INTERNATIONAL JOURNAL OF INTEGRATED ENGINEERING
ISSN: 2229-838X e-ISSN: 2600-7916

IJIE

Vol. 16 No. 9 (2024) 284-293
https://publisher.uthm.edu.my/ojs/index.php/ijie

Ion-Plasma Formation of Nanosized Coatings with Fractal Topology

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DOI: https://doi.org/10.30880/ijie.2024.16.09.023

Article Info

Received: 27 May 2024
Accepted: 24 November 2024
Available online: 29 December 2024

Keywords

Nanosized coating, fractal topology, magnetron sputtering, arc evaporator, droplet fraction, plasma flux

Abstract

Nanosized coatings with fractal topology have significant technological applicability due to their unique properties. There are several ways to provide fractalization of the coating, one of which is placing a seed into the vacuum chamber. The most used method for producing nanosized coatings is magnetron sputtering, which allows obtaining a high-quality film with a sufficiently high productivity. The notable disadvantage of such devices is a low degree of ionization of the plasma flux, which leads to poor adhesion of the obtained coating. More promising is the electric arc spraying of matter from the plasma by an anomalous glow discharge, which in turn has a significant concentration of the droplet fraction existing together with the plasma flux. These droplets greatly increase the coating roughness and make electric arc installations inapplicable in nanotechnology. A specially designed arc evaporator, including metallic nonmagnetic dampers, provides elimination of the droplet fraction. Experiments on the deposition of titanium nitride and copper coatings on high-carbon steel substrates have shown both the possibility of obtaining uniform nanostructured coatings and coatings with fractal topology in the case of introducing specially prepared seed into a vacuum chamber.

1. Introduction

Formation of nanosized coatings with fractal topology is not only a fundamental problem, but also has an important technological aspect, since the properties of such coatings will be distinguished from the characteristics of coatings synthesized in the traditional way [1]–[8]. A similar situation developed at some time with the assessment of the crystalline and amorphous state. Until a certain time, it was believed that only the crystalline state corresponded to the concept of a solid body. However, as it turned out, the amorphous state is very interesting, especially from the point of view of technology, since in the amorphous state it is possible to implement structures with completely unusual properties [9], [10]. Since then, interest in substances with an amorphous structure has only increased.

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Similarly, there is an increasing interest in obtaining nanosized coatings with fractal topology [11]–[17]. Forming fractal structures became very promising after their unique characteristics were found, which makes it possible to use them in medical applications, suppressors of atypical electromagnetic waves, light filters, etc. It is very important to produce these coatings for extended analysis of their characteristics, which nowadays have been examined insufficiently. The most common and technologically advanced method for producing nanosized coatings is magnetron sputtering, which allows obtaining high-quality films with a sufficiently high productivity [18]–[23]. The process of fractalization of nanosized coatings in the course of their synthesis can be triggered by various technological techniques and methods.

2. Materials and Methods

2.1 Methods for the Formation of Fractal Nanosized Coatings

The simplest method for producing fractal nanosized coatings is to create a transition thin-film layer on the plane of the substrate, not necessarily nanosized, but already having a fractal topology obtained in one way or another [24]–[33], but less complex than the synthesized one (Fig. 1). On the surface of this transition layer, the required nanosized coating will be synthesized. The problem is with which physical agent and how the fractal structure of the transition layer will be implemented.

