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EFFECT OF SUBSTRATE BIAS ON THE DIAMOND GROWTH USING MICROWAVE PLASMA CVD

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Abstract

On the effect of substrate bias at first stage of diamond synthesis at lower substrate temperature(approximately 673K) using microwave plasma CVD and effect of reaction gas system for the bias enhanced nucleation were studied.

The reaction gas was mixture of methane and hydrogen or carbon monoxide and hydrogen. The nucleation density of applied bias -150V using CH_4-H_2 reaction gas system, significantly higher than that of C-H_2 reaction gas system. When the CH_4-H_2 reaction was used, nucleation density was increased because of existence of SiC as a interface for diamond nucleation. By use of this negative bias effect for fabrication of CVD diamond film using two-step diamond growth without pre-treatment, fabrication of the diamond film consist of diamond grains 0.2㎛ in diameter was demonstrated

Key words : *Diamond, CVD, Bias, Nucleation, Reaction gas*

1. Introduction

On the diamond synthesis using various CVD methods, Si was often used as substrates for diamond growth. To obtain CVD diamond films on this Si substrate, it is necessary to scratch the substrate by high hardness powders such as diamond and cubic boron nitride (cBN).

On the other hand, substrate bias applied during diamond growth using microwave plasma CVD is effective for nucleation densities of CVD diamond grains[1]. Especially, negative bias increase the diamond nucleation densities on the Si substrates using microwave plasma CVD at high substrate temperatures such as 1173K[2]. So we have studied on the effect of substrate bias at first stage of diamond synthesis at lower substrate temperature (approximately 673K) using microwave plasma CVD. Also effect of reaction gas system for the bias enhanced nucleation was investigated.

2. Experimental procedure

Improved microwave plasma CVD apparatus was used to synthesize the diamond. Fig. 1

shows the schematic illustration of bias enhanced microwave plasma CVD apparatus. Table 1 shows diamond synthesis conditions. The reaction gas was used mixture of methane and hydrogen or carbon monoxide and hydrogen. The substrates were silicon wafers. Surface morphologies of the deposits were observed by Scanning Electron Microscope. The deposits were identified by X-ray Diffraction. The quality of the deposits was estimated by Raman spectroscopy and Auger Electron Spectroscopy. The surfaces of the substrates were analyzed by X-ray Photoelectron Spectroscopy

3. Result and discussion

On the nucleation densities using CH_4-H_2 reaction system, when the no bias voltage was applied, nucleation density was low and only diamond grains were observed by SEM. Positive + 100V bias, few huge diamond grains 1㎛ in diameter and fine grains 0.1㎛ in diameter were obtained. SEM image of the deposit applied bias was -150V is shown in fig. 2. Fine grains less than 0.1㎛ in diameter were observed and nucleation density was increased significantly, and as the result surface was covered with diamond grains.

Table 1. Synthesis conditions.

	CH_4-H_2	CO-H_2
CH_4 flowrate (SCCM)	6	-
CO floerate (SCCM)	-	50
H_2 flower (SCCM)	194	150
MW power (W)	300	300
Bias voltage (V)	-200 ~ +100	-200 ~ +100
Pressure (kPa)	1.3	1.3
Substrate temperature (K)	≒673	≒673
Reaction time (h)	3	3

When CO-H_2 reaction gas system was used instead of CH_4-H_2 system, effect of substrate bias was changed. When no bias voltage was applied, diamond grains 0.5㎛ in diameter was observed and then nucleation density was 10^4 numbers/cm^2 order. Fig. 3 shows SEM image of the deposit whose applied bias was -100V. nly few grins were observed and nucleation density

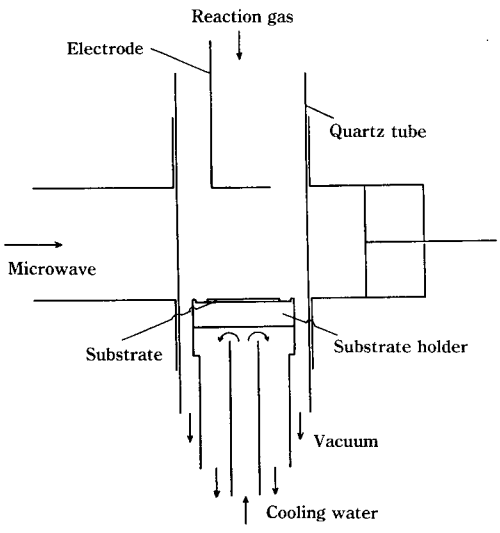


Fig. 1 Schematic Illustration of bias enhanced microwave plasma CVD appatatus.

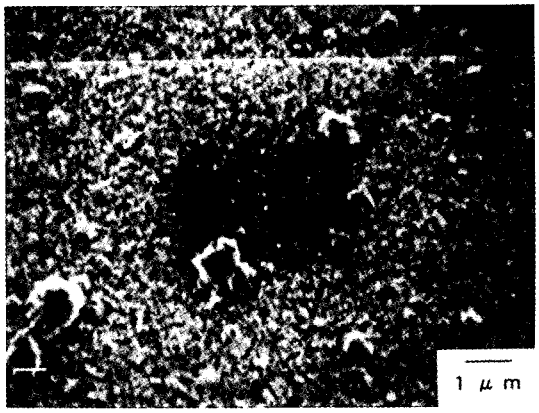


Fig. 2 SEM image of the deposit.(CH_4-H_2, -150V)

