APPLICATION OF PULSE POWER SUPPLY FOR DIAMOND COATINGS DEPOSITION IN GLOW DISCHARGE PLASMA

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Comparative studies of diamond coatings deposition have been carried out in the glow discharge plasma stabilized by a magnetic field in methane-hydrogen medium with using the DC or pulse power supply. It was shown that using of a pulsed power supply leads to an increase of the input power range at which the discharge is stable in comparison with DC one. In addition a significant increase of the samples temperature and the diamond coating growth rate were achieved at the same average power introduced into the discharge. The resulting diamond coating was of high quality and purity.

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INTRODUCTION

Due to the unique properties, diamond films can be widely used in various fields of science and technology [1-3]. One of the methods for obtaining diamond films is the deposition from the gas phase in a glow discharge plasma. To excite a glow discharge in carbon-containing gases, both direct current and pulsed supplies can be used. According to the literature, the use of pulsed power supplies for the excitation of microwave plasma leads both to an improvement in the quality of diamond coatings and to an increase in their growth rate without increasing the average power of the power supply [4, 5].

To date, the processes of diamond coatings deposition from the gas phase in DC glow discharge plasma in crossed E/H fields have been well studied and developed on the equipment available at the NSC KIPT [6-8].

This paper is devoted to the study of the diamond coatings deposition from the gas phase using a pulsed power supply.

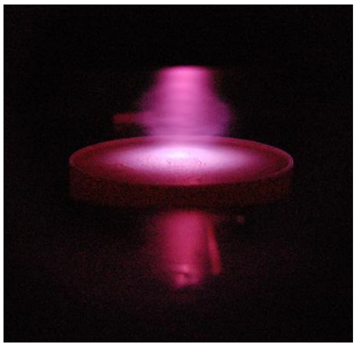
METHODS AND RESULTS

In these studies, a combined disk cathode Ø 115 mm and substrate holder Ø 52 mm were used. The temperature of the samples was measured by a "Promin" pyrometer with a "vanishing" filament and the coating thickness was determined by weight gain per unit surface area. Single crystal silicon wafers of 340 μm thickness pretreated with AFM 2/3 diamond powder served as coating substrates.

The use of a pulsed power supply on such equipment has shown that the stability of the discharge compared to a DC one is much higher. This made it possible to increase the average power supplied to the discharge gap without the risk of contraction of a glow discharge or its braking to an arc one (Fig. 1).

The use of a pulsed power supply leads to an increase of the samples temperature compared to a DC one. So, at a DC discharge of 3.85 kW, the temperature of the samples was 920…950 °C, while at a pulsed mode (frequency F = 50 kHz; duty cycle S = 1.11; average discharge current I = 5 A) and lower power (3.2 kW) the temperature of the samples increased to ~1030 °C. The growth rate of the diamond coating also increased by approximately two times compared with the DC mode (Fig. 2,a). The dependence of the diamond coatings growth rate on the temperature for different concentrations of methane in the hydrogen-methane gas mixture both in the DC discharge [9] and in a pulsed one have a similar appearance (Fig. 2,b).

a



b

Fig. 1. Photograph of the a) – pulsed and b) – DC supplying of the glow discharge. Hydrogen medium, pressure $8{\cdot}10^3$ Pa, I = 5 A

Fig. 3 shows the temperature of the samples and the growth rate of diamond coatings in a hydrogen-methane medium as a function of the pressure in the reaction chamber (a). The diamond coatings growth rate at a constant temperature of (1045 ± 10) °C increases with enlargement pressure of the gas mixture, despite the decrease in discharge power, which was regulated to maintain the temperature of the samples at the same level (b). An increase of the pulses frequency from 10 to 50 kHz, at a constant duty cycle and power, leads to an increase in the temperature of silicon substrates by 40...50 °C regardless of the methane concentration in the mixture.

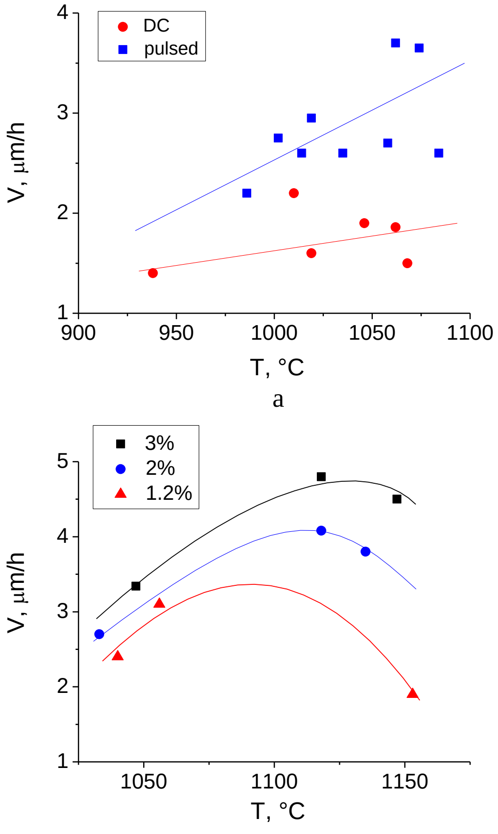


Fig. 2. Dependences a) – growth rate of a diamond coating in a glow discharge plasma under pulsed and DC mode (power W = 2.7 kW, methane concentration 2 %) and b) – diamond coatings growth rate for various methane concentrations. The pressure is P = 16·10^3 Pa, F = 25 kHz, S = 1.11

