



Bias enhanced nucleation of diamond thin films in a modified HFCVD reactor

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A B S T R A C T

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In this study we investigated the nucleation of synthetic diamond thin films on Si substrates by double bias enhanced Hot Filament Chemical Vapour Deposition (HFCVD) method. First, we investigated the influence of the bias voltage and secondly the influence of the nucleation time under different bias voltages. The bias voltage was varied from -120 V up to -220 V as well as the nucleation time was changed from 30 up to 120 min in order to obtain the optimized nucleation conditions for following growth of continuous diamond layer. Samples were analyzed by Scanning Electron Microscopy (SEM) and Raman Spectroscopy. SEM was used for determination of cluster sizes and their distribution on the Si surface, while Raman Spectroscopy for determination and analysis of carbon phases.

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1. Introduction

Synthetic diamond thin films have remarkable properties comparable with natural diamond [1] and have been used for long time as hard coatings or protective layers [2]. Nowadays they have great interest for high-power and high-frequency electronics, where commonly used semiconductors have their limitations. However, in electronics there are higher demands on the quality of the material, i.e. the number of defects and impurities. This property is not realised in the case of diamond, which always contains many defects depending on the growth method.

The most important step in synthetic diamond growth process, which strongly influences the quality, is the so-called nucleation. We can imagine it as a pretreatment of the Si substrate surface before the diamond deposition. It is necessary because diamond has low intrinsic nucleation density on Si. Beside methods like scratching [3], ultrasonic treatment with diamond powder, ion bombardment [4] and various etching methods [5], the bias enhanced nucleation (BEN) is an efficient mechanism to increase the nucleation density [6]. Moreover, this method provides oriented nucleation of diamond on Si substrates.

2. Experiment

Experiments were carried out in the modified HFCVD apparatus described previously [6]. This apparatus is characterized by its specific double bias voltage set up (Fig. 1). There is a possibility to

apply a bias voltage with various polarities between the substrate and tungsten filaments and between the tungsten filaments and grid, respectively. In the nucleation process, a negative bias voltage is applied to the substrate and positive voltage to the grid placed above the tungsten filaments. Stable plasma can be generated between this grid and the filaments. Ions produced in the plasma are driven to the substrate by a negative substrate bias voltage.

The gas phase was a mixture of 2% CH₄ in H₂, the total pressure in the reactor was 3000 Pa and flow rates were 6:300 sccm. Gases were activated by 5 tungsten filaments 0.7 mm thick, 120 mm long, heated to 2100 °C. The substrate temperature was about 700 °C. During the experiments two parameters were changed: the nucleation process time which varied from 30 min up to 120 min and the bias voltage applied to the substrate in respect of the tungsten filaments from -120 V up to -220 V. In this study first we investigated the influence of process time at constant bias voltage of -170 V, then the influence of bias voltage at constant process time of 120 min.

The surfaces of samples were analyzed by a Leo 1550 Scanning Electron Microscopy (SEM). Raman spectra were used only for determination of carbon phases onto the surface and were performed using an ISA Dilor-Jobin Yvon-Spex Labram confocal system with 632.8 nm radiation from a He–Ne laser.

3. Results and discussion

3.1. Effect of process time

The investigation of the influence of process time was carried out at constant bias voltage of -170 V. SEM micrographs in Fig. 2

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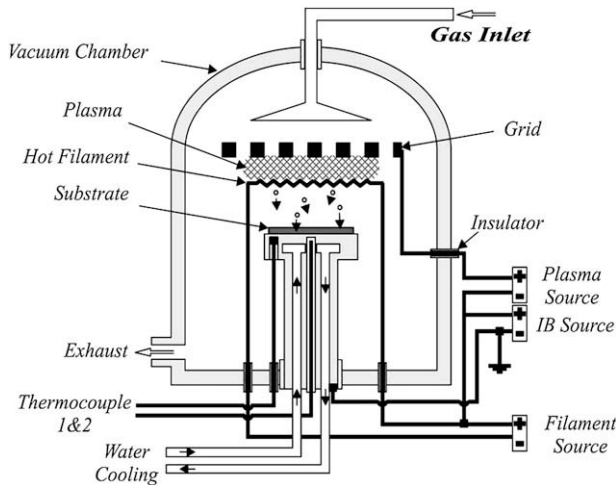


Fig. 1. Schematic view of our modified double bias enhanced HFCVD apparatus.

show the morphology of the sample surfaces with the same magnification of 100 000 \times . Sample A was treated for 30 min, sample B for 60 min and sample C for 120 min.

For the sample with shortest nucleation time no diamond clusters were observed, only very small white luminescence points (under 10–20 nm). With increasing process time more and more diamond clusters appear on the Si surface. At the process time of 60 min we observed a relatively homogeneous diamond cluster array with high density of $\sim 10^8$ cm $^{-2}$. The sizes of diamond clusters are in the range of 50–100 nm in diameter. As process time is increased, the diameters are enlarged to 100–130 nm, and the cluster density decreases to $\sim 10^7$ cm $^{-2}$.