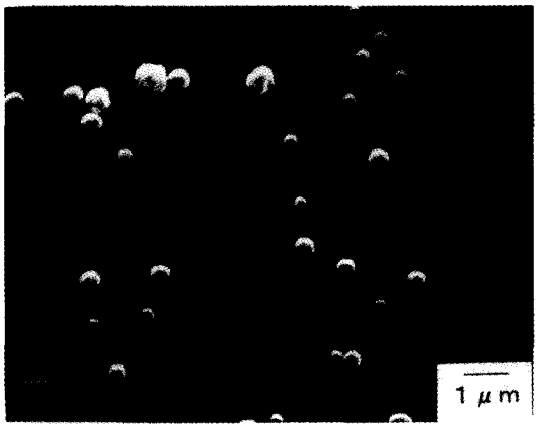
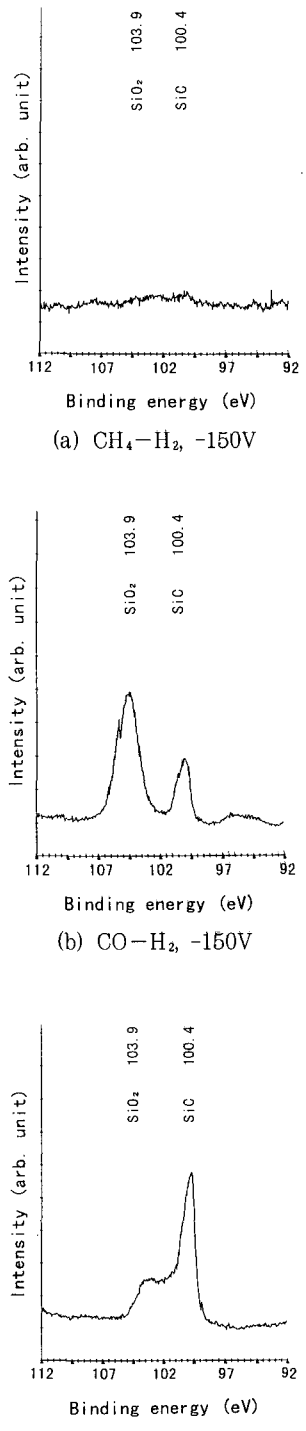


Fig. 3 SEM image of the deposit.(CO-H_2, -100V)

was so low. Nevertheless applied bias was -150V, nucleation density was increased just a little. The nucleation density of applied bias -150V using CH_4-H_2 reaction gas system was significantly higher than that of CO-H_2 reaction gas system. Increasing of nucleation densities to form the diamond films without pre-treatment were not recognized when CO-H_2 reaction gas was used.

To make clear the effect of reaction gas using bias enhanced nucleation, the Si substrate surfaces were analyzed by XPS. Fig. 4 shows XPS spectra of the Si substrate after bias treatment. When the reaction gas was CO-H_2 mixture and bias voltage; -150V Si_2 and SiC peaks were observed and Si_2 peak height was higher than that of SiC. Nevertheless using CH_4-H_2 reaction gas system, though both Si_2 and SiC peaks were observed, the ratio of Si_2/SiC peak height ratio was changed and Si_2 peak height was lower than SiC. As a result, negative bias voltage was effected to the acceleration of ions in the plasma and ion implantation was occurred on the surface of Si substrate. When CO-H_2



(a) CH_4-H_2, -150V

(b) CO-H_2, -150V

(c) CH_4-H_2, -100V

Fig. 4 XPS Spectra of the Si substrate after bias treatment

reaction gas was used, some kind of oxygen included ions were accelerated to the substrate and Si_2 was formed. However using CH_4-H_2 reaction gas was used, only ions without oxygen were accelerated to the substrate only SiC was formed. (Detected Si_2 was must be native oxide layer.) Consequently, when the CH_4-H_2 reaction was used, nucleation density was increased because of existence of SiC as a interface for diamond nucleation.

As the application of this negative bias effect for fabrication of CVD diamond film using two-step diamond growth without pre-treatment was demonstrated. SEM image of the deposit obtained by 2-step growth is shown in Fig. 5. First step, -200V was applied for 3 hours and after then bias voltage was reduced to 0V and diamond was deposited for 5 hours. As a result, the diamond film consisted of diamond grains 0.2 ㎛ in diameter. Still the substrate temperature was lower temperature near 673K, by using of negative bias applied the first stage of diamond growth, the diamond film could be obtained without pre-treatment.

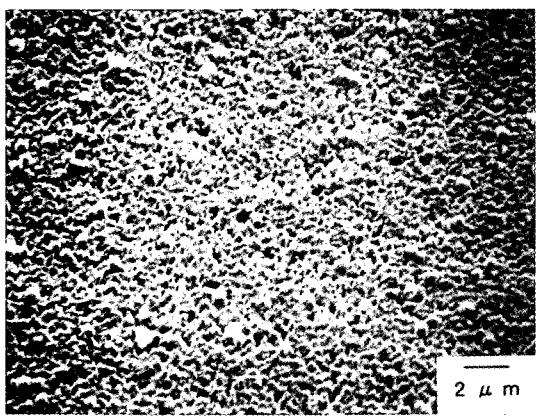


Fig. 5 SEM image of the deposit obtained by 2-step growth.

4. Conclusions

Investigation was carried out on the effect of substrate bias at first stage of diamond synthesis at lower substrate temperature (approximately 673K) using microwave plasma CVD and effect of reaction gas system for the bias enhanced nucleation.

As a result, the nucleation density of applied bias -150V using CH_4-H_2 reaction gas system shows significantly higher than that of CO-H_2 reaction gas system at the same bias voltage. When the CH_4-H_2 reaction was used, nucleation density was increased by the formation of SiC as a interface between diamond and Si substrate for diamond nucleation. Also using this negative bias effect for fabrication of CVD diamond film without pre-treatment, fabrication of the diamond film consisted of diamond grains 0.2㎛ in diameter was succeeded using two-step diamond growth.

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