UDC 621.793.7:620.3
doi: 10.32620/aktt.2020.6.08

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DISCHARGE CHARACTERISTICS OF THE MAGNETRON SYSTEM FOR SPUTTERING, DEPOSITION, AND NANOTECHNOLOGY APPLICATIONS

Magnetron sputtering is known for years as a powerful tool for coating deposition of cutting tools and machine parts. However the experimental measurements of the magnetron discharge parameters are still necessary to provide a consumer of the magnetron system with the reliable characteristics. A voltage-current relation is the most applied characteristic of the discharge, and it is described as the power low of a type $U = U_0 + aI^n$, where U and I are the voltage drop and the discharge current, respectively, and U_0 and n are constant. First part of the research is dedicated to the experiments conducted in the magnetron setup provided with the titanium cathode in a vacuum chamber filled with argon or argon-nitrogen mixture, and the constants are determined for the particular geometry of the magnetron sputtering system. The obtained results can be used to choose the operation modes for the traditional applications of the magnetron discharge such as ion cleaning and heating of the non-magnetic workpieces arranged on the cathode, as well as for the sputtering deposition of the titanium and titanium nitride coatings on the surfaces of the workpieces located above the magnetron cathode. In the next part of the research the novel application of the magnetron for production of carbon nanostructures is considered. For the purpose, a layer of expanded graphite is arranged on the magnetron cathode, and the discharge is initiated in oxygen atmosphere. It was found that for the time interval of a few hours the discharge is described as a superposition of the typical magnetron glow with arc spot generation, and the intensity of the arcs is not decreased with time. At that, the arc initiation was accompanied with the formation of clusters of the graphite cathode. The process is explained in terms of the cathode spot generation at the interaction of the arc plasma with the non-melting material. This process can be beneficial for the development of the plasma reactors for the large-scale production of the carbon species at the low gas pressures suitable for the magnetron discharge operation. Thus, the magnetron sputtering systems provided with the expanded graphite cathode can be considered as the tool to grow carbon nanospecies in the arc discharge cathode spots.

Keywords: *plasma; magnetron discharge; voltage-current relations; nanotechnology; carbon nanoparticles.*

Introduction

Magnetron sputtering systems and vacuum arc guns are considered as the main technological plasma sources used to generate flows of plasmas of conducting materials [1, 2]. Magnetron discharge is widely applied in science and technology for decades, while investigation of its operational principles is still a problem [3, 4], and large number of theoretical and experimental researches is dedicated to it [5, 6].

Magnetron discharge is considered as glow discharge sustained by the secondary electrons emitted from the cathode surface at the interaction of the cathode with plasma ions [7, 8]. Magnetic field applied above the cathode, is the distinguishing feature of the magnetron discharge; the topography of the field is essential for the discharge operation. Usually, the magnetic field is between $10^{-3}…10^{-2}$ T, and cannot affect the motion of the relatively heavy ions, while the motion of the electrons is affected greatly by the field. Thus, the electron component of plasma is considered as "magnetized" in the magnetron discharge, while the ion component – as "not magnetized". The magnetic field lines are considered as equipotential under the condition of the limited mobility of the plasma electrons across the magnetic field, and relatively strong electric field can be generated in plasma to accelerate plasma ions toward the cathode [9].

The magnetic field directed perpendicular to the electric field, significantly increases the electron path between the cathode and anode of the discharge system, thus providing the effective ionization of the background gas to obtain the high density plasma [10]. The electron mobility across the magnetic field is the discussion issue at the development of the magnetron sputtering devices [11], thus making it difficult to theoretically predict the conductivity of the discharge gap. The discussed conductivity mechanisms include classical [12], Bohm [13], and near-wall conductivities [14], to mention a few.

Nowadays, radiofrequency (RF) [15] and pulsed system [16] technology are implemented in industry to overcome the limitations implied by the basic direct current (DC) approach: arcing, small fraction of ions in the gas-plasma mixture (the ionization degree of the background gas is usually of a few percent), low energy of the ions, as well as limitations implied by the cooling system of the magnetrons. However, DC setups are still widely applied for coating deposition or for surface cleaning and heating by use of flows of energetic ions. In this paper we describe the application perspectives not only in traditional coating industry but also in nanoscience technology.