Raman spectra in Fig. 3 show carbon phases on the surface. There are three bands: at 950 cm $^{-1}$, ~ 1332 cm $^{-1}$ and ~ 1608 cm $^{-1}$ approximately. The first band at 950 cm $^{-1}$ is the second order Raman peak of the Si substrate [7], the second band is the so-called D (or “disordered”) -band and the third band is known as G- or “graphite-band”. Wide D- and G-bands are attributed to sp 2 -bonded carbon. The G-band is characteristic for microcrystalline and the D-band for disordered graphite [7–9]. The D-band becomes active when small graphite crystallites are present. A sharp peak at 1332 cm $^{-1}$ which is typical for diamond (it is the so-called D-peak and in our case not observed,) is expected for crystalline diamond and indicates the sp 3 carbon bonds. But due to the fact that with visible laser excitation wavelength (632 nm) the sensitivity for this Raman line is 50 times lower than the one for graphite, no conclusion on the presence of any sp 3 bonds can be drawn from the measured spectra [12]. For more precise characterization of sp 3 and sp 2 bonds UV excitation is necessary [13,14]. By UV excitation it is possible to obtain the so-called T-peak at 1100 cm $^{-1}$, which more precisely characterized the sp 3 bonds.

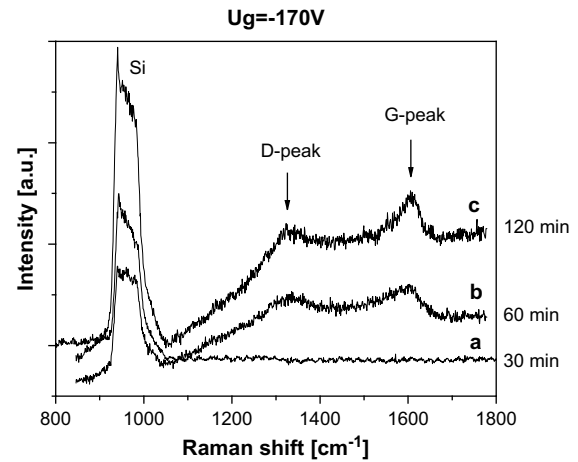


Fig. 3. Raman spectra of samples prepared at the same bias voltage and different process time.

From our Raman spectra we compared only the ratio of the G- and D-bands. The second order Si-peak was used as a reference that spectra were not shifted. From the spectra it is obvious that amorphous carbon or graphite phases on the sample surfaces are dominant because of the higher intensity of the G-peak. The nucleation was carried out using a gas mixture of 2% CH $_4$ in H $_2$, which is the typical ratio for the nucleation step [9–11]. Higher content of CH $_4$ allows creation of more sp 2 bonds, but more H $_2$ content in the mixture acts as etching medium and removes sp 2 bonds at the expense of sp 3 bonds. At the largest nucleation time the sharpest G-peak appeared and also the wide D-band became sharper. Although the D-band is related to the increase in sp 3 carbon bonds, the simultaneous increasing G-peak indicated that diamond clusters are created inside graphitic clusters which are created first on the substrate. Similar results were published by Liao et al., where they observed that most of the diamond crystallites are embedded in a matrix of amorphous carbon and only a few are directly grown on Si [12].

3.2. Effect of bias voltage

The influence of bias voltage was investigated at a constant nucleation process time of 120 min. In case of sample A a bias of -120 V, for sample B -170 V and for the last sample C -220 V was applied. SEM micrographs in Fig. 4 show the morphology of the sample surfaces after the process at the same magnification of 100 000 \times . The results are similar to the first case where we investigated the influence of process time. We can conclude that 120 min nucleation time at -120 V negative bias voltage is not enough, the result is very poor because no diamond clusters were

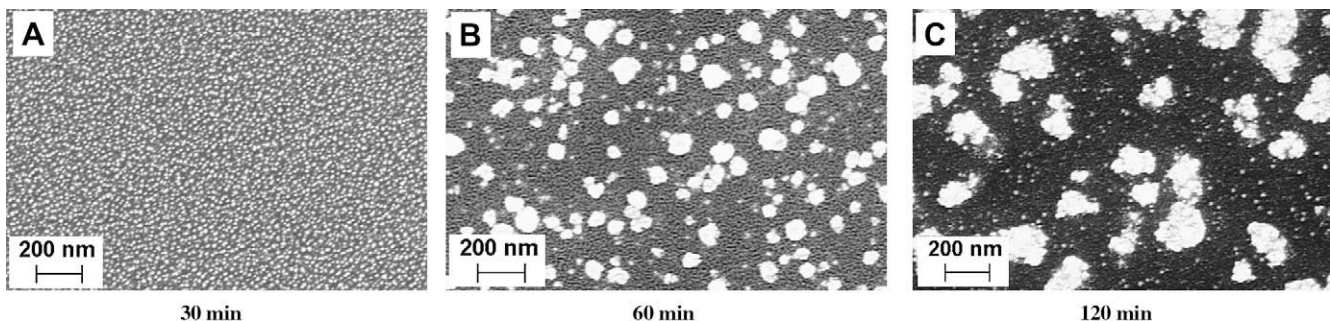


Fig. 2. The morphology of sample surfaces at constant bias voltage -170 V for each sample for A.) 30 min, B.) 60 min and C.) 120 min process time.

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