With a further increase of the frequency up to 100 kHz, the temperature of the substrate remains almost unchanged. The diamond coatings growth rate with an increase in the frequency of the pulses also increases, which is most likely associated with a decrease in the pause duration, during which the temperature drops and the recombination of active radicals occurs.

A change in the duty cycle from 1.11 to 1.43 with unchanged other discharge parameters (substrate temperature, pressure, gas medium composition, etc.) leads to an increase in substrate temperature by ~ 45 °C at a methane concentration of 0.4 % and by 160 °C at methane concentration 1 %.

With an increase in the duty cycle up to 1.33 (pause duration up to 10 μs) and methane concentration of 2 %, the stability of the deposition process is disturbed.

The discharge periodically and spontaneously passes from the center of the substrate holder to the rim and again to the center, which significantly changes the temperature mode of the samples (Fig. 4).

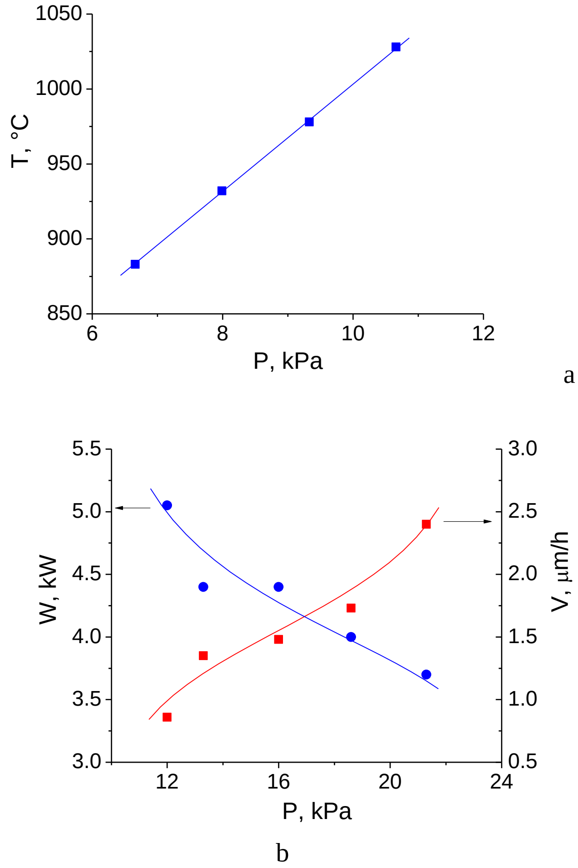
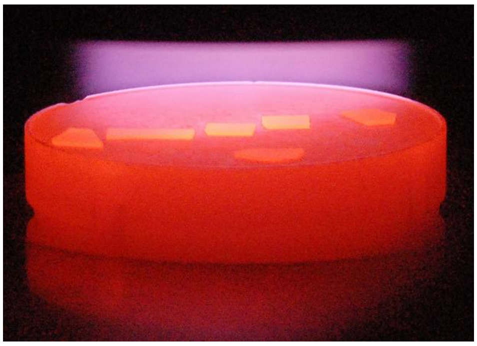


Fig. 3. Dependences a) – sample temperature (I = 5 A) and b) – diamond coatings growth rate (■) and average power (●) at T = (1040 ± 10) °C on the pressure of the hydrogen-methane medium. F = 25 kHz, S = 1.11, methane concentration 2 %

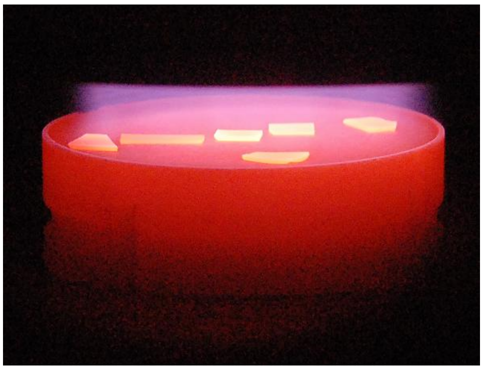
An increase in the pulse discharge pause has a limitation associated with the recombination of charge carriers, and, at a significant pressure in the chamber, the pulsed voltage may not reach the cathode-anode gap breakdown level and the deposition process is interrupted.

