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USING DC GLOW DISCHARGE FOR DIAMOND COATINGS SYNTHESIS

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The results of studies on the influence of the floating potential on substrate holder of various sizes on the shape and stability of the DC discharge burning in the presence of a magnetic field have been presented. The conditions for stable burning of the discharge for a long time (up to 6 hours) were determined at a zero potential on the substrate holder. It was shown that in the current range from 4 up to 8.5 A and in the pressure range from 75 up to 120 mm Hg on the samples located on the substrate holder it was possible to reach the temperatures from 1100 up to 1250 °C, which is quite sufficient for the nucleation and synthesis of high quality diamond coatings.

INTRODUCTION

One of the main problems in the use of diamond coatings obtained by the CVD method remains their high cost and not always high quality. Therefore, considerable efforts have been made to reduce the energy consumption of diamond coatings production and improve the quality of the structure of polycrystalline diamond coatings, which is not least due to the optimization of the gas medium excitation system, as well as to the increase in the total power of the used equipment [1–3].

At the NSC KIPT, for the synthesis of diamond coatings from the gas phase, an original method based on the use of a DC glow discharge stabilized by a magnetic field was developed and successfully used at present time [4]. Equipment of this type is much simpler and cheaper than the currently widely used microwave devices. A characteristic feature of this method is the presence of a negative floating potential on the substrate holder, that reaching several hundred volts. However, the presence of a high floating potential on an isolated substrate holder causes a bombardment of the diamond coating surface by accelerated ions and leads to the formation of structural defects in the diamond crystal lattice, and also increases the probability of the formation of new crystalline nuclei on the surfaces of growing diamond crystals. All these processes can negatively affect the quality of the obtaining diamond material.

The purpose of this work was to study the regularities of a direct current glow discharge burning and the shape of a discharge from the magnitude of the floating potential on substrate holders of different sizes in the presence of a magnetic field under conditions that ensure the synthesis of polycrystalline diamond coatings.

EXPERIMENTAL

It is known [5] that the shape of a DC discharge burning and its stability depends on the geometry of the electrodes, the distance between them, the pressure and composition of the gaseous medium, the voltage on the electrodes, the discharge current, and some other factors. This applies to any type of DC discharges in the gaseous medium, including the so-called glow discharges, which have been used to synthesize diamond coatings in this work. The general scheme of the experimental equipment is shown in Fig. 1.

To study the effect of the value of the substrate-holder potential for the stability of the discharge burning and its shape, it was provided opportunity to control this value. To do this, the substrate holder was connected to ground via a variable resistance. An ammeter was also connected to this circuit to control a portion of the current that passed through the substrate holder from the total value of the glow discharge current.

As in [4], an annular permanent magnet, closed with a copper ring anode insert, was left in the vacuum chamber. The presence of a permanent magnetic field makes it possible to increase the density of electrons in the plasma column, which increases the efficiency of the deposition of diamond coatings at the expense of additional activation of the gas mixture due to electron collisions [6].

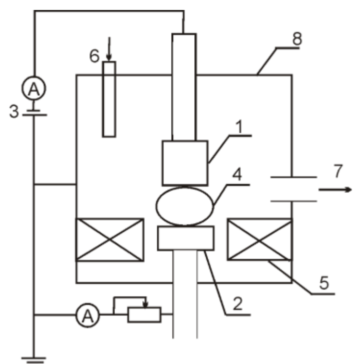


Fig. 1. Scheme of experimental equipment:
1 – cylindrical water-cooled cathode;
2 – substrate holder; 3 – power supply; 4 – discharge;
5 – annular permanent magnet; 6 – gas inlet;
7 – pumping; 8 – vacuum chamber

To study the effect of the size of the substrate holder on the discharge shape and the stability of its burning, molybdenum replaceable substrate holders with diameters of 41, 51, and 62 mm were made. In accordance with this, the diameter of the water-cooled molybdenum cathode, which was used earlier in the design to activate the gas mixture in the device with an insulated substrate holder, was increased from 60 mm to 66 mm and its shape changed from hemispherical to cylindrical.

