Non-Destructive Diagnostics Diamond Coatings of Hard Alloy Cutters

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INTRODUCTION

When laboring with difficult-to-work materials, hard alloy tools with DC, which increase tool life and reduce process time, are actively used. MPCVD method, which allows obtaining homogeneous CVD diamond coatings, is effectively used for DC. The work [1] reports on the creation of the process of group deposition of diamond from the gas phase in the microwave plasma. Its main stages are shown in **Fig. 1**. The group method significantly increases the productivity of the process and improves the quality of the tool. However, when creating such a tool, one has to face a number of difficulties associated with the need to register the parameters of the technological process of DC, taking place at T ~ 800 °C. The analysis showed that optical methods of non-destructive testing are most suitable for these purposes.

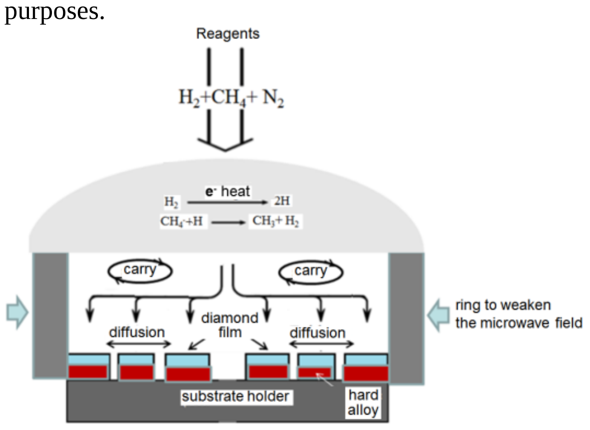
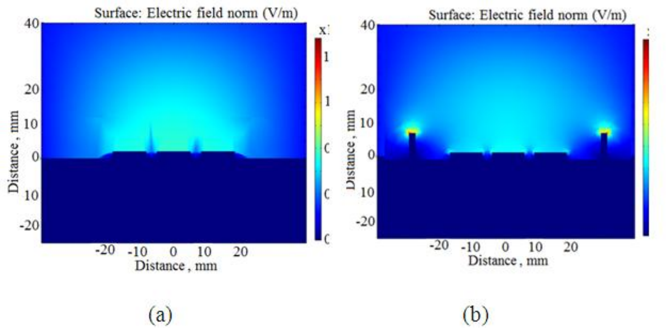


Fig. 1. *The main stages of the process of group deposition of diamond from the gas phase in the microwave plasma*

EXPERIMENTAL METHOD AND RESULTS

Experiments on the deposition of diamond coatings on hard alloy cutters (DCHAC) have shown that the most difficult problem for obtaining a high-quality coating is DC coating a tool with thin cutting edges protruding above the cutter body. The reason is extreme effects at the very edge of the wedge, which block diamond growth due to its overheating because of plasma concentration irregularities and thermal phenomena. Experimentally [1] and on a three-dimensional model of the distribution of the electromagnetic (EM) field, it has been found that the restriction of the central region of the basic conducting platform of the reactor by placed on it coaxially conducting ring, aligns the growth conditions of the film on a flat substrate and, in particular, the temperature distribution on its surface approaches a homogeneous one. Simulation of the EM field in a microwave reactor with a "ring" was performed using the computer program COMSOL Multiphysics using the finite element method. To simulate high-frequency radiation, the EM waves, frequency domain calculation module was used. Three-dimensional model EM field distribution of the installation in a simplified geometry corresponding to the ARDIS-100 microwave reactor was studied for the case of group growth on six DCHAC, located inside the encircling "ring" when the ring is shifted from 2 mm to 10 mm against the upper surface of the substrate holder.

It has been established that the shift of the “ring” does not make significant changes in the distribution of fields in the reactor outside the area of its location, and the operating mode of the reactor does not change at different positions of the “ring”. It is shown that all changes occur on a local scale near the growing surface.



(a) (b)

Fig. 2. *- Local distribution of the EM field in the reactor when the “ring” is shifted from 2 (a) to 10 mm (b).*

The principle of influence of the “ring” is EM field concentration control, which allows to manipulate the shape of the plasma “antinode” and to influence on ionization, due to exceeding the critical field strength (**Fig.2**). With the redistribution of fields due to the shift of the “ring”, not only the total intensity changes, but also the relative intensity of the central and external substrate. Thus, the shift of the “ring” makes it possible to level the growth conditions on the surface of various substrates, which is one of the requirements of uniform synthesis during group growth (**Fig.2**).