Formulation of the problem

Due to the problems in the theoretical description of the magnetron discharge, the prediction of the discharge operation based just on the knowledge of the discharge gap geometry, gas flows, partial pressures, and characteristics of the electrical power supply, is difficult. The configuration of the arced magnetic field with negatively-biased surface is widely applied for the ion treatment, and is equivalent to the configuration of to the magnetron sputtering systems. The control of the operation parameters in such systems means the necessity of studying the current-voltage relations of the discharge. With respect to the surface treatment, three operation modes can be distinguished: with the negative rate of the surface geometry change (i.e. the surface sputtering); with zero rates (i.e. the surface modification), and with the positive rate (i.e. the coating deposition). The temperature mode of the surface is important as well. To control the surface treatment parameters, two characteristics of the ion flow should be precisely controlled, namely, ion energy, and density of the ion current extracted from plasma to the substrate. The former depends on the drop of the electric potential across the discharge gap, while the latter are determined by the electric supply power adsorbed by the discharge gap. Unfortunately, in spite of the progress in the magnetron researches, each system requires the experimentally established dependencies, because large number of parameters affects them, such as strength of the magnetic field, its topography, a configuration of a vacuum chamber, gas supply rate etc. At that, the discharge voltage is usually estimated as a dependence $U \approx \varepsilon_c/\gamma_{eff}$, where ε_c is the energy loss for generation of one ion-electron pair, and γ_{eff} is considered as the “effective” coefficient of the secondary electron emission [17]. The ionization and conductivity processes specify the voltage-current relations of the magnetron discharge, which are very important from the practical considerations, since the energy of the plasma ions and the current of the ions extracted from the plasma to the cathode, are dependent on the parameters [18]. For DC magnetrons the discharge current is usually described by the following power law: $I = aU^n$, where U is the discharge voltage and n is considered as a parameter describing the effectiveness of the electron confinement by the magnetic field. At that, n can be varied from 1 to above 20 at the dependence on the cathode material, applied magnetic field, and gas pressure [19]. Unfortunately, this approach is not fruitful for the description of the newly-tested configurations of the magnetron sputtering systems.

To overcome the obstacle, the experimentally measured characteristics of the discharge obtained for a specified vacuum setup arrangement are used. Since the surface coating is often conducted through two-step process, when the ion cleaning and heating are performed during the first stage, and the reactive coating deposition is carried out on the second stage, both of the stages are studied in the research. Argon is the main gas for the preparatory (cleaning and heating) stage, while number of gases are applied for the second stage. The gases should match the magnetron cathode which is a source of the metal atoms sputtered at the interaction of the ion flux with the cathode material. Titanium nitride is the most used coating material which requires titanium cathode and nitrogen as the reactive gas, and this composition is the main subject of our study. In this paper, the current-voltage characteristics of the magnetron discharge over titanium cathode are investigated at the different gas pressures and rates.

In addition to the traditional implementation of the magnetron sputtering system, a novel application in the field of nanotechnology is also studied in the paper. As it is known, arc spots initiated above the graphite cathode, are powerful tool in generation various nanospecies of graphite, such as nanoclusters, nanotubes, and nanosheets [20, 21]. Usually, the process is conducted under the high-pressure conditions; however, here the magnetron cathode made of expanded graphite, is treated at low pressure, and the discharge parameters suited for the nanomaterial production, are studied.

Experimental part

A schematic of the experimental setup is shown in Fig. 1. The vacuum chamber is a vessel of a cylindrical shape with the outer diameter 310 mm, inner diameter 300 mm, and height of 350 mm. A planar magnetron with a titanium cathode with a diameter of 236 mm was mounted in the chamber, and the chamber was filled with argon, or argon-nitrogen mixture at a certain gas flow ratio. When measuring the voltage-current relations in the configuration with the titanium cathode, ten measurements were made for each experimental point.

In the experiments where the discharge characteristics of the system with the graphite cathode were performed, the relations were obtained by use of the customary-designed data acquisition system, which is able to write the discharge parameters such as voltage, current, and gas pressure at the frequency of 25 Hz. When carrying out the research, the atmosphere at 25 Pa of oxygen pressure was maintained.

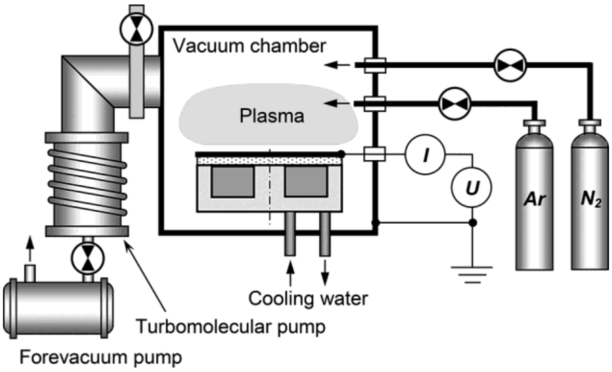

Fig. 1. A schematic of the experimental setup

A photograph of the setup is shown in Fig. 2. The experiments showed quite different behavior of the discharge for the setups with titanium and expanded graphite cathodes.

