further, in order for the effect of annealing residual stresses in the coating as a whole to work, it is necessary to ensure a sufficient intensity of the ion flux with increased energy so that during the deposition of a certain coating layer, the total volume of thermal peaks constitutes a noticeable part of the volume of this layer. It should be noted that such a monotonically decreasing dependence, also valid for carbon coatings, can change to a more complex one in the case of multicomponent nitrides with two or more phases (for example, CrAlN [24], where the competing crystal structures are cubic and wurtzite).

– Control of the structure and properties of carbon coatings, diamond-like (DLC), as well as nitrogen-doped (CN) depending on the energy of the deposited carbon ions. DLC is characterized by an amorphous structure containing a high proportion (up to 90%) of carbon sp^3 bonds, as well as sp^2 bonds and may contain hydrogen. Diamond-like properties are most pronounced in the case of an increased content of sp^3 bonds [25]. In many studies of DLC, the dependence of hardness and internal stresses on the amplitude and duration of the bias potential pulses during deposition was found [26, 27]. This opens up the possibility of implementing a multilayer structure in which stressed hard and more plastic soft layers alternate when depositing DLC coatings using the PIIID method by periodically changing the amplitude of the bias potential [28]. This multilayer architecture allows for the production of a fairly hard DLC coating of micron thickness with a high level of adhesion, which is achieved by combining hard and soft layers. Electronic means make it possible to regulate the time parameters of the potential pulses in a very wide range from microseconds and above, which makes it possible to deposit individual coating layers with a thickness ranging from units to hundreds of nanometers, creating multilayer nanostructured coatings.

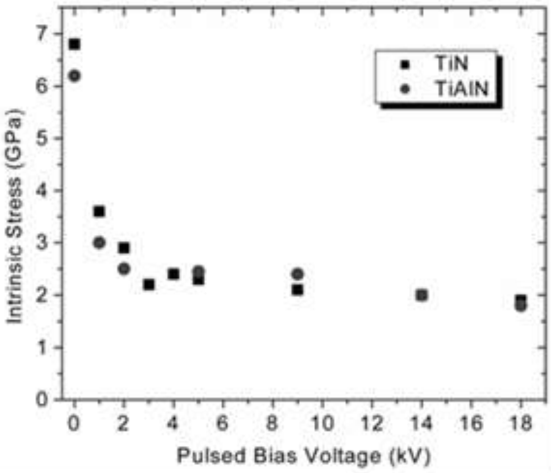

Fig. 3. Dependence of stresses on the amplitude of the substrate bias potential pulses in TiN and TiAlN coatings deposited in the PBIID process; pulse duration 20 μs, repetition rate 500 Hz (duty cycle 1%) [8]

– Formation of so-called gradient carbon coatings, with hardness close to diamond-like ta-C, but with a significantly lower stress level, with better adhesion to the substrate, by creating a gradient of mechanical properties across the thickness of the coating. This type of coating was implemented in [29], where deposition was carried out from filtered vacuum-arc plasma, gradually reducing the amplitude of the pulsed substrate bias potential from 3 kV to 0 V with a gradient of 5 V/s (pulse duration 25 μs, repetition rate 600 Hz). In such architecture, a soft layer, which ensured adhesion, was deposited near the substrate at high voltage amplitude, and the hardness of subsequent layers increased in accordance with the decrease in amplitude. The stress level in the coating was 1.5 GPa, which is 6 times less than in the coating deposited under conditions of applying a DC bias potential.

3. EQUIPMENT FOR DEPOSITION OF NANOSTRUCTURED FVAD + PIIID COATINGS

Supplying a high-voltage pulsed bias potential to the substrate is the most effective in case of deposition from vacuum-arc plasma, the degree of ionization of which is high. The FVAD method, which uses plasma filtration from particles of cathode material, so-called macroparticles, provides a significantly lower level of coating defects.

Plasma filtration is used in both experimental and industrial equipment. To produce high-quality coatings with a small number of defects, magneto-electric filters of various modifications are used, including curved ones with different plasma flow deflection geometries, as well as rectilinear ones. In some applications, such as cutting tool reinforcement, where perfect filtration is not required, simple blinds design is a viable option [30].

The filtered vacuum-arc plasma flow consists of almost 100% ions, which allows the energy of the deposited particles and the properties of the coating to be controlled synchronously with the duration of the pulses and the pauses between them. In the intervals between pulses, conventional PVD deposition process take place, at which the potential is either not applied to the substrate (“floating” mode), or set from a direct current source usually at a level from -50 to -300 V.

DC plasma sources are most often used in vacuum arc deposition equipment. However, in some cases, pulsed vacuum arc sources are used to create plasma. Such sources are installed not only in experimental but also in some industrial equipment. Deposition of alloyed and multicomponent coatings is carried out either from multiple sources or using a multicomponent cathode. Among the works on the industrial use of the FVAD PIIID method, it is worth mentioning the deposition on the OPDB-FVAD equipment (vacuum-arc source with filtration due to a double change in the direction of the carbon plasma flow) of DLC wear-resistant coatings with a total thickness of 1 μm, which consist of soft and hard layers of equal thickness within 12.5…75 nm each. A soft layer with 60% sp^3 fraction and hardness of 25 GPa was deposited under a pulsed bias potential of 3 kV, 600 Hz, 25 μs, and a hard layer with 80% sp^3 fraction and hardness of 52 GPa was deposited in the absence of pulses [10, 11].

The use of such methods allows the production of micron-thick diamond-like coatings with a high level of adhesion.

4. EXPERIMENTAL AND THEORETICAL STUDIES OF VACUUM-ARC PIIID COATINGS AT THE NATIONAL SCIENCE CENTER KIPT

Research on vacuum-arc PIIID coatings at the NSC KIPT has been conducted for a long time. During this time, high-voltage pulse equipment was developed and manufactured, which is installed on experimental installations equipped with filtered vacuum-arc plasma sources. Based on this equipment, the following processes have been developed, tested, and prepared for practical use:

– Surface preparation of samples/products in vacuum by ion bombardment in glow discharge plasma in argon while applying high voltage pulses of microseconds duration to the substrate holder.

– Creation of a transitional adhesive mixed layer, which is formed on the substrate due to subsurface implantation of vacuum-arc plasma ions accelerated to an energy of 1…2.5 keV.

– Deposition of two- and multi-component hard nitride coatings of various systems: TiN, TiAlN, TiCrN, TiAlYN, CrAlN, TiAlCrN on engineering materials; the results of the study are presented in the works [31–34].

It is known that the use of DC bias potentials ranging from hundreds of volts to units of kilovolts during vacuum arc deposition of coatings of the Ti-Al-N and Ti-Al-Y-N systems improves adhesion, but at the same time leads to a significant decrease in the relative Al concentration in the coating. In contrast, in [32] it was shown that the use of the PIIID method allows the deposition of the coating with maximum hardness, in which the relative concentrations of the base metal components (Ti, Al) and the alloying element Y are almost the same as in the cathode material. A nitride with a cubic structure of the NaCl type is formed in the coating. The texture becomes 110 (in contrast to 111 at DC bias potential), the CSZ size decreases to 7…14 nm, (in contrast to 20…30 nm at DC), which significantly increases the cavitation resistance of the coating [31].

By varying the high-voltage pulsed bias potential regime during deposition, the optimal process parameters were found, under which a dense homogeneous nanostructure with fine crystallites and weak columnar morphology was formed (Fig. 4). Testing has shown that the wear resistance of such a coating is significantly improved [31]. The average rate of cavitation and abrasive wear of the (Ti,Al)N + 1 at.%Y coating is from 3 to 5 times lower than that of the (Ti,Al)N coating and is 10 times lower than that of the TiN coating deposited under the same conditions, which is characterized by a clearly pronounced columnar structure, stronger inter-crystalline boundaries, and significantly reduced surface roughness.

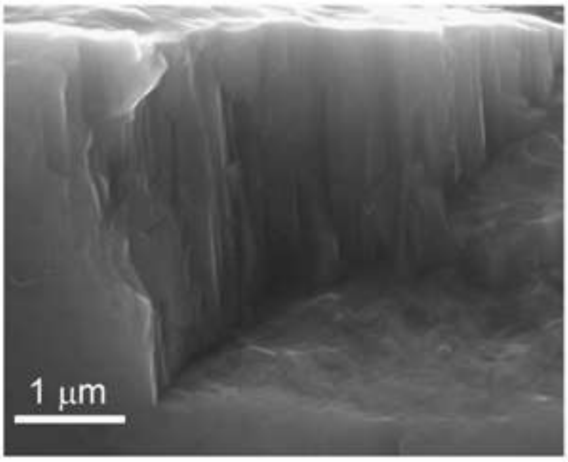

Fig. 4. SEM of the transverse fracture of the (Ti,Al)N + 1 at.% Y coating deposited by the PIIID method [31]

– PIIID vacuum arc deposition of carbon coatings, including diamond-like DLC and multilayer coatings based on them [35–37]. The dependence of nanohardness, elastic modulus, adhesion level to substrates made of various materials, as well as the content of the sp^3 phase of carbon on the parameters of high-voltage pulses of the substrate bias potential was investigated. By combining DLC layers with different properties, the search for the architecture of coatings with the highest hardness with a sufficiently high level of adhesion to engineering materials was carried out. Processes for deposition on silicon carbide the quasi-gradient DLC coatings and multilayer DLC with repeating pairs of hard-soft layers have been developed [36, 37].