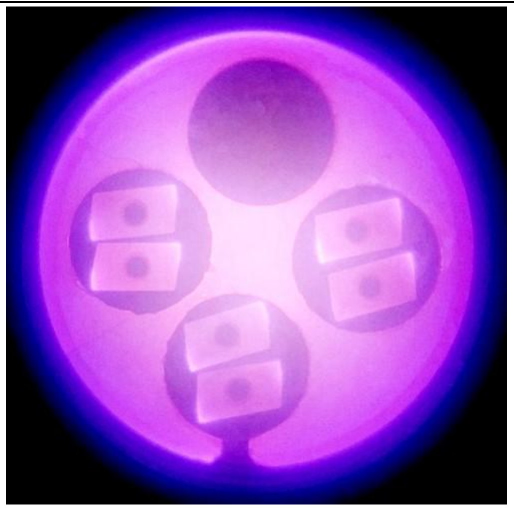
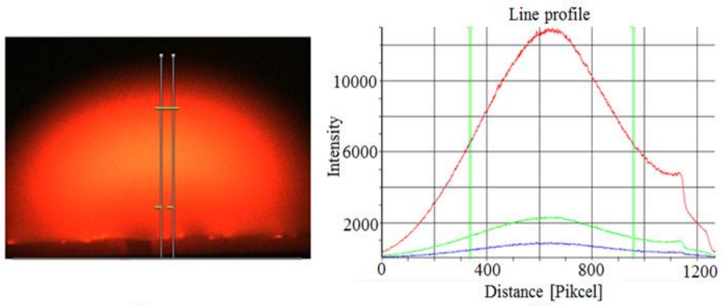


Fig.3. *Deposition of DC in microwave plasma was carried out on a group of six samples DCHAC. (photo through the upper window of the CVD reactor*)

To control the growth process, it was necessary to conduct a precise control of the group deposition parameters of DCHAC. It included determining the average power density of the discharge in the microwave reactor, measuring the temperature of the plasma gas using optical spectroscopy, measuring the temperature of DCHAC using an infrared pyrometer, monitoring the heating of the DCHAC in the microwave reactor by feedback with the indication of an infrared pyrometer. Experiments on deposition of DC in microwave plasma were carried out on a group of six samples DCHAC (CCGT120404-LH BU810). Including - on C-rhombic plates of 80° with a radius at the top r: 0.4 mm, length L: 12.9 mm and width S: 4.76 mm for the turning operation (**Fig.3**) and one-sided parallelogram with toroidal through-hole (ASGT 170504 PDFR – AJ alloy KS05F) for fastening in the TPS-17 body milling cutter during a milling operation.



a) b)

Fig. 4. *Photographs of plasma in the reactor through the H_α filter and the side window (a). Red brightness profile (b) in vertical section along the Z axis of brightness at the wavelength of the H_α line*

The average microwave power density P/V gives an idea of the intensity of plasma-chemical reactions and the concentration of active particles in the plasma, including such important ones as CH_3, C_2 and atomic H, which determine the growth rate and quality of the DC material [2]. In addition, the parameter of average power density makes it possible to more correctly compare various operating modes of the reactor and scale up the process of DC deposition during group growth, faster selecting the parameters of the discharge to preserve the basic characteristics of the plasma. The upper level of the H_α line (652 nm) is excited by electron impact, the intensity $I_{H\alpha}$ is proportional to the electron concentration n_e, therefore, we can say that where is a glow of the H_α line, there are electrons too, which means that there is also a plasma [3]. Estimating the size of the plasma by the intensity $I_{H\alpha}$ (photo through a narrowband filter with transmission band 653 ± 6 nm) allows us to more accurately calculate the size of the plasma **(Fig.4)** [4].

Preliminary calibration of the photo scale was carried out in order to correctly calculate the plasma size from the photo. Considering the axial symmetry of the chamber and plasma along the vertical axis of the reactor Z, passing through its center, the radial (dependence on R) distribution of the local emissivity $I_{H\alpha}(R)$ over the measured $I_{H\alpha}$ profile (X) was obtained, the Abel inversion procedure was applied to this $I_{H\alpha}(X)$ profile. [5, 6]. The cross section of the photographs determined the width at half-height $\Delta Z_{(0.5)}$.