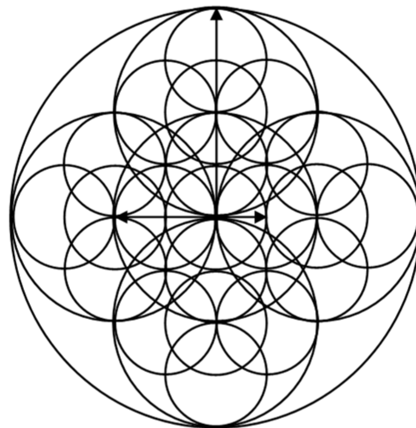

Fig. 1 *One of the variants of the matrix with a fractal structure*

The special graphics on the plane of the substrate can be created, for example, in the form of a complex system of straight lines or grooves intersecting at different angles and representing a fractal self-similar structure. Then the adsorbed atoms of the synthesized nanosized coating deposited on the substrate will be integrated into its structure by the mechanism of self-organization [34], [35]. It is more difficult to introduce a fractal structure into the vacuum chamber and this may be a previously obtained nanosized coating film with a fractal topology [36], [37]. Such a sample, called a seed, can be placed in different positions inside the vacuum chamber. For example, on the walls of the chamber or in the immediate vicinity of the substrate on which a new coating is to be applied. One more important detail is no observed correlation between the synthesized structure, the fractal topology of the seed and the location of the seed.

The other way is the use of structured electromagnetic radiation, introduced in one way or another into the space of the vacuum installation. The structuring of the electromagnetic radiation used (and usually this is monochromatic scattered radiation from an incoherent source) can be carried out by applying special diffraction gratings.

2.2 Application of Sputtering Devices for the Formation of Nanosized Coatings

From the point of view of the productivity and efficiency of the synthesis of nanosized coatings by magnetron spraying methods, the use of arc discharge is a priority [38], [39]. An effective type of such device is a magnetron ion sputtering device, in which anomalous glow discharge plasma is formed, into which neutral target atoms fall, later knocked out of it by a flux of incident plasma ions and subsequently applied on the substrate and forming the synthesized coating.

The disadvantages of magnetron ion sputtering devices include a low degree of ionization of the vapors of the sprayed substance, which is approximately 5–10 %, which leads to a deterioration in the adhesion of the applied coating. On the other hand, magnetron ion sputtering installations do not allow heating the substrates immediately before coating when it is required, for example, when hardening metal-cutting tools to a

temperature of 350–400 °C and in a number of other applications, which does not contribute to obtaining high-quality resistant coatings and also impairs adhesion.

More promising is the utilization of the electric arc spraying of matter from the anomalous glow discharge plasma. The benefit of using the arc evaporator for coating application is the better level of adhesion of the substance deposited onto the substrate [40]–[43]. When an arc discharge occurs, a so-called cathode spot is formed on the working side of the evaporator with a burning arc discharge diameter of about 1–3 μm and measured current values of 100–150 A. A very high level of current density is formed on the working side of the arc evaporator, which provides local melting in the spot area. The molten metal evaporates, and the metal atoms are fully ionized (~100 % ionization) [44]–[46]. These ions are accelerated by an electric field (the substrate is set at a negative potential) and deposited with high energy, which provides a high level of the obtained coating adhesion. Such an installation is called a cathode-ion bombardment device. The disadvantage of the cathode-ion bombardment design is the existence of droplets in the plasma flux, which absolutely excludes the use of such devices for forming nanosized coatings, since the dimensions of the droplet clusters exceed the thickness of the coating, which does not allow obtaining uniform thickness, as observed in Fig. 2. This coating structure is irretrievably damaged.