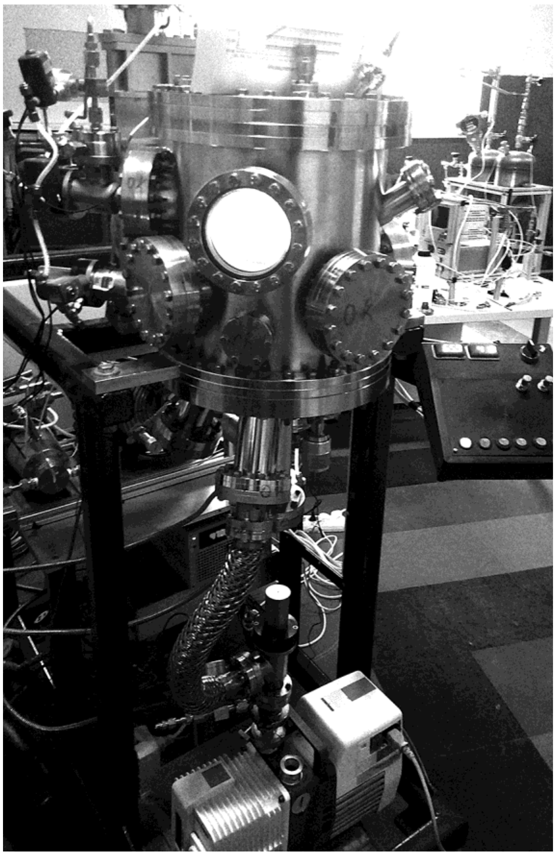

Fig. 2. Photograph of the experimental setup

When treating the titanium cathode, plasma appeared as a bright torus above the cathode surface at the discharge ignition, thus forming the classical shape of the magnetron plasma discharge where the brightest part corresponds to the region where the magnetic field lines are parallel to the cathode surface. A sputtered trench known as ‘race track’ was formed on the cathode under the plasma. In general, the observed phenomenon corresponded to the appearance of the magnetron discharge in both of the operation modes – as for the sputtering in the argon atmosphere as for the deposition in the nitrogen environment. A photograph of the discharge operation is shown in Fig. 3.

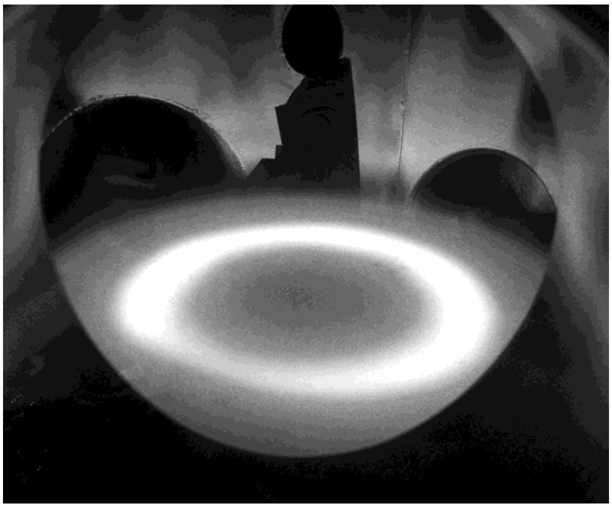

Fig. 3. Photograph of the magnetron plasma discharge

Results and discussion

When investigating the parameters of the system with respect to the sputtering a range of the discharge currents up to 1.3 A for the discharge voltage drop of 370 to 900 V, and the argon pressure range of 4.6 to 5 Pa was studied. The pressure range is conditioned by the rational operation of the magnetron plasma source with respect to the discharge ignition and sustaining, as well as to get the widest range of the process control. The voltage-current relations obtained in the experiment are shown in Fig. 4.

