Center for Coatings and Laser Applications

Investigation of diamond deposition uniformity and quality for freestanding film and substrate applications

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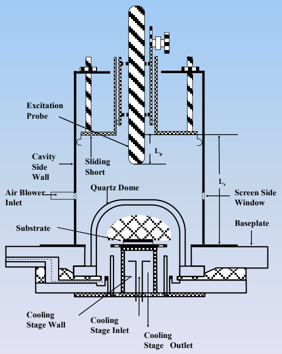
Presentation Outline

- Description of the MSU designed microwave plasma-assisted CVD reactor;
- Typical relationships among substrate temperature, microwave power and reactor gas pressure;
- General growth conditions and performance outputs;
- Description of laser cutting, polishing, lapping, back etching silicon and plasma etching;
- Deposition and diamond film thickness uniformity and optical transmission analysis;

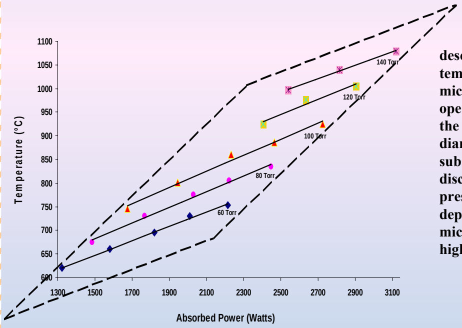
The Microwave Plasma-Assisted CVD Applicator

The polycrystalline diamond films are grown in a microwave plasma-assisted CVD reactor using hydrogen & methane chemistry. The methane percentage is nominally 1-2 %. The deposition reactor is a microwave cavity applicator with the plasma confined inside a 12.5 cm diameter quartz dome [1]. The substrates utilized are 2 and 3 inch silicon wafers with thickness of 1 mm. The silicon substrates are prepared using mechanical polishing with diamond powder for nucleation.



The substrate is actively cooled with a water cooled substrate holder to achieve a substrate temperature of 850-1200 °C. The pressure utilized is 80-140 Torr and the microwave power is 3.5-4.5 kW. The substrate holder is designed to achieve a uniform substrate temperature distribution.

Substrate Temperature vs. Microwave Power and Pressure – The Field Map



The figure to the left describes the substrate temperature, absorbed microwave power and operating pressure regime for the uniform deposition of diamond over 2 inch diameter substrates. Since the plasma discharge size decreases as the pressure is increased, uniform deposition requires higher microwave input power for higher pressure operation.

General Growth Conditions and Performance Output

Sample	Pressure (Torr)	Substrate temperature (°C)	Gases			Power absorbed (kW)	Process duration (hrs)	Ave. thick (um)		Growth rate (um/hr)	
			H2 (sccm)	CH4 (sccm)	Ar (sccm)			Weight gain	Linear Encoder	Weight gain	Linear Encoder
2"-20	140	970	400	5.56	0	2.66	48	58		1.20	
2"-25	140	1080	400	5	0	3.60	25	19		0.76	
2"-31	140	955	400	4	0	3.00	96	75		0.79	
3"-07	100	900	400	6	200	2.49	48		53		1.09
3"-09	120	980	400	6	200	2.64	48		71		1.47
3"-22A	100	985	400	8	200	2.73	21		31		1.79
3"-22B	100	1040	400	8	200	2.61	68.5		110		1.98
3"-22C	100	1055	400	8	200	2.68	134.9		226		1.93
3"-22D	100	985	400	8	200	2.63	182.9		312		1.90
3"-13	120	920	400	8	0	3.32	48		116		2.42

Post-Processing to Create Diamond Windows and Ion Beam Stripping Foils

Once the diamond is deposited on the silicon substrate a number of post processing steps are performed to fabricate smooth, flat and uniformly thick films or substrates. These processing steps typically include laser cutting, lapping and polishing of the growth side of the diamond, removal of the silicon substrate, and plasma etching to remove a thin layer on the nucleation side of the diamond film. Laser cutting is performed with a pulsed Nd-YAG laser operating with the third harmonic. Lapping and polishing is performed with a Logitech LP 50.

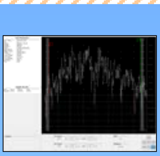
Left: Rectangular pieces and circular disks cut from the deposited diamond. The rectangular pieces are 12 × 18 mm. The diameter of the circular disc #1 is 0.5 inch or 12.7mm. The other two circular discs are 9mm in diameter.

