직류 플라즈마 제트를 이용한 고속 다이아몬드 막 증착기술

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High-Speed Deposition of Diamond Films by DC Plasma Jet

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Abstract

A low pressure DC plasma jet has been used to obtain diamond films from a mixture of CH_4 and H_2 with high deposition rate (>1μm/min). The effects of the deposition conditions such as torch geometry, substrate temperature, gas mixing ratio, chamber pressure, axial magnetic field on the diamond film properties such as morphology, purity, uniformity of the film and deposition rate, etc. have been examined with the aid of Scanning Electron Microscopy, X-Ray Diffraction, and Raman Spectroscopy. Both the growth rate and particle size increased rapidly for low methane concentrations but saturated and the morphology changed from octahedral to cubic structure when the concentration exceeded 1.0 %. Higher growth rates (>1.5μm/min) can be obtained by applying an axial magnetic field to the DC plasma jet. Diamond obtained from the magnetized plasma jet also shows a sharp peak at $1332.5 cm^{-1}$ in the Raman Spectra and this result implies that higher growth rate with a good quality diamond films can be obtained by applying an external magnetic field to the plasma jet.

1. Introduction

A variety of deposition techniques have been used to synthesize diamond films from the gas phase at low pressure.[1] These techniques are hot filament assisted CVD,[2] MW plasma CVD,[3] RF plasma CVD,[4] Electron Assisted CVD (EACVD),[5] Laser Induced CVD,[6] DC plasma CVD,[7] RF induction thermal plasma CVD,[8] ECRCVD,[9] DC plasma jet,[10] Hollow Cathode CVD,[11] ion-assisted deposition,[12] arc discharge plasma CVD,[13] combustion flame deposition,[14] MW plasma torch CVD,[15]etc. Among these techniques, the DC thermal plasma jet reported the highest growth rates,[16] but little has been known about the quality of the film and optimum process conditions. In this work, the dependence of the surface morphology, film quality and growth rate of the DC thermal plasma enhanced diamond coatings on the process parameters ,especially on the external magnetic field, has been studied.

2. Experimental

A schematic drawing of our DC thermal plasma jet system is shown in Fig.1. A magnet is placed around the torch to generate an axial magnetic field. Before the deposition, the chamber is evacuated down to 10^{-3} Torr. The plasma gas is introduced into a non-transferred type DC thermal plasma torch to produce a plasma jet. The plasma jet is directed onto a Mo substrate and the substrate temperature during the deposition is measured by an optical pyrometer and a thermocouple. The substrate temperature can be varied from 700 °C to 1100 °C by changing the arc power, substrate cooling rate,etc. The substrates have been abraded by diamond powder and cleaned by acetone and methanol. In our experiment, the shape of jet flame and the position of the substrate in this flame are very important factors to produce high quality and high growth rate diamond films. Therefore the substrate temperature must be controlled within the limits of not interfering with the other process parameters such as the shape of the flame and the position of the substrate,etc. The chamber pressure can be varied from 1 Torr to 760 Torr by controlling the gas flow rate and the throttle valve. Magnetic fields are calculated numerically and confirmed by the Gauss-Meter measurements. The maximum magnetic field strength is 2600 Gauss at the center of the bobbin and 1200 Gauss at the nozzle of the torch. Experimental conditions are listed in Table 1.

3. Results and Discussion

The effects of the substrate temperature on the particle morphology and growth rate is one of the most important factors in the deposition of the diamond film by DC plasma jet. In our experiment, the growth rate and particle size increased with the temperature but the morphology changed from the faceted to unshaped when the temperature exceeded 1050 °C. The growth rate measured for each substrate temperature is shown in Fig.2. It increases from 0.7 μm/min to 1.0 μm/min when the substrate temperature changes from 800 °C to 1000 °C. In X-ray diffraction pattern, only diamond and Mo peaks appeared.[17] The obsered values of interplanar spacings of deposited diamond films are in a good agreement with the values reported in ASTM data. Raman spectra of diamond films at different substrate temperatures show that the sharp peak at $1332.5 cm^{-1}$ due to diamond is observed at 950 °C but the broad peak centered around $1560 cm^{-1}$ due to the graphitic structure dominates when the temperature deviates from its proper range of 900 - 950 °C.

The effect of the methane concentration change on the quality of the deposited film is the other important factor in the deposition of diamond film by DC plasma jet, and it is shown in Fig.3. The morphology of the film is quite different for each different condition of the methane concentration. It changes from the octahedral to cubic structure and the area of <100> planes increases as the methane concentration increases. The growth rate measured for each methane concentration is shown in Fig.4. It shows that the growth rate increases rapidly for low methane concentrations but saturates when the concentration exceeds 1.0 %. Raman spectra of diamond films at different methane concentrations show that all deposits have the sharp diamond peak of $1332.5 cm^{-1}$, but the broad peak due to the graphitic structure increases with the methane ratio.

By lowering the chamber pressure, higher velocity and higher enthalpy plasma jet can be obtained. Thus in our experiment, low chamber pressures have been preferred and high quality diamond films were produced at pressures of about 50 Torr. As the chamber pressure increases (more than 100 Torr), diamond films could also be obtained but the uniformity and bonding strength of the deposited films degraded. X-ray diffraction patterns and Raman spectra of these films are similar to those of deposited films at 50 Torr.[17]

As an external, axial magnetic field was applied to the torch, the arc voltage increased and the arc discharge became more stable. The plasma density and plasma temperature is thought to be higher in the presence of the magnetic field. Thus more intense, stable plasma jet can be obtained as the magnetic field increases. The growth rate measured for each different magnetic field condition is shown in Fig.5. It shows that the grain size and deposition rate increases with the magnetic field. The growth rate increased from 1 μm/min to 1.5 μm/min but the film uniformity seems to be degrading. Raman spectrum of the deposited film with the magnetic field of 600 Gauss is shown in Fig.6. It shows a sharp peak at 1332.5 cm^{-1} due to diamond and this result implies that higher growth rate with a good quality diamond films can be obtained by applying an external magnetic field to the plasma jet.