As it can be seen, the higher argon pressure of 5 Pa results in the characteristics with the low regulation ability because of the saturation on the voltage drop, when the big change in the current does not lead to the significant change in the voltage drop and the ion energy, respectively. Since the ion energy is approximately equal to the voltage drop, it can be concluded that the energy does not exceed 600 eV in this case. This value is useful for heating the samples mounted on the cathode, when the cathode serves as the sample holder, yet the effectiveness with respect to the sputtering is not high. Unlike this, the decreased argon pressure of 4.6 Pa allows regulating the ion energy up to 900 eV with the current change, thus making the operation mode suitable for the effective sputtering.

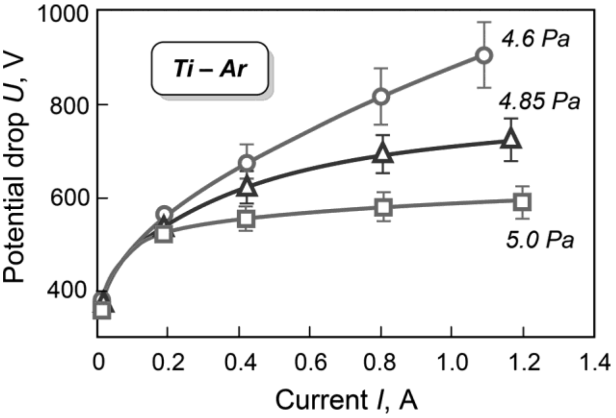

Fig. 4. Voltage-current relations of the magnetron discharge with the titanium cathode in argon environment

The measurements of the voltage-current relations confirmed the power law described in the previous section. In the experiment, the following dependencies of the voltage drop U (V) on the discharge current I (A) and gas pressure P (Pa) were obtained:

$$U(I, 4.6\,\mathrm{Pa}) = 370 + 500I^{0.6}, \tag{1}$$

$$U(I, 4.85\,\mathrm{Pa}) = 370 + 350I^{0.42}, \tag{2}$$

$$U(I, 5.0\,\mathrm{Pa}) = 370 + 220I^{0.2}. \tag{3}$$

For the deposition stage, the current-voltage relations of the magnetron discharge with titanium cathode in argon and nitrogen gas mixture at the different total gas pressures and argon-to-nitrogen ratio are shown in Fig. 5. As it can be seen, presence of nitrogen decreases the voltage drop with respect to the values shown in Fig. 5 at the same currents; thus, the less energy is required to get the same ion current. This fact correlates with the decrease in the ionization potential of nitrogen (14.53 eV) with respect to argon (15.76 eV).

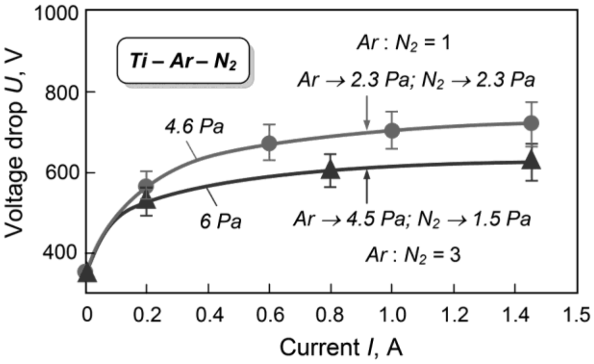

Fig. 5. Voltage-current relations of the magnetron discharge with titanium cathode in argon and nitrogen gas mixture at the different total gas pressures and argon-to-nitrogen ratio

The following dependencies of the voltage drop on the discharge current and gas pressure were obtained:

$$U(I, 4.6\,\mathrm{Pa}) = 340 + 370I^{0.3}, \tag{4}$$

$$U(I, 6.0\,\mathrm{Pa}) = 395 + 220I^{0.3}. \tag{5}$$

The discharge behavior changes drastically, when the discharge is ignited above the expanded graphite cathode under condition of oxygen atmosphere in the chamber. At that, the stable glow is not reached for the time intervals comparable with the whole time of the ion treatment, and the discharge appearance is a set of arc discharge breakdowns superimposed with the glow discharge. A typical fragment of the time dependence of the discharge voltage *U* and current *I* on time *t* is shown in Fig. 6.

