Heteroepitaxial Growth of Diamond on SiC Substrates

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Abstract:
Diamond’s high carrier mobility, thermal conductivity, and breakdown voltage allow for faster and higher power electronic devices than those made of silicon. Growth of thin, high quality diamond films has been consistently achievable since the 1980’s by chemical vapor deposition (CVD) and works best on diamond substrates. However, as diamond substrates are expensive and cannot be commercially grown large enough for mass production, this project explores silicon carbide as an alternative growth substrate. Diamond films were grown using hot filament chemical vapor deposition (HFCVD) under two filament configurations, with differing growth times and carbon-to-hydrogen ratios to determine optimal growth conditions. Quality of the diamond films was characterized by Raman spectroscopy, scanning electron microscopy (SEM), and energy-dispersive x-ray spectroscopy (EDS). Hall measurements were performed on selected samples to determine the electrical properties of the films. Fabrication of Schottky diodes was attempted, but samples were too conductive for electronic device applications.

Introduction:
Diamond’s superior carrier mobility, high thermal conductivity and high breakdown voltage make it ideal for high-speed power transistors. Silicon carbide (SiC) makes a good growth substrate for diamond because it also has a high thermal conductivity and breakdown voltage, making it suitable for use under the same high-power conditions. Additionally, SiC wafers can be grown large enough for mass production, while diamond substrates cannot [1]. Our project focused on growing unintentionally doped layers of diamond on semi-insulating SiC.

Experimental Procedure:
To prepare clean substrates, SiC wafers were brushed with soap and deionized water, and rinsed with acetone and methanol. The samples were then seeded for diamond growth by immersion in an equal volume mixture of 5 nm-size diamond particle slurry and methanol, and placed in an ultrasonic bath for ten minutes. The sonication process scratched up the SiC surface and deposited the nano-size diamond particles on the surface; both the scratches and particles acted as nucleation sites during growth. Six samples of SiC were prepared this way, five of the hexagonal 6H polytype and one of the cubic 3C polytype.
Growth of diamond was achieved in a hot filament chemical vapor deposition (HFCVD) reaction chamber. Diatomic hydrogen (H₂) and methane (CH₄) were passed over an electrically heated tungsten filament, causing them to react and deposit carbon on the surface of the SiC. One 6H sample was grown under a configuration of three straight filaments, while the remaining four 6H samples and the 3C sample were grown under a single, coiled filament configuration. Before growth, carburization was performed; a larger concentration of methane-to-hydrogen was passed through the chamber at a sub-growth temperature to bond carbon atoms to the outside of the tungsten filament and thereby prevent contamination of the substrate and diamond layer. The carbon ratio was then reduced and the filament current increased to achieve the desired growth temperature. The coiled filament setup had a shutter to protect the substrate from unintentional deposition of carbon during the carburization process.

The straight filament sample was grown for eight hours at a temperature of 2200°C and a pressure of 30 torr, with a 1.5% concentration of methane-to-hydrogen. The samples in the coiled filament setup were all grown at a temperature of 2600°C and pressure of 20 torr. Of the 6H samples, we grew in a 2% concentration of methane-to-hydrogen for 18 hours, a 1.5% concentration of methane-to-hydrogen for 21 hours, and a 1% concentration of methane-to-hydrogen for 20.5 hours. Subsequently, a 6H sample and 3C sample were both grown at 2600°C and 20 torr in a 1.5% carbon concentration for a short run of 2.25 hours.

Results and Conclusions:
Scanning electron microscope (SEM) images revealed well-faceted, polycrystalline diamond on all samples. All samples exhibited microcrystals of diamond except the 6H and 3C grown for 2.25 hours, which exhibited nanocrystals of diamond. The 6H 1.5% sample grown under the coil filament experienced the highest deposition rate (0.86 µm/hr) and the highest purity Raman spectrum; no graphite peak was visible. The 6H 2% and 1% samples both experienced film stress. The 2% sample had almost as high a deposition rate, but with a significant graphite peak; the 1% sample had a small graphite peak but a low deposition rate. A concentration of 1.5% carbon under the coil filament was determined to be the best sample.

Hall measurements were performed on the 1.5% 6H sample under the straight filament, and the 1.5% and 1% 6H samples under the coil filament. Mobility of the straight filament sample was 112 cm²/V-s, while the coil filament 1.5% and 1% mobilities were 9.28 and 32.34 cm²/V-s, respectively. Carrier concentration for the straight filament sample was on the order of 10¹⁸ cm⁻³, while for the coil filament samples, it was on the order of 10¹⁵ cm⁻³. Schottky diodes were unsuccessful, as all metal-diamond contacts were ohmic even without annealing.

Future Work:
The high carrier concentrations indicate unintentional dopants; energy dispersive x-ray spectroscopy (EDS) on the back of free-standing diamond from the 2% sample revealed 9% tungsten from the filament. Improving the carburization process to reduce the contamination of the substrate will decrease the conductivity. Annealing may also reduce bulk defects and decrease carrier concentration [2].

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References: