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X-Rays Response of Diamond Detectors Constructed Using Diamond Layers Produced by Low Power Microwave Chemical Vapor Deposition Reactor

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The low power reactor for microwave chemical vapor deposition process is described. The rotating Mo holder of 12 mm diameter and 6 mm height with the diamond substrate was heated by 2.45 GHz microwaves to temperature about 800 °C in the range of (1.5–7)% CH_4/H_2 mixture to create plasma at pressure 70 Tr. Stabilization of the holder temperature was performed by optical observation of radiation from the holder followed by adjusting of the magnetron power. Diamond detectors are produced using microwave chemical vapor deposition process grown on single crystal diamond high pressure high temperature Sumimoto substrates, [100] oriented. The response of diamond detectors for X-rays has been measured in the current mode using medical X-rays tube. The linear response of the diamond detector current versus X-ray tube current (dose) is presented.

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1. Introduction

Diamond detectors for X-ray dosimetry are produced in different ways: using high-quality, single crystal chemical vapor deposition (CVD) diamond layers grown on high pressure high temperature (HPHT) [100] oriented substrates [1–3] or by application of single crystal CVD layers [4, 5]. For X-ray dosimetry applications of the diamond polycrystal is also used [6]. In all above works the microwave CVD (MWCVD) process was used. However, thin polycrystalline diamond films (10/20 μm) for diamond detectors are deposited by hot filament vapor deposition technique on metallic (tungsten) [5].

The aim of this paper is to present our low power MWCVD reactor and investigation of diamond detectors produced using this reactor [7–10].

2. Low power microwave chemical vapor deposition reactor

The MWCVD diamond is created in the following conditions: gas composition few percentage of hydrocarbons like CH_4, C_4H_{10} or C_6H_{14} in H_2, temperature about 800 °C and pressure 70 Tr. In these conditions the gases are ionized into active radicals and diamond is created on the substrate (Si, tungsten, molybdenum, diamond substrate and other materials). The diagram of the reactor is presented in Fig. 1.

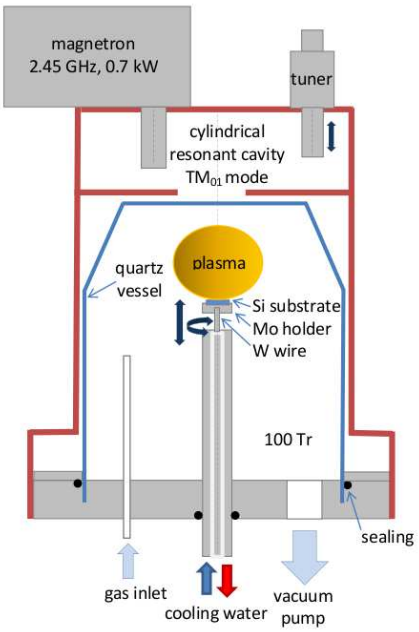

Fig. 1. MWCVD reactor.

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The cylindrical resonant cavity with web camera observing the Mo holder for temperature measurement is presented in Fig. 2. The visible vertical rift in the resonant cavity enables radiation observation from plasma and Mo holder by eye.

Fig. 2. Cylindrical resonant cavity of MWCVD reactor with Web Camera.

Fig. 3. Bottom of the chamber with Mo holder.

A photo of the Mo holder on the bottom of the chamber (quartz vessel and resonant cavity is removed) is presented in Fig. 3.

The Mo holder is supported by 1 mm diameter and 10 mm length tungsten wire to decrease the heat transfer from the warmed Mo holder to the bottom of the chamber. We used pulsed magnetron operating at 2.45 GHz frequency with maximal power 0.7 kW. The substrate is put into rotating Mo holder of diameter 12 mm and 6 mm high. In practical application the magnetron power of 300 W is sufficient to heat the Mo holder to temperature 800 °C. The holder is covered by quartz vessel of diameter 97 mm and 135 mm height. The vessel filled by methane and hydrogen mixture at pressure about 70 Tr is located by the cylindrical resonant cavity operating at TM_{01} mode. The typical flows at pressure 70 Tr of CH_4 and H_2 are 0.4 cm^3/s and 5 cm^3/s, respectively. The speed of diamond layer growth is about 0.1 μm/h.

