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"Materials Processing and Manufacturing Technologies for Diamond Substrates Multichip Modules"

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CONTRACT #N00014-93-C-2026
"Materials Processing and Manufacturing Technologies for Diamond Substrate Multichip Modules"

Overview
This quarter has seen the first production of continuous diamond films in our 75kW reactor. Our efforts have concentrated on three fronts: improving reliability and capability of the reactor, understanding the diamond growth chemistry and how it compares to the prototype operation, and further modeling for refining the reactor design. On the first front, we are regularly operating the reactor around the clock, and steadily improving the power utilized in diamond growth as well as the design and thermal control of the substrate stage. On the second front, we have initiated a series of deposition runs using a matrix of conditions to investigate the effects of gas composition and throughput on growth rate and diamond quality. These runs have included real time pyrometry, emission spectroscopy, and residual gas analysis for comparison with prototype operation and modeling. Our first iteration has yielded what we believe is a world record of over 350 mg/hr of CVD diamond at only 40 kW of microwave power.

Our near term goals include: 1) production of free-standing 6" diameter films, 2) finishing the current iteration of chemistry matrix runs and understanding the similarities (and differences) with our 2.45 GHz prototype; 3) further increasing power delivered to the plasma, and power removal capability of the substrate stage.

Facilities
As previously reported, all 75kW support facilities are now complete. The upgrades to electrical power, gas handling, and cooling facilities are fully operational. We have instituted several small improvements recently as our facility requirements have been refined: we have re-plumbed our cooling water drop to accommodate improved cooling to the dummy load and air-heat exchanger in the power supply, and we have added facilities for an additional process gas to our gas handling facilities for more complex chemistry experiments.

75kW Reactor
We reported (last quarter) our first plasma, achieved on 4 June 1993. Since this time, we have worked to increase the microwave power delivered to the plasma, and improve the power removal capabilities of the substrate stage.

The early work in improving power delivery to the plasma resulted in greatly improved coupling efficiency, primarily through modifying the impedance of the applicator and pre-tuning several sections of waveguide. We have experienced nearly 40 dB improvement in coupling since our first plasma was obtained. This has reduced reflected power considerably, thus improving the reliability of the magnetron, stability of the discharge, and giving reduced heating of waveguide components. This has also allowed unattended around-the-clock operation at the 40 kW level. We still observe 10%-20% reflected power under most conditions, thus additional refinements in pre-tuning will give greater efficiency in power utilization, and greater reliability of components (especially as the power delivered is increased to the full 75kW specification).

Our early efforts have also inspired further refinements in the design of the substrate stage. The original stage was found to be sensitive to thermal stresses, and accumulated deformations over large areas were sufficient to result in uneven cooling of the substrate, thus uneven diamond deposition thickness and morphology. We have installed several short-term modifications to compensate for these deformations. In addition, we have completed the design of a modified stage, optimized to accommodate extremes of thermal stress, and provide greater and more uniform cooling. We are, in parallel, experimenting with variations in substrate stage materials, and the subsequent effects on both thermal uniformity and deposition chemistry.
Near term plans include further waveguide pre-tuning, higher power delivery to the reactor, installation and testing of the modified stage, and further refinements in the chamber shape for improving deposition uniformity.

**Process Transfer, Ongoing Experiments**

We have been quite successful in transferring the diamond growth process in conventional (H₂, CH₄, O₂) chemistries from our prototype to the 75kW reactor. Our initial experiments have shown that we can achieve higher carbon utilization efficiencies (above 10%) than in the prototype, thus implying further reductions in projected costs of diamond from reduced gas feedstock requirements. We have made extensive use of our emission spectroscopy diagnostic as an *in-situ* benchmark for runs in progress.

We have primarily explored deposition on three substrates: 6" Si wafers, 8" Si wafers, and sets of four 3" Si wafers on an 8" circle. The latter have been quite useful for less expensive and more accurate measurements of growth rates on short runs, and thus have been used extensively for experimenting with variations in growth chemistry. The single wafers have been used to benchmark the results of the 3" wafers, and to measure the radial variations in power flux and heat removal. Some of the earliest runs with 8" wafers have yielded throughputs of over 350 mg/hour (>3/hour).

