A Description of The Experimental Microwave Discharge Behavior Versus Pressure, Power and Reactor Geometry for MPACVD Diamond Synthesis Reactors

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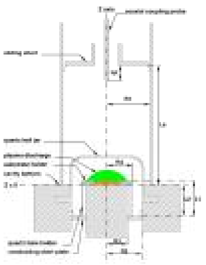
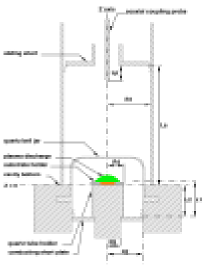
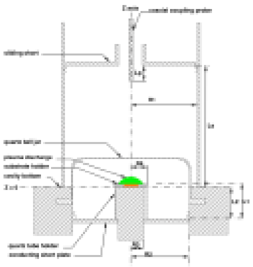
INTRODUCTION

Recently two new microwave plasma-assisted chemical vapor deposition (MPACVD) diamond synthesis reactors [1,2] were designed, built and experimentally evaluated and their performance was compared to earlier MPACVD reactor designs [3]. In order to take advantage of the improved CVD diamond synthesis conditions that occur within the high pressure regime (160-300 Torr) the two reactors were designed to operate with high discharge power densities and at high pressures. When these reactors operate at high pressures single crystal MPACVD diamond synthesis occurs over a large range of reactor conditions [1,2] such as: (1) pressure, p = 100 - >300 Torr. (2) input power 1-3 kW, (3) flow rates ~ 400 SCCM, (4) methane to hydrogen concentrations of < 1% to greater than 9%, (5) discharge power densities of 200-1000 W/cm^3, (6) different substrate holder designs/geometries, and (7) a variable plasma/substrate position, Δz. Within the 120-300 Torr operating pressure regime the discharge gas temperature is estimated to vary between 2500-4000K [4]. In order to efficiently supply the CH_3 and H growth species to the substrate the discharge itself must be located away from the reactor walls and in close contact with the substrate. CVD diamond synthesis typically requires a substrate temperature of 800-1300K. Thus substrate cooling is required. During MPCVD diamond synthesis the microwave discharge supplies both the growth species and thermal energy to the substrate. The design, operation and optimization of these MPACVD reactors is a new, complex, and challenging multivariable problem. It is important to develop an experimental understanding of the behavior of the microwave discharge versus pressure and input power, and to understand the variation of diamond synthesis rates versus reactor design. Here we briefly summarize the experimental performance of several MPACVD designs.

DIFFERENT MICROWAVE CAVITY REACTOR (MCPR) DESIGNS


Reactor A


Reactor B


Reactor C [3]

1) Reactor A – the benchmark reactor
- "Hybrid TM_{013}/TEM_{001} electromagnetic mode" excitation
- Cooled substrate holder on the "powered electrode" located around the z=0 plane.
- R1= 5.08cm, R2= 8.89 cm, R3= 4.13cm.

2) Reactor B
- Substrate holder/cooling stage radius is reduced from 4.1 cm to about 1.9 cm, thereby decreasing the area of the powered electrode by a factor of 4-5 over Reactor A.
- "Hybrid TM_{013}/TEM_{001} electromagnetic mode excitation" as in Reactor A but a length tunable TEM section.
- Substrate position Δz = L1 –L2 is variable from + 4mm to -9mm.
- Reactor now incorporates four tuning variables for matching and process optimization and control, i.e. Ls, Lp, L1 and L2.

3) Reactor C
- Same substrate holder/cooler size and variable substrate position Δz as Reactor B.
- The applicator cavity and quartz dome diameters increased by >50%.
- The excitation of a new hybrid mode, i.e. the TM_0/TEM_{001} mode, that focuses the electromagnetic energy onto the substrate


Commercial realization of Reactor C by Lambda Technologies

OBJECTIVE

To experimentally understand the behavior of the MCPR versus the many experimental and design variables. In order to develop an understanding of the reactor behavior we experimentally measure and plot the nonlinear performance of the MCPR versus reactor design; i.e., a set of experimental "road map" curves are measured that describe the reactor's nonlinear performance versus the many experimental input and design variables.

REACTOR DESIGN VARIABLES

- § Reactor size
- § Powered electrode size
- § Powered electrode position - Δz
- § Substrate size
- § Substrate holder design

EXPERIMENTAL VARIABLES

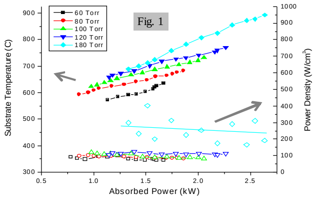
q Input Variables
- § Absorbed Power: Pi – Pr = P_{abs}
- § Pressure: p
- § Flow rate ~ 400 SCCM
- § CH_4/H_2: fixed at 3% in roadmap measurement

q Internal Variables
- § Substrate temperature: T_s (900-1400 °C)
- § Plasma volume: V_p (4-10 cm^3)
- § Absorbed power density: P_{abs} / discharge volume (200-1000 W/cm^3)

REFERENCES

[1] K.W. Hemawan, et. al., Diamond and Related Materials, 19, 1446-1452 (2010).
[2] Y. Gu, et.al. , Diamond and Related Materials, 24, 210-214 (2012).
[3] K. P. Kuo and J. Asmussen, Diamond and Related Materials, 6, 1097 (1997).
[4] K. Hassouni, F. Silva, A. Gicquel, J. Phys. D Appl. Phys. 43 (15), 153001 (2010).