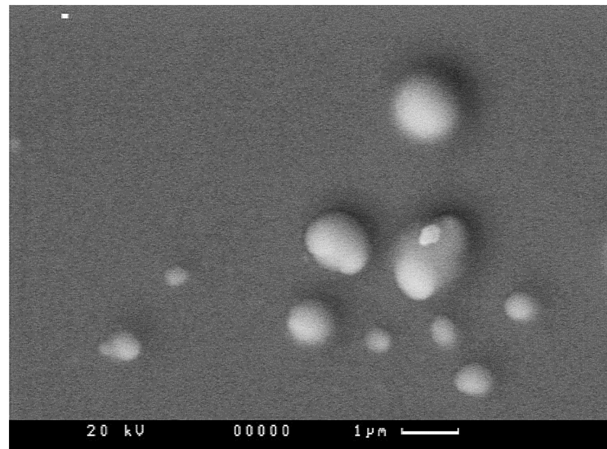

Fig. 2 *SEM image of copper droplets on the surface of a coating*

The droplet fraction appears because the flux of ions descending on the molten metal provides force action on the melt, which brings it to spray in the shape of drops up to 10 μm in size [47]–[50]. Moreover, local excessive heating of the spot on the target and explosive evaporation are very probable. These phenomena greatly raise the coating roughness, which additionally leads to the impossibility of using cathode-ion bombardment type devices in nanotechnology. Until now, there are no well-founded theoretical ideas, full-fledged theoretical models and accurate experimental data on the processes in the cathode spot and the cathode plasma flux of the arc, despite the abundance of serious research in this direction [51]–[61]. These features prevent the use of computer modeling methods to describe the processes occurring in a plasma flux. This leads to the need to apply various empirical concepts based mainly on experimental research. Other disadvantages of the cathode-ion bombardment design also include overheating of the substrates by a high-energy metal plasma flux with energy of about 4 keV, which can lead to heating of the vacuum chamber itself and initiates the release of various gases from the chamber walls, resulting in alteration in both the physical and chemical characteristics of the obtained coating.

3. Results and Discussion

3.1 Modernization of the Installation for the Formation of Nanosized Coatings

To reduce the possibility of transporting droplets to the substrate, an auxiliary magnetron sputtering device with supplemental AC and DC power sources, connected according to a certain scheme and algorithm, was introduced into the chamber. However, this does not entirely solve the problem of eliminating the droplet component. Therefore, a more complex modernization of the arc evaporator was made, consisting of a cooled cathode, an arc discharge ignition system, an arc discharge stabilization coil, and a focusing coil installed coaxially with the cooled cathode. The arc evaporator was supplied with a metallic nonmagnetic damper, having the shape of a disk, the transverse size of which is equal to the size of the cooled cathode. This damper was located between the focusing and stabilization coils coaxially with the cooled cathode and an additional damper located inside the focusing coil. The latter is made of nonmagnetic metal in the shape of a truncated cone, the

radius of the larger base of which is the same as the inner radius of the focusing coil, and the diameter of the smaller base facing the cooled cathode is equal to twice the diameter of the first damper, as presented in Fig. 3.

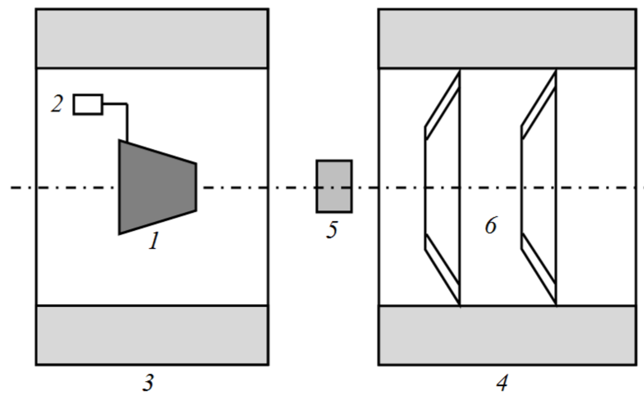

Fig. 3 *Modernized arc evaporator scheme*

In this scheme, 1 is a cooled cathode that serves as a source of sputtering substance. An arc discharge ignition system 2 with the help of a stabilization coil 3 forms an arc discharge on the working surface of the cathode, which vaporizes the cathode substance. Using the focusing coil 4, a plasma flux is created, aimed along the axis of the system. The plasma flux includes both ions of the sprayed material and its droplets. A damper 5 placed on the axis of the arc evaporator purifies the flux of the sprayed substance from the direct-flying drops, and dampers placed inside the focusing coil housing 6 remove the droplets moving at an angle from the cathode. The system works as follows. An arc discharge is excited on the side surface of cathode 1, which is transferred to the end surface of the cathode using the magnetic field of the stabilization coil. Further, the discharge is held on it by the same magnetic field and forms a randomly moving cathode spot. The cathode substance evaporates from the cathode spot, moves through the plasma area and is nearly entirely ionized. The power of the arc discharge is such that ions return to the melt of the cathode spot, exert pressure on it and, as a result, lead to splashing of the melt (Fig. 4). The observed droplet component clusters are electroneutral and aimed away from the cathode [40], [48], [50].