We have instituted a series of chemistry modification experiments to observe the effects of gas composition and throughput in this reactor (under constant power and pressure). The first iteration has given a family of growth curves similar to our prototype -- thus we can now estimate more accurate scaling factors between the two reactors and more accurately predict the results of new recipes using our prototype database. Preliminary results with one iteration from this study have increased the linear growth rate to 4.5/hour (at the same power and pressure) over an 8" diameter area (with four 3" wafers), or an effective 8" throughput of over 510 mg/hour.

We have also begun experiments designed to test the effect of CHₓ to C₂Hₓ radical conversion in high temperature and long residence time (diffusive) cases. We are attempting to direct CH₄ injection locally (to the substrate) to improve growth rate and quality, and perhaps improve carbon utilization efficiency. These techniques have the additional benefit of flexible control over the gas composition profile close to the substrate, thus allowing finer control over deposition uniformity. These experiments are in progress and the results to date are inconclusive.

Near term plans include: finishing initial experiments in conventional chemistry growth rates, refinements in deposition process (substrate mounting, power, pressure) for improved uniformity, and production of free standing films of 6" or 8" diameter. Additional work is planned to investigate non-conventional chemistry; the gas handling facilities have already been upgraded to accommodate these experiments.

**Reactor Prototype Related Studies**

We are continuing to study high growth rate (HGR) process in our PDS 19 reactor, the prototype for the 75kW system. We have recently taken delivery of an 8kW supply (upgraded from 5kW), and have investigated process scaling with higher power. We have instituted a series of runs to refine operating conditions for maximum uniformity of thermal management material at high power for 2", 2.5", 3", and 4" substrates. We are finding that one of our principal limitations is a peaked substrate temperature profile, which results in non-uniform deposition rates and morphology.

As our operating experience with the prototype (especially at 8kW) and the 75kW reactor grow, we continue to find a great deal of confirmation between modeling predictions and design features of both reactors. As such, we are making use of the prototype to test refinements in reactor design, as hardware changes are faster and more cost effective.
Our near term plans for prototype operation are to refine run conditions for optimum uniformity, and transfer this information to the 75kW reactor. In parallel, we will be testing improvements suggested by modeling for improving uniformity, and transferring successful designs to the large reactor as well.

**Reactor Modeling**

As reported last quarter, under DARPA Contract #DAAH01-92-C-R109 for “Computational Modeling of Fluid Flow in PECVD Reactors,” we have developed an axisymmetric electromagnetic code to model the power input to the microwave discharge. This code is now well benchmarked against the PDS19 prototype, and early benchmarks with the 75kW reactor (40kW 70-90 torr) operation match observed operation quite well. We have previously noted that the flatness of the plasma disk (and hence the power coupled to the substrate) can be significantly tuned by changing the reactor shape, which modifies the electric field boundary conditions at the plasma. This result has been extensively confirmed experimentally with the PDS19 prototype, and verified for a few points on the 75kW reactor.

Our stage redesign efforts have been intimately linked to the modeling work. The modeling has shown a peaked profile of atomic hydrogen flux from the plasma to the substrate, an observation confirmed by peaked thermal profiles on both reactors. While the modeling efforts are directed to minimize this peaking factor, the thermal stage design has been enhanced to compensate as much as possible, in order to achieve a uniform temperature profile and deposition rate and quality. These factors are also quite sensitive to the substrate height, thickness, and the size of the plasma compared to the substrate -- depositions ranging from strongly peaked to very uniform (10%) have been achieved for various conditions. In general, however, a given uniformity can be achieved only by enlarging the plasma to substrate size ratio sufficiently, thus reducing the effective throughput of the reactor.

Our goal is to refine the reactor configuration to flatten the atomic hydrogen flux profile as much as possible. Our near term plan is to expand our investigation of reactor shape modifications to optimize the uniformity, and test modified designs on the PDS19 prototype before installing on the 75kW reactor. These modeling studies are already underway, with several CPUs running different configuration changes daily.