EXPERIMENTAL RESULTS

The experiments showed that with decreasing the resistance between the substrate holder and the grounded anode insert from 250 to 50 Ω, the discharge practically did not differ from the discharge with a completely isolated substrate holder. The floating potential on the insulated substrate holder in dependence on the discharge current and the distance between the cathode and the anode could be of 180…260 V. With a resistance of 50 Ω, the floating potential of the substrate holder decreased to 80…100 V, while the current share through the substrate holder was about 20% of the total current discharge. Further reduction of resistance in the pressure range from 120 to 160 mm Hg led to frequent contraction of the discharge with an uncontrolled temperature increase on the substrate holder, which led to the impossibility of stable synthesis of the diamond film. The experiments showed that when the distance between the cathode and the substrate holder was reduced to the value equal the ratio of the distance between the cathode and the substrate holder to the cathode diameter about 1:3, it is possible to achieve a long-term stable burning of a glow discharge in the pressure range from 60 to 120 mm Hg with a completely grounded substrate holder. In this case, the discharge burns between the cathode and the substrate holder, which in this case is the anode.

The discharge burning under such conditions was stable and controlled within the limits of the discharge current changing that could be provided by an electric power source with a maximum power of 7 kW and a maximum current load up to 9 A. It should be noted that the share of the current passing through the substrate holder was 90…95% of the total discharge current. Fig. 2 shows a photograph of a glow discharge burning at a substrate holder with a diameter of 41 mm and a discharge current of 5 A. Such stable burning was observed in the pressure range of 60…120 mm Hg and methane concentrations from 0.25 up to 1%.

In the case of larger substrate holders, namely 51 and 62 mm, studies have shown that discharge with grounded substrate holders behaves in a similar manner. But it should be noted some features in the behavior and shape of the discharge with increasing the size of the substrate holder from 41 up to 50…60 mm. This concerns the influence of the discharge current on the above characteristics. Thus, the burning region of a glow discharge when a pressure in the chamber does not exceed 75 mm Hg covered the entire surface of the substrate holder with a diameter of 62 mm only when a current value was more than 8…8.5 A and had a domed shape with a flat top near the cathode center (Fig. 3).

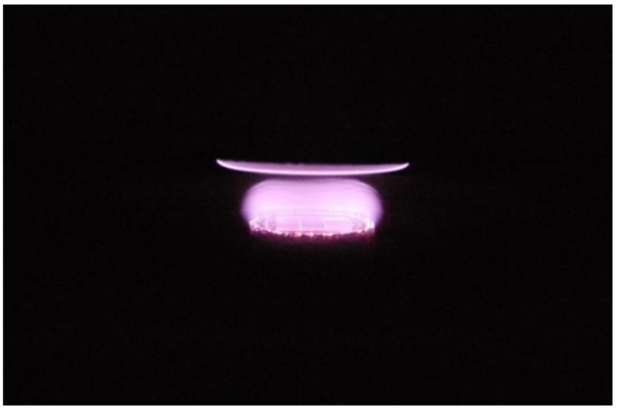


Fig. 2. A photograph of a discharge with an exposure of 1/1000 s in hydrogen medium with addition of 0.25% methane in the case of the grounded substrate holder with a diameter of 41 mm

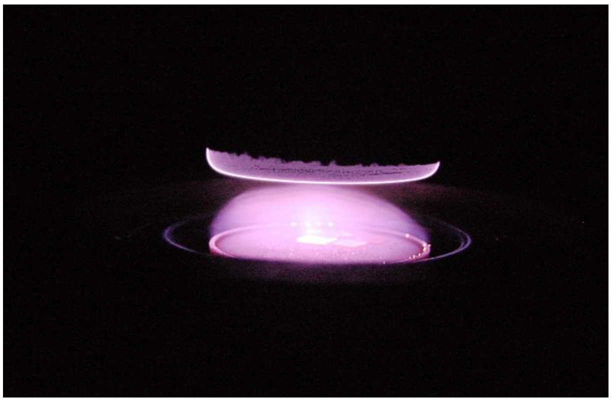
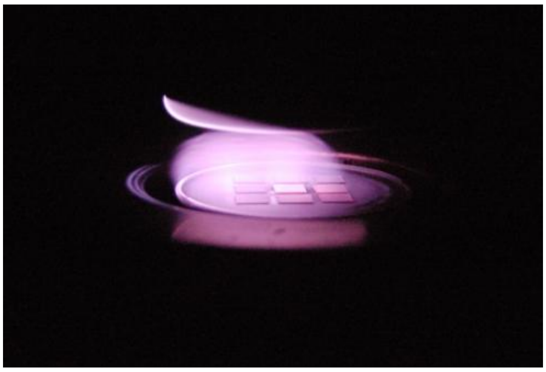