Due to the axial symmetry of the reactor chamber and the discharge in the Z-direction, the spatial radiation profile $I_{H\alpha}(x, y, z)$ can be described by a 3-dimensional Gaussian function ($Y_{0.5} = X_{0.5}$)

$$I_{H\alpha} = I_0 \times \exp[-(x^2+y^2)/2s_x^2] \times \exp[-z^2/2s_z^2] \qquad (1)$$

where $s_x = X_{0.5}/[2\sqrt{(2\ln 2)}]$, $s_z = Z_{0.5}/[2\sqrt{(2\ln 2)}]$, and $I_0 = I_0(0,0,0)$ intensity at the center of the plasma cloud. The shape of the plasma cloud is not spherical, so the plasma volume can be calculated using the ellipsoid formula.

$$V = (\pi/6) \times (\Delta X_{1/e})^2 \times (\Delta Z_{1/e}) \qquad (2)$$

where $\Delta Y_{1/e} = \Delta X_{1/e} = \Delta X_{0.5}/\sqrt{(\ln 2)} \approx 1.44 \times \Delta X_{0.5}$ and $\Delta Z_{1/e} = \Delta Z_{0.5}/\sqrt{(\ln 2)} \approx 1.44 \times \Delta Z_{0.5}$ are the width at which the brightness of plasma along the Y, X and Z axes decreases to the level of I_0/e (i.e., decreases by e times, where e = 2.718) as compared with the maximum of I_0.

Photo images are recorded as digitized RAW data obtained from a matrix with minimal processing, which ensures the preservation of linearity in intensity on the image and allow the measurement of X and Z profiles for the radiation of H_α line. The volume of the plasma ellipsoid was determined by intensity level 1/e = 1/2,718.

Taking into account: $X_e = 2\sqrt{2} \times X_{0.5}/[2\sqrt{(2\ln 2)}] = X_{0.5}/\sqrt{(\ln 2)}$, the plasma volume was calculated:

$$V = d_{ex}^2 \times d_{ez} \times \pi/6 = d_{0.5x}^2 \times d_{0.5z} \times \pi/(6 \times (\ln 2)^{3/2})\ [\mathrm{cm}^3] \qquad (3)$$

Following the photo and corresponding sections (as on **Fig.4**) the width were determined: $d_{0.5x}$ =3.35cm; $d_{0.5z}$ = 1.68cm and the plasma volume: 17.1 cm^3 . Therefore the average microwave power density, taking into account the power P = 2.9 kW for this group growth mode:

$$P/V \approx 170\ \mathrm{W/cm^3} \qquad (4)$$

The substrate temperature was measured in the middle of the sample with a dual-band IR pyrometer (“Williamson”, model PRO-81-35-C) and was taken as the initial one. The pyrometer recorded the temperature of the edge of DC (spot Ø =2 mm). It is known that the temperature measured by the pyrometer strongly depends on the emission coefficient of the sample (for single-range mode) or on the ratio of radiation coefficients in two working ranges (for dual-range mode). Therefore, the correct temperature measurement was performed on the lateral surface of DCHAC samples, specially coated with DC having a high absorption coefficient and high emissivity in a wide range of wavelengths. In this case, the ratio of radiation coefficients in the two working ranges of the pyrometers approached unit. The emissivity of DC deposited on a hard alloy substrate was tested in a vacuum furnace equipped with a calibrated Pt/Pt + 10 % Rh thermocouple (S-type) with an accuracy of ± 5°C. The working vacuum in the furnace was 2×10^{-5} Torr, which excluded chemical interaction of oxygen with the DC and kept its emission properties unchanged when heated to 1200 °C. In parallel, the temperature measurements of DCHAC samples were carried out with an infrared pyrometer through a quartz window in a vacuum furnace and compared with the readings of a Pt/Pt + 10% Rh thermocouple. It was found that the ratio of the emission coefficients of DC on the substrate in two working ranges of the Williamson pyrometer is 0.98. This allows measuring the temperature of the cutters in the CVD reactor by the optical method, using an IR pyrometer with an accuracy of ± 30 °C.

CONCLUSIONS

1. The displacement of the “ring” can lead to extreme impacts at the very edge of the wedge and solve the problem of its overheating during the diamond film growth.
2. Photographing the plasma from the side through the H_α-filter allows one to reliably estimate the average MW power density and control the discharge conditions.
3. The correct measurement of the temperature of the cutters with a two-range pyrometer using the pre-measured value of the ratio of emission coefficients allows to accurately reproducing the growth conditions of the diamond film.
4. Thanks to optical methods of non-destructive testing, for the first time it was possible to implement a method of obtaining high-quality DC on hard alloy tool [1].

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