Since the flow of gases is very low we used fermentation locks as flow-meters (which are commonly applied for registration carbon oxide in vine fermentation process) for measurement of H_2 and CH_4 flows by counting bubbles with fermentation locks filled by vacuum pump oil, see Fig. 4.

Fig. 4. Flow-meters based on the fermentation lock for methane (left) and for hydrogen (right).

The number of bubbles is counted by transoptors and sent to the computer system. Gas flow is proportional to the bubbles counting rate. Since the flow of hydrogen is about 100 times bigger than for methane therefore diameter of fermentation locks for hydrogen and methane is 35 mm and 8 mm, respectively. To ensure precise measurements and calibration of flow-meters with gas bubbles we have used mechanical bellows flow-meters.

For gas pressure measurement we have used manometer with pressure-frequency converter based on the RC generator whose capacitance C changes its value as a function of the gas pressure.

Measurement of temperature is achieved by radiation registration from the hot Mo holder. For this purposes we have used plasma generation by pulsed magnetron supplied with AC frequency 50 Hz. The plasma is generated when the 2.45 GHz microwaves are emitted. For parts of the 50 Hz pulse when microwaves are stopped the thermal radiation is generated only by hot Mo holder without light radiation from the plasma. Using principles of web camera working where pixels are read in series rows by rows it is possible to register of pictures in time regions where plasma radiation is stopped and light is emitted only by hot holder (the method was proposed and elaborated by Tarasiuk). For proper temperature measurements the web camera is calibrated by the thermocouple.

Magnetron power regulation is adjusted by AC power supply in the range about 5 V with 7 bits precision, see Fig. 5.

Seven inductances with inductance proportional to 2^n are connected in series with magnetron and

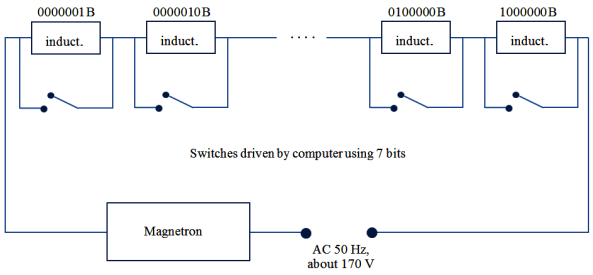

Fig. 5. Magnetron power regulation by adjusting of AC power supply in the range 5 V with 7 bits precision.

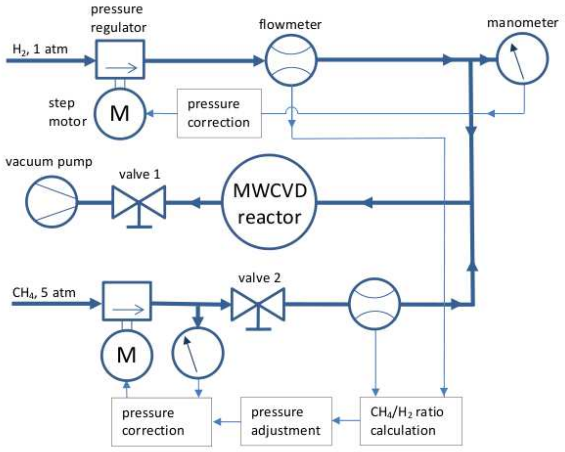

Fig. 6. Stabilization of H_2 pressure and CH_4/H_2 gases ratio.

50 Hz AC supply with voltage about 170 V. Each inductance can be closed or opened by relay driven by computer, depending on the Mo holder temperature needed. Stabilization of gasses pressure and gasses ratio in MWCVD reactor is presented in Fig. 6.