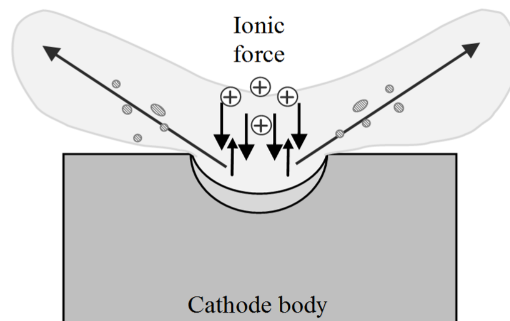

Fig. 4 *Appearance of a droplet fraction from the cathode surface*

The ions attracted from the plasma produce a flux with the maximum current density distribution along the axis of the considered system. Drops of molten metal move in all possible directions. Since the damper in the form of a disk is not electrically connected to any other element of the evaporator, the ions falling on it from the cathode will charge it. The charge of the damper will grow until the electric field created by it totally repels the ions, which will move around the damper and fly along the same path, rotating around the magnetic field lines of the coils. The droplet component of the evaporation products traveling along the axis of the evaporator is bounded by the damper located on the axis of the installation, since the drops have no charge, and the transverse size of the damper is equal to the area of the cathode cross-section. At the same time, drops that are sprayed at an angle are eliminated by the damper in the form of a truncated cone, fitted closely to the body of the focusing coil. As a rule, one damper is sufficient, but in some cases their number can be increased to two. The slope of the cone towards the cathode prevents the reflection of small droplets from it and eliminates them from the deposition stream.

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Since the damper in the shape of a disk is charged, the ion flux will move around it and the ions will travel in a straight direction, unlike the droplet component. The transverse size of the disk-shaped damper must be equal to the area of the cathode cross-section to separate the coaxial droplet flux. If it is narrower, then part of the droplets will still move in the forward direction. If it is wider, it will lower the flux of ions around the damper. As is demonstrated by the experimental study, the narrower size of the cone should provide elimination of the droplet component and the transfer of the plasma flux. Meanwhile, it is satisfactory that its transverse size is equal to twice the size of the damper, coaxial with the cathode of the evaporator.

Unfortunately, in such a design it is impossible to control the plasma flux, which is formed by an arc evaporator and, consequently, it is quite likely that the operational characteristics of the coatings obtained are not high enough and the consumption of the sprayed material is increased. Therefore, to control the plasma flux, the winding of the focusing coil is connected to its power source through an additional adjustable switch that allows changing the direction of the current in the focusing coil to the opposite. The housing of the focusing coil is connected to an additional power source through an additional switch that allows it to supply positive or negative smoothly adjustable potential relative to zero to the housing of the focusing coil, and also completely exclude the supply of any potential to its body. So it is perceivable to increase the efficiency of the sprayed material consumption when depositing thin coatings in the case of objects of small sizes and classes (one focusing) and large extended objects (panels, tapes and pipes – another focusing). This also makes it possible to reduce the consumption of the sprayed substance, since the deposition area of the material from the plasma flux correlates to the measurements of the workpiece, and to improve the quality of the coatings obtained by isolating the focusing coil from the sprayed material.

If the magnetic fields of the stabilization and focusing coils of the system are simultaneously turned on oppositely, the positive ions of the plasma flux will bend around the damper, rotating around the magnetic field lines. If the housing of the focusing coil is not electrically connected to any element of the vacuum installation, the plasma ions will be deposited on it until the created electric field repels them, causing the effect of narrowing (focusing) of the flux. A similar pattern (focusing effect) will be observed when a positive potential (up to +50–70 V) is applied to the focusing coil housing, but at the same time a more significant focusing effect can be obtained than with the floating potential of the coil housing.

