



# Growth of diamond structures using high speed gas jet deposition activated in heated tungsten channels

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## ABSTRACT

A method for obtaining diamond films using gas jet deposition with activation on extended surfaces made of tungsten heated up to high temperatures is considered in the article. The original reactor had been designed for activation of carbonaceous gas mixtures. A distinguishing feature of this method is to provide an extended interaction of hydrogen, methane, and its fragments with a heated tungsten surface. It is proposed to implement the increase of catalytic thermal dissociation of hydrogen molecules by providing multiple collisions during passage through the heated tungsten channels. In the researches carried out the effect of thermo-gas-dynamic parameters on resulting carbon structures had been studied. During the film deposition on the molybdenum surface, a strong dependence of the film morphology on the temperature of the tungsten surface was observed in the range of 2000–2150 °C. As a result of conducted research next objectives has obtained: the optimal substrate temperature, the value of the pressure in the reaction zone, ways of minimizing the carburization of the activation surfaces were found.

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## 1. Introduction

At the moment, diamond is one of the most promising functional materials rather than a precious jewel. This material is in high demand in various fields of industry, including production of electronic devices, cutting and drilling tools, optics, high-pressure engineering, etc. [1–4].

If diamond is obtained by means of chemical vapor deposition (CVD) [5–8], its mineral properties are retained and its extreme properties can be also used, whereas the overall cost of the material is decreased [9]. In terms of types of activation precursor gases necessary for growth of diamond structures, the CVD technology is implemented by several different methods: activation in radio frequency [10] and microwave plasmas [11], in the arc discharge plasma [12], in a flame [13] and thermocatalytic activation on hot wires [14] made of refractory metals (W, Mo, Ta, or Re). All these methods have certain advantages and, correspondingly, disadvantages.

The main goal of activation is to decompose the gases to active fragments. The kinetic features of the formation and transportation of particles are different in plasma [15] and thermocatalytic methods [16] of activation. In electric arc plasmatrons, activation occurs in the electric arc column and particles are mainly set into motion due to a pressure gradient. In the case of deposition on hot wires, activation occurs due to thermocatalytic reactions, and particles become transported to the substrate surface owing to thermodiffusion process. It is known that

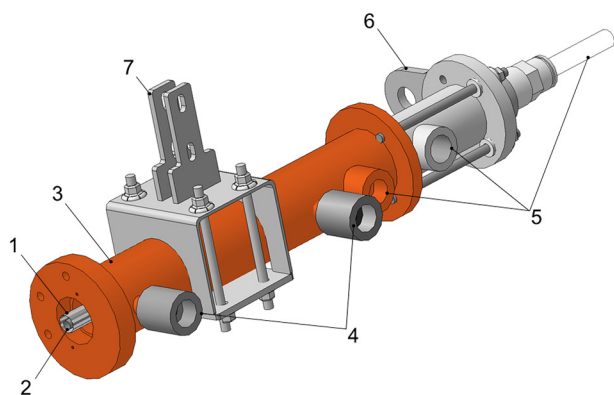
atomic hydrogen plays a key role in obtaining diamond structures by the CVD method [17,18], because it assists in methane decomposition to methyl CH<sub>3</sub>, which serves as a source of diamond growth, activates the diamond surface, prevents graphitization of the substrate surface, and saturates broken bonds on the diamond surface [19]. In CVD methods with thermal activation on hot wires, there is a problem to ensure a sufficiently high concentration of atomic hydrogen in the H<sub>2</sub> flow [20] and also to prevent carburization of activating surfaces.

The paper presents the development one of CVD methods, namely hot – filament or catalytic CVD. An important feature of this approach is using thermocatalytic activation in the case of interaction with extended heated metal surfaces. This process can be organized both under the condition of a frozen composition of the mixture after gas decomposition in the reactor and in a completely equilibrium state. The main goal of optimization of diamond film deposition is to identify parameters that produce the most important effects on the growth process. These are the pattern of injection of the mixture into the regions of activation and heating, the temperature of the activation surface, the gas flow intensity, the pressure in the test section, the substrate material, the methane concentration in the H<sub>2</sub> + CH<sub>4</sub> mixture, and the substrate temperature. A novel source of the activated gas mixture was used in the present study, which ensured a possibility of changing test parameters within wide limits.

Specific features of hydrogen dissociation in channels was numerically studied in [21,22], where a possibility of reaching a high degree of hydrogen dissociation at the channel exit owing to multiple collisions with the surface was demonstrated. This fact gave grounds for using a

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**Fig. 1.** Sketch of the reactor: 1 – external tungsten tube, 2 – internal tungsten tube, 3 – copper shroud, 4 – channels for water cooling, 5 – channels for injection of gases, 6 – current lead, 7 – holder.

special reactor whose design allows one to combine the advantages of different activation methods, thus, expanding the parameter adjustment options.

