Growth of Diamond by MPCVD Process

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Introduction

Diamond is a material with diverse use, ranging from jewelry, cutting tools to particle detectors in high energy physics. Diamond comes in naturally occurring and artificially produced varieties. The defects and impurities in naturally occurring diamond give its characteristics colors, but these defects are not desirable when the material is to be used for detector applications. The alternative is synthetic diamond, which can be grown in laboratory with less defects and less impurities. There are several methods of growing diamond in laboratory. Microwave Plasma Chemical Vapour Deposition (MPCVD) is one such method used today for growing high quality diamond films.

MPCVD Process

In this process diamond is grown by starting with a carbon containing gas, like methane, aliphatic or aromatic hydrocarbons, alcohols, ketones, amines, ethers and carbon monooxide. Methane is widely used because it can be obtained with high purity and also has the same structure (tetrahedral) as diamond. In addition to these gases, surface site preparation requires gases such as hydrogen, oxygen, or fluorine atoms. In this process we take Methane (1%) and hydrogen (99%) for growing high quality films. The plasma is created by high frequency (2.45GHz) and high power (kW) microwave which is responsible for the growth on the substrate [1]. To grow single crystal diamond we can use High Pressure High Temperature (HPHT)(100) diamond as the substrate material. This is called homoepitaxy (when substrate and material grown are same). We can also use non-diamond substrate such as Silicon, Iridium, cubic Boron Nitride- in that case film may be crystalline or poly-crystalline depending on surface energy, crystal structure and lattice constant mismatch, this is called heteroepitaxy. The quality of films is characterized by X ray diffraction(XRD), Raman spectroscopy and Scanning Electron Microscope (SEM). There are some advantages of using MPCVD Process:

1. It is an electrode-less process and hence energy efficient, because no plasma sheath formation take place around the electrodes as in the case of Direct current plasma assisted CVD.

2. The stability and reproducibility of non-isothermal plasma allows us continuous deposition and for many hours or days.

3. The increased availability of 1-2 KW microwave power supply and applicators (since growth rate is proportional to microwave power) allows the experimenter to use readily available modular units.

4. It has the potential for scaling up the process to larger substrates.

MPCVD System

The MPCVD system contains a Microwave generator of frequency 2.45GHz and power up to 2kW. The microwave from the rectangular wave guide is coupled to the vacuum chamber through a quartz window. Gas flow is controlled by mass flow controllers (MFCs) calibrated in standard cubic centimeter per minute (sccm). The substrate temperature can be controlled by the position of the plasma and is measured by a thermocouple.

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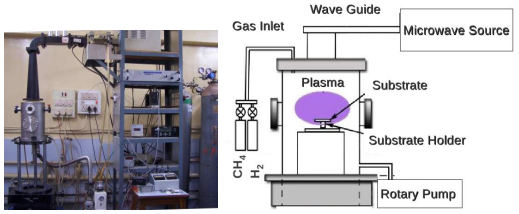

FIG. 1: (Left) MPCVD system and (right) the schematic of the system

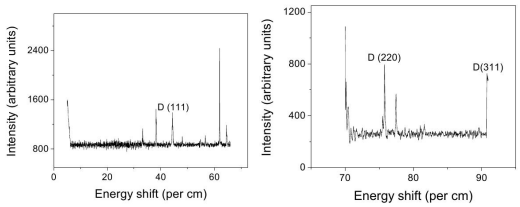

FIG. 2: XRD peaks for diamond are D(111), D (220) and D (311).

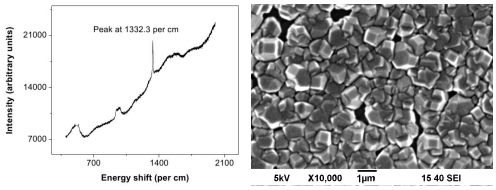

FIG. 3: (Left)Raman spectroscopy shows a sharp diamond peak (Si peak suppressed) and (right) SEM image of diamond film of 0.66 μm.

Growth Parameters of film

We used Si(100) wafer as the substrate. The substrate was first cleaned in Iso Propyl Alcohol (IPA) in an ultrasonic bath. Then it was roughened with 2 micron diamond powder for about 5 minutes to increase the nucleation sites and cleaned in ultrasonic bath again. The growth parameters used were: Si(100) substrate, CH_4 = 2 sccm, H_2 = 250 sccm, Substrate temperature = 800 $^\circ C$, Pressure = 80 torr, Power input 0.8kw, Power reflected = 0kW and Time = 5hours[2].

Characterization of film

XRD of the sample from 5 to 90 ° shows the peaks at the following positions of 2θ = 33.3 °, 38.2°, 44.3°,48°, 56.6°, 61.9°, 64.5°, 70.5°, 75.8°, 77.5°, 90.8°. XRD scans were taken in two steps first from 5 to 65 ° and then 70 to 91 ° (fig. 2) to remove silicon(100) peak (around 69.3 °) which can suppress other peaks because of its high intensity. The XRD scans have the peaks corresponds to diamond (111), diamond (220) and diamond (311) i.e. film is polycrstalline diamond film. Raman spectroscopy with Si peak suppressed show the sharp peak at 1332.3 cm^{-1} (fig. 3). This also confirms the formation of diamond (sp^3-form of carbon). SEM image also shows the formation of poly crystalline diamond film with small grain size (fig. 3) [3].

Future Steps

We are currently optimizing the growth parameters to increase the grain size and to reduce the defect levels.

References

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[3] "Research on Preparation Technology of High Quality Diamond Film." C. Zhoua *et al.* , *Materials Science Forum*, **749**, 392-400 (2013)