In the case of a zero or a small negative potential applied to the body of the focusing coil and unidirectional magnetic fields of the stabilization and focusing coils, the plasma flow will begin to expand, thereby increasing the sputtering zone, which is important when coating long substrates. When the magnetic fields of the stabilization and focusing coils are on the opposite side, and when the focusing coil housing is disconnected from the ground, the spraying time increases by 3–5 times. Otherwise, the matched magnetic fields of the coils and the grounded housing of the focusing coil or its small (10–30 V) negative potential allows coating with a maximum length of up to 350–400 mm. Thus, there are two modes of operation that ensure the absence of a droplet phase in the coating:

1. Application of high-quality coating on parts of small (up to 50–60 mm) sizes – counter magnetic fields of stabilization and focusing coils and positive or zero potential of the focusing coil housing;
2. Coating on long parts or decorative coating on numerous small (up to diameters of about 30 mm) and up to 300 pieces – with coordinated, unidirectional magnetic fields of stabilization and focusing coils with negative or zero potential on the body of the focusing coil.

2.1 Formation of Nanosized Coatings Using the Developed Installation

As an assessment of the efficiency of the proposed system, 4 samples of high-carbon steel with dimensions of 30×30×3 mm with surface heterogeneity of 0.2–0.3 μm were considered as substrates. The first sample: titanium nitride coating, Bulat-6 installation [62], standard factory arc evaporator. The result is that the sample coated with titanium nitride had a coating heterogeneity of 1.5–1.8 μm. Obviously, there can be no question of any nanosized coatings in this case, since the unevenness of the resulting coating due to the droplets is an order of magnitude greater than its thickness. The second sample: titanium nitride coating, the arc evaporator according to the above design. The result is an inhomogeneity of the coating surface of 0.3–0.5 μm, which is significantly better than with a standard arc evaporator, but it is not quite suitable for the formation of nanosized coatings, since the resulting inhomogeneity is comparable to the thickness of a coating.

The third sample: the coating is refined copper, the arc evaporator is standard. The coating thickness is 0.5 μm. The size of the droplets on the surface is 1 μm or more, which does not allow talking about the nano size of the coating. The fourth sample: the coating is refined copper with no droplet phase, the arc evaporator according to the above design. The coating thickness is 0.2 μm, the heterogeneity of the surface is 0.05–0.1 μm (Fig. 5). The coating meets the nanosize criterion. However, it should be mentioned that the copper coating obtained in this way, when analyzing the internal structure using scanning electron microscopy, revealed a latent internal structure with signs of fractality.

Measured reflection coefficients at a wavelength of 450 nm as a factor of resulting coating uniformity were: first sample 40 %, second sample 82 %, third sample 50 % and fourth sample 85 %. To obtain the fractal topology of the coating, a seed substrate was created, the surface of which is a matrix constructed of several equal elements. Any and each of these elements have central symmetry and represent a section of the whole fractal picture with a fractalization degree of three or more [16], [17]. By excluding the droplet component from the plasma flux and introducing the appropriate specially prepared seed into the vacuum chamber volume, nanosized coatings of refined copper with fractal topology were obtained (Fig. 6, 7).