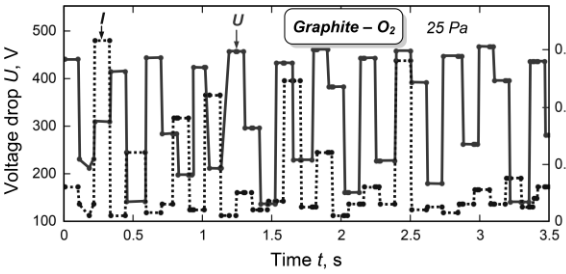

Fig. 6. Characteristics of the magnetron discharge at oxygen pressure of 25 Pa

A photograph of the discharge is shown in Fig. 7. It can be seen that the arc initiation is followed by the formation of the clusters of the cathode material, which can be associated with the bright beams protruding from the arc spot. This process is defined by the graphite properties that quite differ from the characteristics of titanium. When the arc discharge spot is generated above the titanium cathode, its generation is promoted by the microprotrusions on the cathode surface, because the electric field is greatly enhanced above the sharp features of the negatively biased surface of the cathode. As a result of the arc generation, the titanium protrusion melts, and the electric field weakens, thus unable to sustain the arc.

After the arc distinguishes, the surface of the cathode made of melting material (titanium) is smoother, thus preventing the generation of a new arc. Opposite to that, the arc generation above the expanded graphite surface does not melt the protrusion yet generates the significant elastic stress, which is released then at the formation of the solid cluster ejected from the surface with the formation of a crater with sharp edges. Thus, the surface of the cathode made of non-melting material does not become smoother after the arc generation, and the number of the protrusions, which are the concentrators of the electric field, is not decreased with time. This process is beneficial for the formation of the nanoparticles and growth of the nanostructures of different dimensionality.

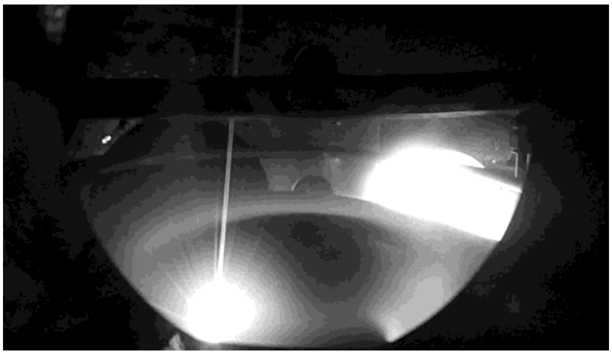

Fig. 7. Photograph of the process of the formation of the clusters of the cathode material in the arc discharge spot

Application perspectives

The results obtained in the research can be directly connected to the needs of the modern industry, as well as for theoretical work. The former application implies the use of the obtained voltage-current relations for the deposition of TiN coatings on the surfaces of the machine parts or cutting tools by use of the developed magnetron sputtering system. For that, the traditional pattern should be introduced, when the voltage-current relations for the setup with argon atmosphere are applied when a titanium sub-layer is formed on the parts or tools before the deposition of the metal ceramics. The cleaning and heating of the sheet part installed in the setup and used as the magnetron cathode is also the way of the implementation of the results.

The results of the oxygen treatment of the expanded graphite cathode in oxygen plasma are promising with respect to the nanotechnology yet the number of the applications is a topic for the separate researches. Up to now, it can be stated that the stable generation of clusters is possible with the proposed technique, and the next stage of the research should imply the collection of the graphite species generated in the arcs and ejected to the volume of the processing reactor.

Conclusions

In this paper the characteristics of the magnetron sputtering system were investigated with respect to the traditional applications such as ion cleaning, heating, and coating deposition, as well as for the prospective use in the field of nanotechnology, namely, generation of carbon nanostructures of various dimensionality. The power law was established for the specific configuration of the magnetron setup with titanium cathode operating in argon or argon-nitrogen environment at the low pressure, were stable magnetron glow discharge was developed. For the generation of carbon species in the magnetron discharge ignited above the expanded graphite cathode, the transient operation mode where the arc generation is superimposed on the glow discharge mode is the appropriate option. Thus, the magnetron system can be considered as the tool to grow carbon nanospecies.

Acknowledgement. The authors would like to acknowledge financing of National Research Foundation of Ukraine under grant agreement No. 2020.02/0119.