References

[1] K.-P. Kuo and J. Asmussen, "An experimental study of high pressure synthesis of diamond films using a microwave cavity plasma reactor," *Diamond and Related Materials*, 6, 9, 1097-1105 (1997).
[2] S. Kamiya, M. Sato, M. Saka, and H. Abe, "Residual stress distribution in the direction of the film normal in thin diamond films," *J. Appl. Phys.*, 86, 1, 224-229 (1999).

Lapping and Polishing

Above: Lapping and polishing is performed with a Logitech LP 50. The polishing system has been used to achieve surface roughness values of 3 nm.



Above: Dektak D6M data plot of surface roughness of a polished CVD diamond shows an average surface roughness of Ra = 3.3nm.

Back Etching (to remove silicon) and Plasma Etching

- A challenge to the fabrication of flat and uniform films is dealing with the residual stress of the diamond film.
- Residual stress occurs at the diamond-silicon interface due to the thermal expansion mismatch and due to intrinsic stress within the diamond film. The intrinsic stress may be caused by grain boundary coalescence, defects and impurities [2].
- The stresses may cause film bowing or cracking during the removal of the silicon substrate and the process of film polishing.
- The small crystal size on the nucleation side can cause degraded optical properties and can contribute to the bowing of the film.
- Free-standing diamond films were plasma etched to remove about 10 µm of the film on the nucleation side. They appear whiter after the plasma etching as shown in the lower-left figure below.

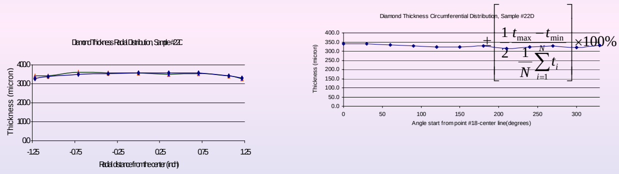
Above: A nucleation side layer is etched away using plasma etching.

Lambda Technologies Plasma Etcher

Typical Reactive Ion/Plasma Etching Conditions

- Input microwave power: 400 Watts
- Gas flow: Ar – 12 sccm; SF_6 – 2 sccm; O_2 – 20 sccm
- Rf Bias: 100 Volts
- Pressure: 4 mTorr
- Etch rate: 3 – 4 µm/h

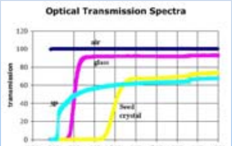
Diamond Film Growth Uniformity



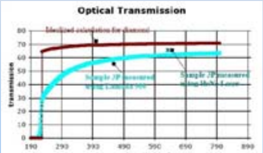
$$\pm\left[\frac{1}{2}\frac{t_{\max}-t_{\min}}{\frac{1}{N}\sum_{i=1}^{N}t_i}\right]\times 100\%$$

Growth uniformity for sample 3"-22D; Top: radial thickness distribution. The radial thickness uniformity is ± 4.7 %; Bottom: circumferential thickness distribution at a radius of 1.25 inches. The circumferential thickness uniformity is ± 4.0 % .

Diamond Film Quality - Optical Transmission

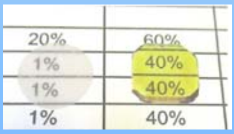


Above: Transmission spectra measured with Perkin Elmer Lambda 900. Sample 3P is cut from a 2"sample (#25). Measured spectra include the air and a nitrogen doped single diamond crystal.

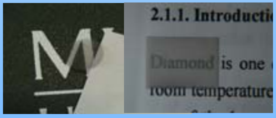


Above: Other methods are also used to measure the optical transmission quality include the H_eN_e laser and visible light transmission for 3P (RA=6.5nm and 11nm on the two sides). The HeNe result is 65.8%±0.9% as compare to the ideal transmission for diamond of 70.7%.

Diamond Window and Ion Beam Stripping Foil Samples



Sample 3P (left) and a Sumitomo seed crystal (right, yellow) supported on a glass microscope slide above a printed white page. The vertical separation provided by the slide was 15 mm.



Above: Transparent circular and rectangular diamond film/foils. The circular film is cut from a 2" sample (#25) and is polished. The rectangular film is unpolished.

A 36 µm thick circular freestanding polycrystalline unpolished diamond film mounted on the microwave transmission test structure .
(Photo credit to J. Booske, U. Wisconsin)

Above: A 35 µm thick polished polycrystalline diamond film mounted on the brass frame for use as an ion beam stripping foil.