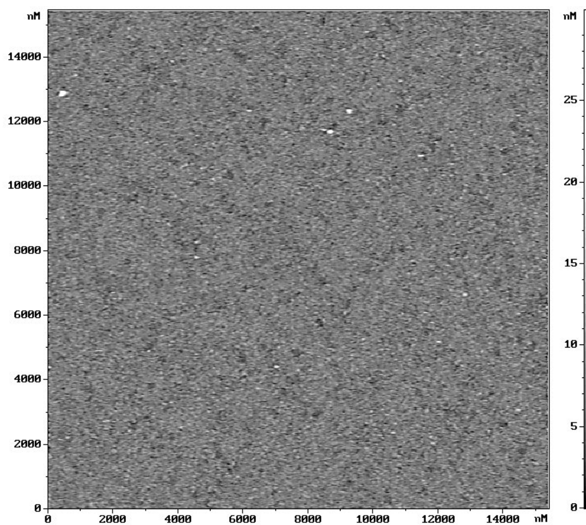

Fig. 5 *AFM image of a condensed coating without droplet phase*

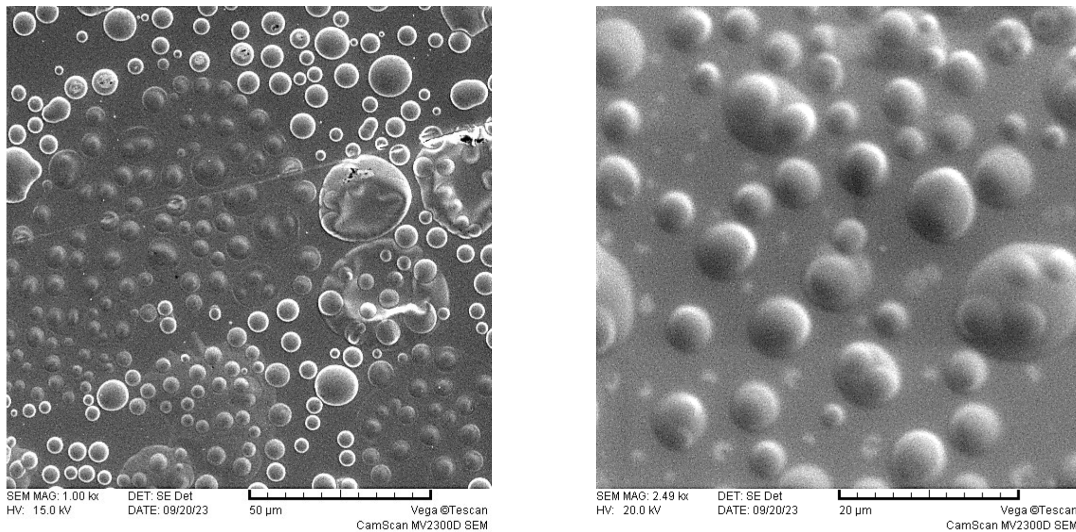

Fig. 6 *SEM images of a copper coating with fractal topology*

When visually examining samples of obtained copper coatings, the following picture is observed: the coating represents a quite erratic grid of tiny embryos that have dome-like shapes of roughly equal dimensions. There is a certain general order of arrangement of these elements, different from the chaotic distribution. Meanwhile, the general surface topology may be considered as a polysyllabic fractal structure with a notable degree of self-similarity. Different surface objects with distinguishing fractalization levels and area filling are synthesized side by side. If these structures are not limited in space for subsequent growth, they develop without confrontation and interosculating objects are not observed. At this point, no precise relationship between the acquired surface topology, technological parameters of the operational regimes and mutual arrangement and type of the previously prepared seed was found.

The appearance of domes and other structures may be associated with local gas release from the substrate during the condensation of the coating. During magnetron sputtering, as a rule, in the zone of highly ionized plasma, the influence of charged particles has a decisive effect on the properties of the coatings obtained. The presence of numerous charged particles (ionized atoms of the sprayed material, electrons with high energies)

causes the growth of the emerging topology during the existence of many crossed electromagnetic fields. At the starting stage of nucleation, the energetically advantageous ion deposition points are determined by the structure of the seed matrix.