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Поступила в редакцию 12.10.2020, рассмотрена на редколлегии 16.11.2020

ХАРАКТЕРИСТИКИ РОЗРЯДУ МАГНЕТРОННОЇ СИСТЕМИ ДЛЯ РОЗПИЛЕННЯ, ОСАДЖЕННЯ ПОКРИТТІВ ТА ЗАСТОСУВАННЯ У НАНОТЕХНОЛОГІЇ

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Магнетронне розпилення роками відомо як потужний інструмент для нанесення покриттів на ріжучий інструмент та деталі машин. Однак експериментальні вимірювання параметрів розряду магнетрона все ще необхідні для забезпечення споживача надійними характеристиками магнетронної системи. Відношення напруги до струму є найбільш застосовуваною характеристикою розряду, і воно описується як ступенева залежність типу $U = U_0 + aI^n$, де U і I – падіння потенціалу та струм розряду відповідно, а U_0 і n є постійними. Перша частина досліджень присвячена експериментам, проведеним з магнетронним пристроєм, який оснащений титановим катодом, та розміщений у вакуумній камері, заповненій аргоном або сумішшю аргону і азоту; константи визначені для конкретної геометрії магнетронної системи. Отримані результати можуть бути використані для вибору режимів роботи для традиційних застосувань магнетронного розряду, таких як іонне очищення та нагрівання немагнітних заготовок, розташованих на катоді, а також для осадження покриттів титану та нітриду титану на поверхнях заготовок, розташованих над катодом магнетрона. У наступній частині дослідження розглядається нове застосування магнетрона для отримання вуглецевих наноструктур. Для цього на катоді магнетрона розміщений шар спіненого графіту, і розряд ініціюється в атмосфері кисню. Було встановлено, що для інтервалу часу в кілька годин розряд описується як суперпозиція типового магнетронного розряду з утворенням плям дугового розряду, а інтенсивність дуг з часом не зменшується. При цьому ініціювання дуги супроводжувалося утворенням кластерів графітового катода. Процес пояснюється умовами утворення катодної плями при взаємодії дугової плазми з неплавким матеріалом. Цей процес може бути корисним для розробки плазмових реакторів для високопродуктивного виробництва різних видів вуглецевих структур при низьких тисках газу, придатних для роботи магнетронного розряду. Таким чином, системи магнетронного розпилення, що оснащені катодом із спіненого графіту, можна розглядати як інструмент для вирощування наноструктур вуглецю в катодних плямах дугового розряду.

Ключові слова: плазма; магнетронний розряд; вольт-амперна характеристика; нанотехнологія; вуглецеві наночастинки.

ХАРАКТЕРИСТИКИ РАЗРЯДА МАГНЕТРОННОЙ СИСТЕМЫ ДЛЯ РАСПЫЛЕНИЯ, ОСАЖДЕНИЯ ПОКРЫТИЙ И ИСПОЛЬЗОВАНИЯ В НАНОТЕХНОЛОГИИ

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Магнетронное распыление годами известно как мощный инструмент для нанесения покрытий на режущий инструмент и детали машин. Однако экспериментальные измерения параметров разряда магнетрона все еще необходимы для обеспечения потребителя надежными характеристиками магнетронного системы. Отношение напряжения к току является наиболее применяемой характеристикой разряда, и оно описывается как степенная зависимость типа $U = U_0 + aI^n$, где U и I – падение потенциала и ток разряда соответственно, а U_0 и n являются постоянными. Первая часть исследований посвящена экспериментам, проведенным с магнетронным устройством, которое оснащено титановым катодом и размещено в вакуумной камере, заполненной аргоном или смесью аргона и азота; константы определены для конкретной геометрии магнетронного системы. Полученные результаты могут быть использованы для выбора режимов работы для традиционных приложений магнетронного разряда, таких как ионная очистка и нагрева немагнитных заготовок, расположенных на катоде, а также для осаждения покрытий титана и нитрида титана на поверхностях заготовок, расположенных над катодом магнетрона. В следующей части исследования рассматривается новое применение магнетрона для получения углеродных наноструктур. Для этого на катоде магнетрона размещен слой вспененного графита, и разряд инициируется в атмосфере кислорода. Было установлено, что для интервала времени в несколько часов разряд описывается как суперпозиция типичного магнетронного разряда с образованием пятен дугового разряда, а интенсивность дуг со временем не уменьшается. При этом образование дуг сопровождалось формированием кластеров графитового катода. Процесс объясняется условиями образования катодного пятна при взаимодействии дуговой плазмы с неплавящимся материалом. Этот процесс может быть полезным для разработки плазменных реакторов для эффективного производства различных видов углеродных структур при низких давлениях газа, пригодных для работы магнетронного разряда. Таким образом, системы магнетронного распыления, которые оснащены катодом из вспененного графита, можно рассматривать как инструмент для выращивания наноструктур углерода в катодных пятнах дугового разряда.

Ключевые слова: плазма; магнетронный разряд; вольт-амперная характеристика; нанотехнология; углеродные наночастицы.

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