The phase composition of diamond films with a thickness of 18 μm on monocrystalline silicon substrates was determined by X-ray diffraction analysis on a DRON-3 diffractometer in filtered Cu-Kα radiation. Sections of one of the diffraction patterns are shown in Fig. 5.

The analysis showed that the structure of diamond films obtained using a pulsed power supply is similar to the structure of diamond films obtained by DC glow discharge [10]. A single phase is formed in the films - polycrystalline diamond (cubic Fm3m spatial group) with the lattice parameter a = (0.3569±0.0002) nm, which, within the error, coincides with the tabular value characteristic of natural diamond (a_{tabl} = 0.3567 nm).



a



b

Fig. 4. Photographs of a glow discharge which concentrated a) – on the rim of the substrate holder and b) – in the center of the substrate holder when power supply operates in pulsed mode (F = 25 kHz; S = 1.33; P = 21.3·10^3 Pa, methane concentration 2 %)

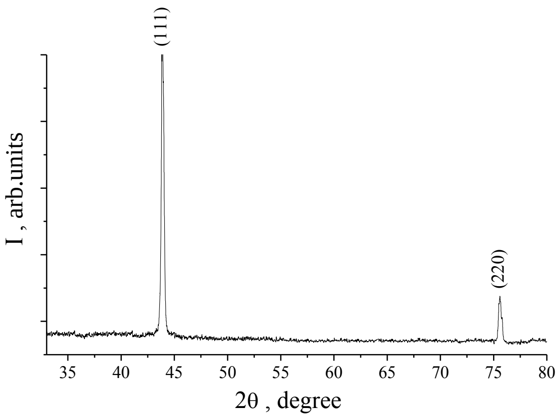


Fig. 5. The diffractogram of a diamond film with a thickness of 18 μm

The lines on the diffractograms are relatively narrow and each is represented by two lines of the $K\alpha_1$ and $K\alpha_2$ doublet (see Fig. 5). The selection of the doublet showed that the diamond lines are rather symmetrical. From the broadening of the (111) diamond diffraction line from the Selyakov – Scherrer ratio, taking into account the broadening of the standard, the average size of coherent scattering region (CSR) in films was determined. According to the results of calculations, the size of CSR in diamond films is 130 nm. The data obtained indicate the high quality and purity of the deposited diamond films.

CONCLUSIONS

Studies carried out using a pulsed power supply in the set up for diamond coatings deposition from the gas phase in a glow discharge plasma stabilized by a magnetic field showed that compared with a DC source:

• increases the range of input power at which the discharge is stable;

• a significant increase in the temperature of the samples and the growth rate of the diamond film are achieved at the same average power introduced into the discharge;

• the resulting diamond coating is of high quality and purity.

It should also be noted that in order to achieve maximum energy efficiency when using a pulsed power source, for each specific process of the diamond coating deposition, it is necessary to select both the parameters of the pulsed source and the conditions of the deposition.

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ПРИМЕНЕНИЕ ИМПУЛЬСНОГО ИСТОЧНИКА ПИТАНИЯ ДЛЯ ОСАЖДЕНИЯ АЛМАЗНЫХ ПОКРЫТИЙ В ПЛАЗМЕ ТЛЕЮЩЕГО РАЗРЯДА

К.И. Кошевой, Ю.Я. Волков, В.Е. Стрельницкий, Е.Н. Решетняк

Были проведены сравнительные исследования по применению как импульсного, так и постоянного источников тока для осаждения алмазных покрытий в плазме тлеющего разряда, стабилизированного магнитным полем в метановодородной среде. Показано, что использование импульсного источника питания приводит к увеличению диапазона входной мощности, при которой разряд устойчив по сравнению с разрядом постоянного тока. Кроме того, при той же средней мощности, вводимой в разряд, достигается значительное увеличение температуры образцов и скорости роста алмазного покрытия. Полученное алмазное покрытие отличается высоким качеством и чистотой.

ЗАСТОСУВАННЯ ІМПУЛЬСНОГО ДЖЕРЕЛА ЖИВЛЕННЯ ДЛЯ ОСАДЖЕННЯ АЛМАЗНИХ ПОКРИТТІВ У ПЛАЗМІ ТЛІЮЧОГО РОЗРЯДУ

К.І. Кошевой, Ю.Я. Волков, В.Є. Стрельницький, О.М. Решетняк

Були проведені порівняльні дослідження по застосуванню як імпульсного, так і постійного джерел струму для осадження алмазних покриттів у плазмі тліючого розряду, стабілізованого магнітним полем у метано-водневому середовищі. Показано, що використання імпульсного джерела живлення призводить до збільшення діапазону вхідної потужності, при якій розряд стійкий в порівнянні з розрядом постійного струму. Крім того, при тій же середній потужності, що вводиться в розряд, досягається значне збільшення температури зразків і швидкості росту алмазного покриття. Отримане алмазне покриття відрізняється високою якістю і чистотою.