The pressure of pure hydrogen is decreased to about 70 Tr by pressure regulator controlled by manometer. The control unit named "pressure correction" stabilizes the assumed pressure of hydrogen by adjusting position of the pressure regulator using the step motor. Then hydrogen enters to the MWCVD reactor. The valve 1 before vacuum pump determines flow of the hydrogen which was selected to about 5 cm^3/s. The input methane pressure was decreased to about 150 Tr by pressure regulator and its flow is limited by the valve 2. The CH_4/H_2 ratio has been calculated using data from both flow-meters. Using these data the unit named "pressure adjustment" select a new pressure of methane which is executed by unit "pressure correction".

3. Results

The best diamonds detectors are produced using diamond single-crystals. Such crystals can be obtained by growing of diamond on single-crystal diamond HPHT substrates. For growing of diamond crystals we used MWCVD process performed on our MWCVD reactor [7–10]. The synthetic single-crystal diamonds grown on the silicon substrate are randomly oriented due to the heteroepitaxy process, see Fig. 7.

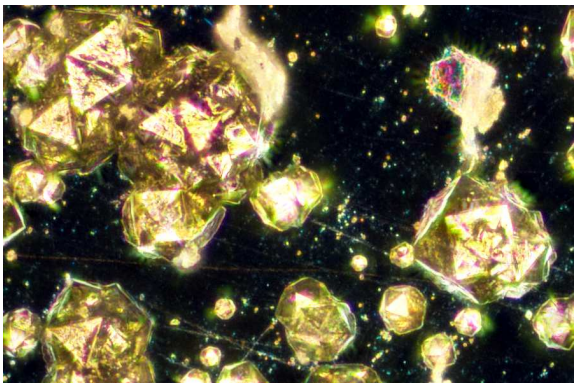

Fig. 7. Single-crystal diamonds are grown during 8 days on the silicon substrate using 2% concentration of butane in hydrogen at temperature about 800 °C and pressure 70 torr. Randomly oriented diamond crystals of diameter about 0.2 mm are obtained in the heteroepitaxy process.

The MWCVD homoepitaxy process is performed on single-crystal Sumimoto $3 \times 3 \times 0.3$ mm^3 diamond substrate HPHT crystal, [100] oriented at temperature about 800 °C and 1.4% of butane/hydrogen concentration with pressure 69 Tr. After 11 days of process the single-crystal diamonds, oriented with [100] direction in shape of square pyramids are obtained, see Fig. 8.

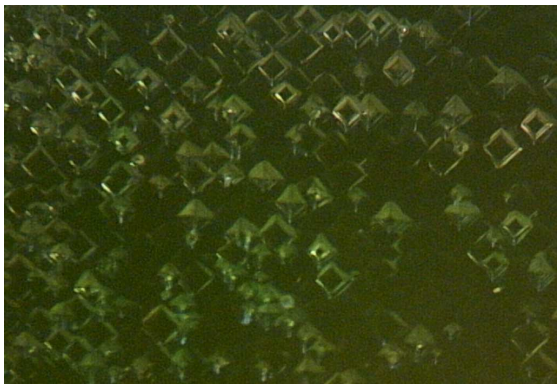

Fig. 8. Diamond [100] oriented single-crystals, grown by MWCVD in homoepitaxy process on [100] diamond Sumimoto substrate are obtained. Parallel oriented diamond square pyramids (some of them are without peaks) are visible.

After increasing concentration from 1.4% to 2.6% of butane/hydrogen with MWCD process lasted 4 days, the diamond layer of thickness 50 μm was obtained, see Fig. 9.

Similar results were obtained with compositions of methane/hydrogen and hexane/hydrogen.

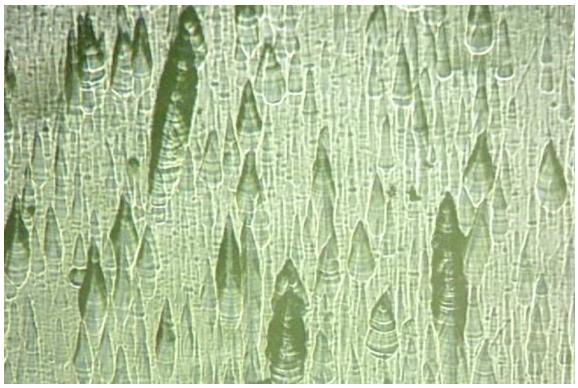

Fig. 9. Single-crystal diamond layer of thickness 50 μm grown during 4 days by MWCVD homoepitaxy process on the 300 μm, [100] HPHT diamond single-crystal Sumimoto substrate.