EXAMPLE REACTOR ROADMAP – defining the safe and efficient operating regime



When operating with a specific fixed design/geometry, the substrate temperature is a function of both the pressure and the absorbed microwave power. Given a constant input chemistry, the major independent experimental variables, i.e. absorbed microwave power, P_{abs}, and pressure, p, have an experimentally repeatable, single-valued nonlinear relationship with the experimentally measured substrate temperature, T_s. Given a specific reactor configuration, gas input chemistry and flow rate, this nonlinear relationship can be measured. The resulting set of curves represent important data describing the useful experimental diamond deposition regions of the reactor system. Such a set of curves is shown in Fig. 1 and is identified here as the "operating field map" of the reactor. If at any given constant pressure P_{abs} is varied within the operating regime the resulting absorbed power density remains approximately constant. This is also shown in Fig. 1.

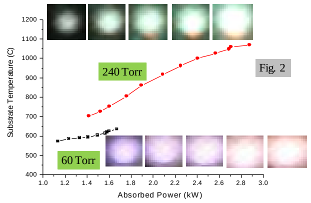


Fig. 2 displays example roadmap curves along with discharge photos for 60 Torr and for 240 Torr for Reactor B. Note that $\Delta T_s/\Delta P_{abs}$ is lower at low pressures than at high pressures.

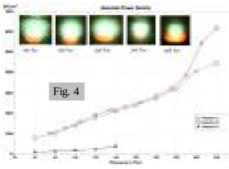


Fig. 4 displays the discharge absorbed power density versus pressure for the three reactors. Each curve is constructed by measuring the discharge volume, V_d , and associated P_{abs} . The absorbed power density, i.e. P_{abs}/V_d versus pressure is plotted as the reactor operation is varied along the green dotted line shown in Fig. 3.

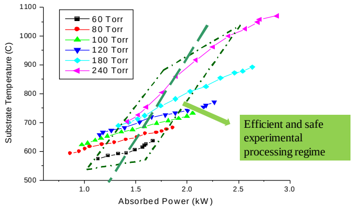


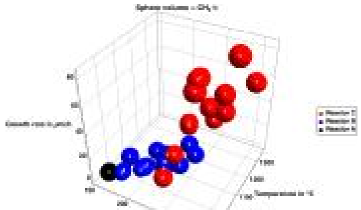
Fig.3 Operating roadmap curves for reactor B

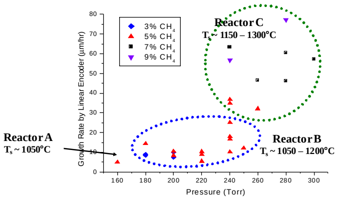
Fig. 3 displays an example of the operating field map for Reactor B. The operation of the reactor at a given specific absorbed input power and operating pressure results in a unique and repeatable substrate temperature. When the pressure is held constant and the input power is independently increased or decreased, the discharge size is also correspondingly increased and decreased and the substrate temperature traces out the curves as shown in Fig. 2. The left and right dashed lines represent for each pressure the allowable operating input power levels that yield useful diamond deposition over a one inch diameter silicon substrate. The left green dashed line represents the minimum power required to form a one diameter hemispherical discharge over the one-inch wafer. Input power levels of less than indicated by this line limit will not produce uniform deposition over one inch. The right dashed line represents the input power at which the plasma expands beyond an one-inch diameter and may even touch the quartz dome walls. Greater input powers may cause dome heating and possibly undesirable plasma dome wall interactions. Thus the region enclosed by the dashed lines represents the efficient and safe plasma processing regime for the reactor.

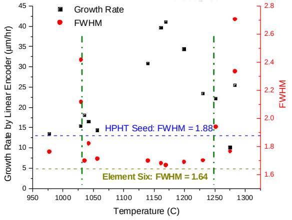
REACTOR PERFORMANCE [1-3]

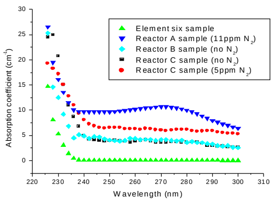
SCD synthesis is a multi-variable optimization problem – when holding substrate size and flow rate (~400 SCCM) constant the major variables are pressure, CH_4/H_2, substrate temperature, and reactor design. A multi-dimensional plot is necessary to evaluate different reactor performance.

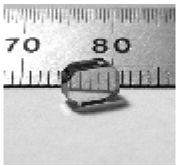
- § Growth rates versus pressures for Reactor A, B, and C (N_2 = 0).
- § Reduction of powered electrode area and the increase in pressure increases the deposition rate.











For Reactor B (240 Torr, 6% CH_4, N_2 = 0)
- § Synthesized SCD was characterized by micro-Raman spectroscopy , IR-UV transmission spectrometry and SIMS.
- § A good quality SCD growth window was observed between 1030 – 1250 °C.
- § The Raman FWHM ranged from 1.65 – 2.0 cm^{-1}. SIMS analysis shows less than 300 ppb N and Si in the synthesized SCD.

- § Without extra N_2 addition, the absorption coefficient of samples from Reactor B and C are comparable.
- § The absorption coefficient is lower when N_2 concentration decreases.
- § Colorless type IIa diamond is synthesized. A cut and polished 1.1carat diamond plate is shown in the above fig..

SUMMARY

- § We have experimentally explored and determined the safe and efficient reactor operating regime; i.e. the reactor operating roadmap has been defined. Within this same and efficient operating regime, the discharge is sufficiently large to cover the substrate but it is small enough to not waste power and also not touch the walls of the quartz dome,
- § SCD can be readily synthesized from 160 – 300 Torr within this safe and efficient operating regime. High quality type IIa SCD synthesis has been demonstrated with growth rate of 40 – 70 μm/hr.
- § Large SCD crystals have been produced by these reactors.