Further growth of films occurs according to the following scheme: an increase in the size of the embryo leads to the concentration of local electric fields and heating of the condensing film due to an intense flow of charged particles. Local overheating of the film–substrate interface leads to the release of gases adsorbed on the surface and absorbed in the subsurface layer. Given the plasticity of the thin film, this leads to its swelling, followed by the formation of domed, hemispherical, and other structures of clusters of micrometer sizes. Described processes completely correspond to the concept of group processing technology, because they make it possible to form in a single technological cycle a regular topology consisting of locally arranged atomic structures on the entire coating surface.

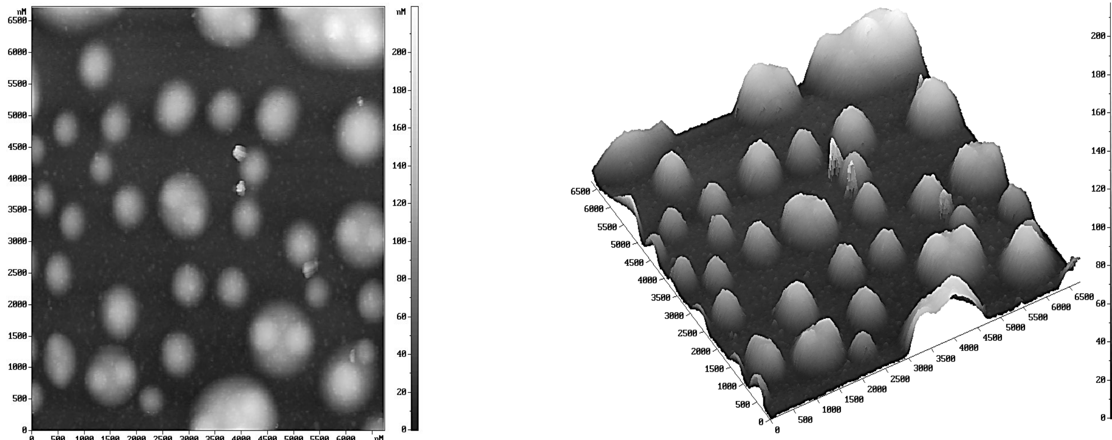

Fig. 7 *AFM images of a copper coating with fractal topology*

3. Conclusion

As a result of the work, a method has been developed for the controlled formation of structured nanosized coatings, which allows creating an ordered topology, in particular a fractal one, in one technological cycle. The results of the synthesis of coatings with fractal topology using electric arc deposition of a substance from anomalous glow discharge plasma onto a seed with a previously obtained fractal topology are presented. The use of cathode-ion bombardment devices requires the adoption of preliminary measures to purify the plasma flux from the electroneutral droplet phase, which does not allow the use of electric and magnetic fields to remove and neutralize them, but requires the use of other structural solutions. Formation of fractal nanoscale coatings is not only a fundamental scientific challenge, but also has many important technological applications, as the properties of these coatings will differ from those of coatings synthesized using more traditional methods. The authors propose to conduct additional experimental studies in the future, the results of which will allow choosing suitable seed parameters and its location to achieve controlled growth on the coating of self-organizing ordered structures that will comply with the principles of group processing and can be applied to obtain elements of electronic technology. Understanding the principals of the growth of coatings with fractal topology will lead to the possibility of creating similar three-dimensional fractal structures.

Acknowledgement

The authors would like to express their heartfelt gratitude to Kotelnikov Institute of Radioengineering and Electronics, Jinan University and Saint Petersburg Electrotechnical University "LETI" for providing the opportunity to conduct this research.

Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

The authors confirm contribution to the paper as follows: ***study conception and design:*** *Alexander A. Potapov, Vladimir I. Margolin;* ***data collection:*** *Viktor A. Tupik, Dmitrii K. Kostrin;* ***analysis and interpretation of results:*** *Alexander A. Potapov, Viktor A. Tupik, Vladimir I. Margolin, Dmitrii K. Kostrin;* ***draft manuscript preparation:*** *Vladimir I. Margolin, Dmitrii K. Kostrin. All authors reviewed the results and approved the final version of the manuscript.*

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