## 2. Reactor design and experimental study

The thermocatalytic activator used in the present study is a reactor schematically shown in Figs. 1 and 2. It contains one or two coaxially aligned tungsten cylinders, which form channels for gas flows. To reduce the power loss, the tungsten cylinders are located in a water-cooled copper shroud thoroughly polished from inside. Argon, which serves as a thermally insulating gas, is fed into the channel between the shroud and the activation cylinder. The activation surfaces can be heated up to 2500 °C; therefore, significant losses of input power are caused by radiation. As the synthesis of diamond structures lasts for hours, it is important to optimize the energy expenses. A detailed description of the reactor and the setup for diamond film deposition can be found in [23].

The test section options with the use of joint (a) and separate (b) injection of gases are shown in Fig. 2. The reactor includes one (ø6 mm) or two (ø3 mm and ø6 mm) axisymmetric cylindrical channels. The channel walls are made of tungsten foil 0.03 mm thick. The external tube is heated in a resistive manner, whereas the internal tube is heated by means of radiation.

**Table 1**  
Typical deposition conditions.

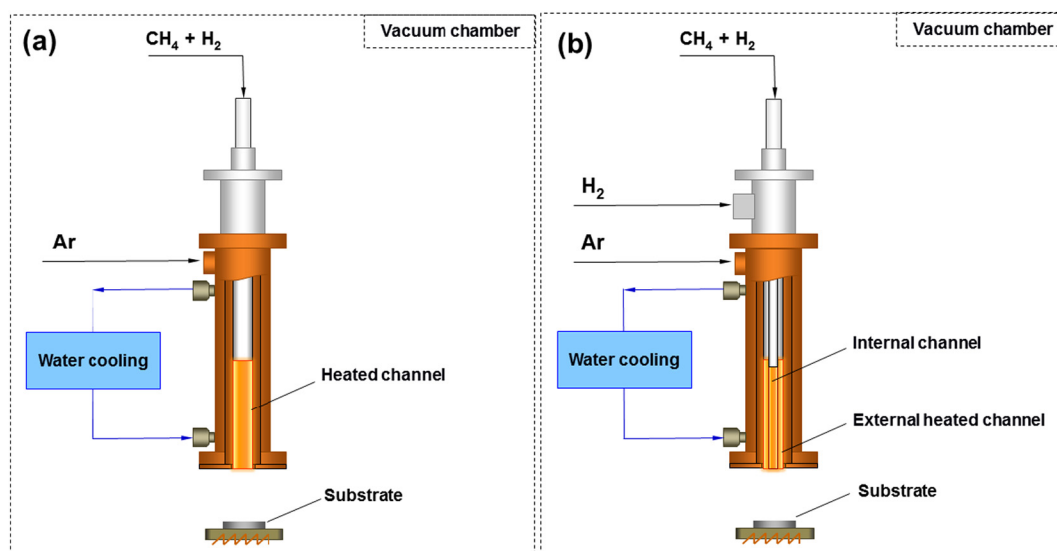
Parameter	Value
Activator temperature	1800–2200 °C
Substrate temperature	700–1000 °C
Methane concentration	0.5–2%
Pressure	0.2–50 Torr
Gas flow rate in the annular channel	375–1500 sccm
Gas flow rate in the internal channel	1500 sccm

Previous studies showed that one of the governing factors of the diamond deposition rate is the chemical reaction of hydrogen atoms with carbon-containing fragments [24]. Therefore, to ensure more effective interaction of hydrogen atoms with carbon atoms, the authors studied in particular the effects of joint and separate injection of gases through extended heated surfaces.

If there is only one channel, it is used for injection of a mixture of hydrogen and a carbon-containing gas (methane). Activation products are transported to the substrate, where the diamond film is grown. If two channels are used (in the system with separate injection of gases), then a hydrocarbon gas diluted by hydrogen is injected into the internal channel, and the hydrogen flow is fed through the external (annular) channel. The injection system with two channels is used to protect the main activation surface against carbide formation, which is induced by interaction of molecules of methane and other carbon-containing species with heated tungsten.

The substrates are molybdenum disks (ø25, thickness 2 mm) mounted at the adjusted distance from the nozzle cross section on a temperature-controlled substrate holder. Located at a distance of 10 mm from activator nozzle, the substrate is heated up to 500 °C at the peak temperature of the tungsten cylinder. This allows to avoid additional water cooling. To achieve the operating temperatures of the substrate, a supporting substrate heater is used.

To obtain continuous films consisting of diamond microcrystals, it is necessary to ensure a high degree of nucleation on the substrate. Carbide-forming materials, such as Mo, W, Ti, and Si, are preferable for growth of diamond structures. Molybdenum is the most suitable materials because it has a high melting temperature and a low coefficient of thermal expansion; it weakly dissolves carbon and actively participates in carbide-formation reactions, interacting with carbon at high temperatures. Molybdenum also has the highest degree of nucleation of the order of  $10^8 \text{ cm}^{-2}$ , in contrast, e.g., to silicon with  $n = 10^5 \text{ cm}^{-2}$ ) [25, 26], even without preliminary preparation. The carbide layer prevents



**Fig. 2.** Reactor systems: (a) joint injection; (b) separate injection of activated gases.

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