In addition to scientific research, the PIIID method is used in the process of depositing protective diamond-like coatings on industrial and medical products. Among them are wear-resistant coatings on ring-shaped silicon carbide sealing elements [37] with 120…200 mm diameter, the adhesion of which reaches 40 N, and bioinert coatings on friction pairs of artificial joints made of CoMoCr alloy and surgical parts (Fig. 5).

Theoretical studies of vacuum-arc coatings deposited under conditions of applying a high-voltage pulse potential to the substrate are performed on the basis of the nonlocal thermal peak (NTP) model [38–41]. The input data of the model, namely the parameters of the collision cascade when an ion hits a solid, are calculated using the SRIM2000 package [42].

Fig. 5. Hemispherical components of friction units of artificial joints with wear-resistant DLC coating

This program allows you to consider cascades created in a solid of a given elemental composition by ions of different elements in a wide range of energies, including the range of the PIIID method. The calculation of internal stresses in a diamond-like coating formed from a beam of carbon ions in the DC and pulsed bias potential modes was performed in [39].

Within the framework of the NTP model, a formula was obtained for calculating internal stresses in a multicomponent material deposited from a mixed beam of ions of various elements with energies up to 2.5 keV [40]. The model takes into account the deposition temperature as one of the parameters.

The process of internal stress formation in the multicomponent coatings deposited from a beam consisting of Cr+, Al+ and Ti+ ions in different percentage ratios in DC and pulsed substrate bias potential modes at different deposition modes and temperatures was investigated [41]. The results of the calculations are compared with experimental data, which allows us to estimate some parameters of processes in a solid, such as the value of the activation energy of defect migration.

CONCLUSIONS

The effectiveness of using the PIIID method for developing new nanostructured coatings with improved properties has been confirmed by many studies. The advantage of the method is that the plasma is the source of ions used for both coating deposition and implantation. To accelerate ions, a pulsed high potential of negative polarity is applied to the substrate. Additional ion bombardment affects adhesion, microstructure, morphology, internal stresses and film uniformity. This approach provides an additional element of process control in plasma PVD deposition technologies, and also allows the creation of nanostructured coatings. As a result of using the PIIID method during deposition, conditions can be created for the formation of metastable phases in the coating, and importantly, this process can be controlled by setting the optimal parameters of the pulse mode.

Vacuum-arc sources, both direct current and pulsed, are widely used to create plasma in PIIID method. The most effective are filtered plasma sources, which provide almost 100% plasma ionization and deposition of coatings with the least number of defects. There are various types of filters, namely magneto-electric, curvilinear with different deflection angles, rectilinear, blinds, etc. High-voltage pulse generators are used to apply a bias potential to the substrate during PIIID coating deposition. The most common scheme includes high-voltage capacitor, from which pulses with amplitude of up to several kilovolts are applied to the substrate holder through an electronic switch.

For the past twenty years, the NSC KIPT has been conducting experimental and theoretical studies of vacuum-arc coatings deposited under conditions of applying a high-voltage pulse potential to the substrate. This activity is based on the developed original equipment, as well as theoretical calculations based on the non-local thermal peak model. In addition to scientific research, the PIIID method is used in the process of applying vacuum-arc protective diamond-like coatings to industrial and medical products.

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ВИКОРИСТАННЯ ВИСОКОВОЛЬТНОГО ІМПУЛЬСНОГО ПОТЕНЦІАЛУ ЗМІЩЕННЯ НА ПІДКЛАДКУ ПРИ ОСАДЖЕННІ ВАКУУМНО-ДУГОВИХ ПОКРИТТІВ (МЕТОД PIIID)

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Розглянуто фізичні основи використання імпульсного потенціалу зміщення підкладки (метод PIIID) у процесах осадження з фільтрованої вакуумно-дугової плазми покриттів, зокрема наноструктурних. Обговорюються технічні деталі та вимоги до обладнання для осадження вакуумно-дугових покриттів за умов використання імпульсного потенціалу зміщення. Зроблено огляд експериментальних та теоретичних робіт, виконаних у ННЦ ХФТІ, з використання методу PIIID у процесах осадження твердих вуглецевих та нітридних покриттів з фільтрованої вакуумно-дугової плазми.