4. Test of diamond detectors

The diamond detector has been prepared using 50 μm CVD layer grown on single-crystal Sumimoto $3 \times 3 \times 0.3$ mm^3 diamond substrate HPHT crystal, [100] oriented using CH_4/H_2 mixture. For comparison we have build diamond detector using CVD diamond single crystal $2.6 \times 2.6 \times 0.3$ mm^3 [100] oriented, obtained from e6 company. The circular Al contacts of thickness about 2000 Å were evaporated on both sides of the diamond CVD/HPHT and CVD plates. The thin wires are connected to the evaporated Al contacts by conductive thermally stabilized epoxy glue, see Fig. 10.

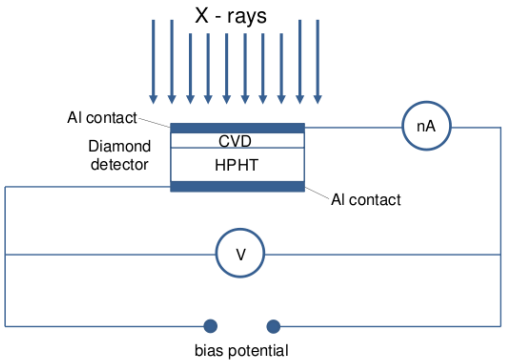

Fig. 10. Tests of diamond detectors in the current mode.

Diamond detectors have been tested using X-rays from medical Philips X-ray Tube Model 9890 000 86 101 with permanent filtration 2.6 mm Al at Medical University of Warsaw, Warsaw, Poland. The X-ray tube operated at 40 kV. The dose strength at 1 mm^2 of diamond detector was 2 Gy/s for 10 mA of X-ray tube current. Measurements have been performed, in the current mode, see Fig. 10.

Response of diamond CVD/HPHT detector on X-ray tube operated at 40 kV is shown in Fig. 11. The detector current is linear as a function on the X-ray tube current (proportional to dose strength) for positive and negative detector bias polarization of about ±200 V.

Similar results are obtained for diamond detector of diameter 1.5 mm made from CVD diamond single crystal obtained from e6 company, see Fig. 12.

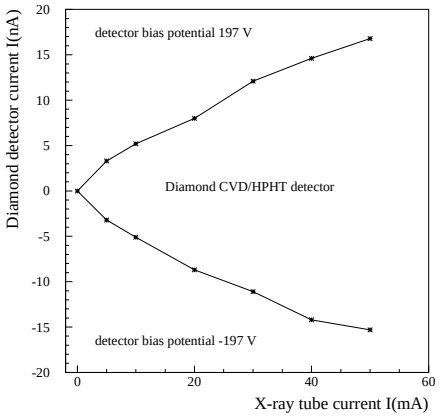

Fig. 11. Response of diamond CVD/HPHT detector for X-ray fluence generated by X-ray tube current.

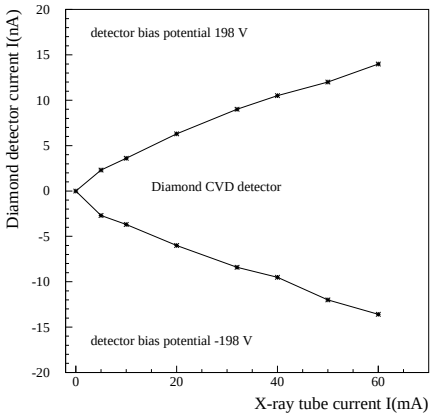

Fig. 12. Response of diamond CVD detector for X-ray fluence generated by X-ray tube current.

Almost symmetrical response of diamond detector for positive and negative bias polarizations confirms that diamond detectors are produced from isolating material.

Obtained linear response of diamond detectors versus X-ray dose strength gives possibility to use such detectors in X-rays dosimetric applications.

Acknowledgments

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