Fig. 3. A photograph of a discharge with an exposure of 1/1000 s in hydrogen medium with addition of 0.75% methane at a pressure of 75 mm Hg and a discharge current of 8.4 A at the grounded substrate holder with a diameter of 62 mm



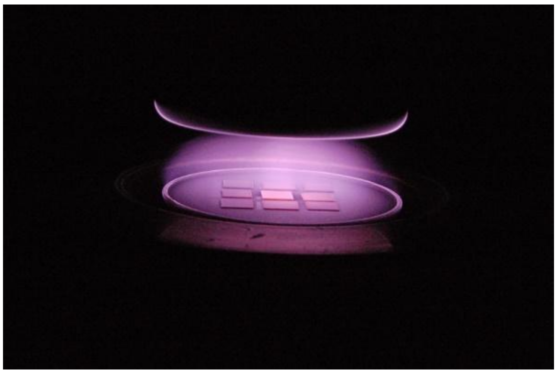


Fig. 4. A photograph of a discharge in a hydrogen medium with addition of 0.75% methane at a pressure of 60 mm Hg and a discharge current of 5.2 A in the case a grounded substrate holder with a diameter of 62 mm; exposures are the following: a – 1/1000 s; b – 1/100 s

At lower current values, the discharge covered only a part of the substrate holder, the top of the dome of the discharge was shifted from the center of the cathode, and the dome itself had an asymmetrical shape. Under the action of the magnetic field, such a dome-shaped discharge rotated around an axis passing through the cathode center with a frequency less than 100 Hz. This is confirmed by the photos of the discharge, which were taken with exposures 1/1000 and 1/100 s (Fig. 4).

CONCLUSIONS

The results of studies of the influence of the floating potential on substrate holders of various sizes on the shape and stability of the DC discharge burning in the presence of a magnetic field have been presented.

The conditions for stable burning of the discharge for a long time (up to 6 hours) were determined at a zero potential on the substrate holder.

It was shown that for the current range from 4 up to 8.5 A and for the pressure range from 75 up to 120 mm Hg on the samples located on the substrate holder it was possible to reach the temperatures from 1100 up to 1250 °C, which is quite sufficient for the nucleation and synthesis of high quality diamond coatings.

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ИСПОЛЬЗОВАНИЕ ТЛЕЮЩЕГО РАЗРЯДА ПОСТОЯННОГО ТОКА ДЛЯ СИНТЕЗА АЛМАЗНЫХ ПОКРЫТИЙ

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Приведены результаты исследований по влиянию величины плавающего потенциала на подложкодержателях различных размеров на форму и стабильность горения разряда постоянного тока при наличии магнитного поля. Определены условия стабильного горения разряда в течение длительного времени (до 6 ч) при нулевом потенциале на подложкодержателе. Показано, что в интервале токов 4…8,5 А и диапазоне давлений 75…120 мм рт. ст. на образцах, расположенных на подложкодержателе, можно достигать температур от 1100 до 1250 °C, что вполне достаточно для зарождения и синтеза алмазных покрытий.

ВИКОРИСТАННЯ ТЛІЮЧОГО РОЗРЯДУ ПОСТІЙНОГО СТРУМУ ДЛЯ СИНТЕЗУ АЛМАЗНИХ ПОКРИТТІВ

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Наведено результати досліджень щодо впливу величини плаваючого потенціалу на підкладкоутримачах різних розмірів на форму і стабільність горіння розряду постійного струму при наявності магнітного поля. Визначено умови стабільного горіння розряду в перебігу тривалого часу (до 6 год) при нульовому потенціалі на підкладкоутримачу. Показано, що в інтервалі струмів 4…8,5 А і діапазоні тисків 75…120 мм рт. ст. на зразках, розташованих на підкладкоутримачі можна досягати температур від 1100 до 1250 °C, що цілком достатньо для зародження і синтезу якісних алмазних покриттів.