4. Conclusion

The effects of the deposition conditions such as the substrate temperature, gas mixing ratio, chamber pressure, external magnetic field on the diamond film properties have been examined. The growth rate increased from 1μm/min to 1.5μm/min and good quality diamond films were obtained when a magnetic field is applied to the plasma jet.

5. Reference

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Arc Voltage 15~30[V]	Ar Flow Rate 3~5[SLM]
Arc Current 100~300[A]	H_2 Flow Rate 1~2[SLM]
Spraying Distance 10~30[mm]	CH_4 Flow Rate 5~15[SCCM]
Substrate Temp. 850~1050°C	Pressure 50~200[Torr]
Magnetic Field 0~900[Gauss]	Deposition Time 30[min]

Table 1. Experimental conditions

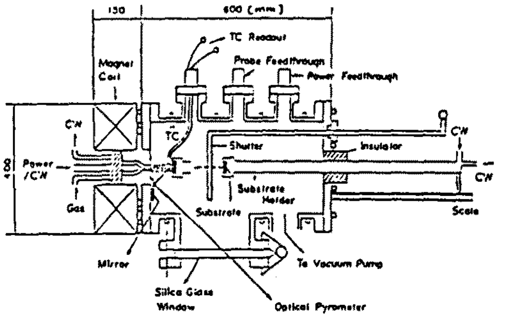


Fig.1. Schematic drawing of DC thermal plasma system

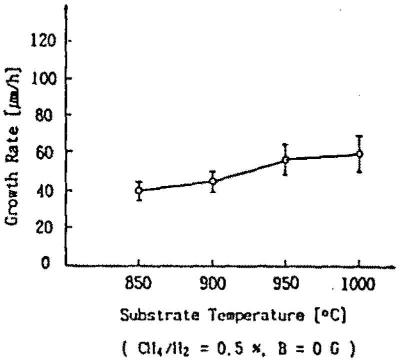


Fig.2. Film growth rate v.s. substrate temperature

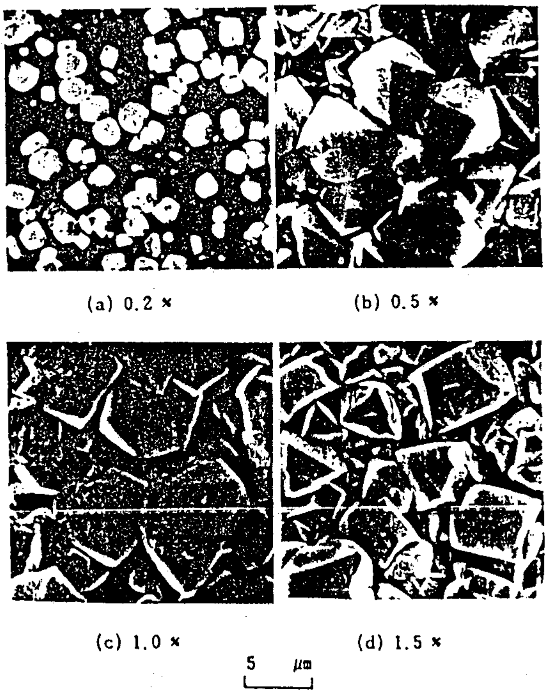
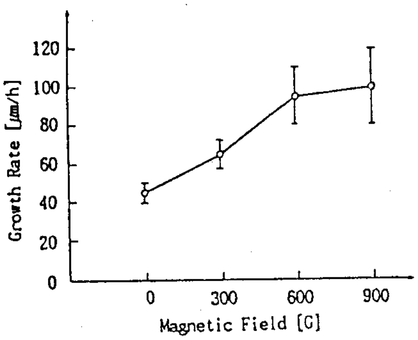
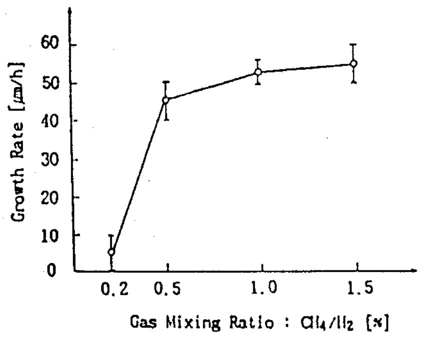


Fig.3. The effect of gas mixing ratio (CH_4/H_2)
(Temperature: 900°C, Deposition time: 30 min.)



(Temperature 900°C, CH_4/H_2 = 0.5 %)

Fig.4. Film growth rate v.s. gas mixing ratio



(Temperature 900°C, B = 0 G)

Fig.5. Film growth rate v.s. magnetic field strength

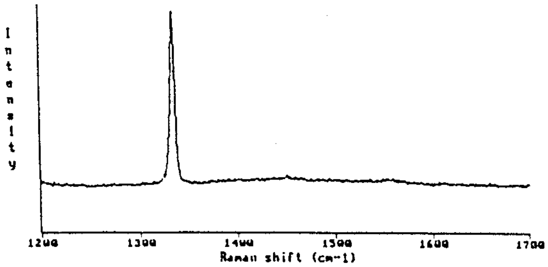


Fig.6. Raman spectra of deposited film under the influence of magnetic field
(Temperature: 900°C, CH_4/H_2=0.5%, B=600 Gauss)