sp3's Experience using Hot Filament CVD Reactors to Grow Diamond for an Expanding Set of Applications

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HOT FILAMENT CVD REACTORS DEMONSTRATE FLEXIBILITY, RELIABILITY, ECONOMY AND SAFETY

Over a span of ten years in development and manufacture of hot filament CVD reactors, sp3 has proved this deposition technique to be flexible in application, consistent in performance, economical in production, and safe.

Uniform diamond films ranging in thickness from 5000 angstroms to 100 microns can be deposited over areas of 350 mm diameter or more.

Electronic applications include silicon wafers, IC thermal management and titanium electrodes. Mechanical applications include a very wide range of cutting tools as well as carbide guides, wear surfaces, pivots and seals.

Background

sp3 was incorporated over ten years ago in Mountain View, California with the goal of developing Chemical Vapor Deposition (CVD) equipment, processes and products that would exploit the unique properties of diamond. One of the main challenges was to select a technical path that would provide for reliable, cost effective manufacturing on a production scale, day after day. After much analysis the path selected was hot filament CVD, as this was clearly the best route to cost-effective large area deposition in two and three dimensions.

The initial products from the sp3 hot filament approach were diamond coated cemented carbide cutting tools. sp3 has performed over 6000 reactor runs and successfully coated over one million carbide cutting tools during its tool coating history.

In addition to producing cutting tools, sp3 designs, manufactures and sells hot filament reactors and, using diamond CVD, has developed additional products and processes for a wide range of applications.

Equipment, Process and Film Growth Using Hot Filament Reactors

Cutting Tools

The sp3 hot filament system deposits over an area approximately 350mm by 375mm. The system employs fine wire filaments (0.12mm in dia.) in a cold wall aluminum chamber; typical total system input power is between 22 kW and 30 kW. The system's filaments are horizontal, and can be arranged in a two-dimensional or three-dimensional array. A sophisticated process controller provides for complex deposition recipes with up to 58 discrete steps. This high degree of program control is critical for the proper management of orderly startup, nucleation, growth, and shut down and, most important, safety.

The sp3 CVD system has grown a wide range of films from nanocrystalline smooth and regular coarse films as thin as 5000 angstroms to as thick as 100 microns. Typically, films used in coating cemented carbide cutting tools are in the range of 10 to 40 microns thick. Diamond and cemented carbides have quite different coefficients of thermal expansion. As the films get thicker, the adhesion bond between the diamond film and the substrate is put under increasing stress as the substrate returns to room temperature from the normal deposition temperature of 800 to 900°C. The present technology to promote the adhesion of diamond on cemented carbides will tolerate films up to about 50 microns thick for cutting tool applications.

Both coarse and fine grain films are used in cutting tools. The coarse films are best suited for roughing applications. Fine grain films are often used when sharper edges are required or chip evacuation is an issue, as with endmills and drills. Grain size can also affect surface finish of the workpiece. With the programmable process controller employed by the sp3 hot filament system it is easy to vary the grain size as well as other properties of the grown films.

Electronic Applications

Typical films are 1 to 3 microns thick and are grown on silicon wafers when aiming to exploit diamond's unique electronic properties. sp3 has grown adherent diamond films on silicon wafers up to 300 mm in diameter. The properties of these films are typically uniform to +/- 10%, including film thickness over the full diameter. In the nanocrystalline films the typical grain size is 10 to 100 times finer than the fine grain film used for cutting tools. Using Raman analysis, these thin films still exhibit the typical diamond 1331 peak**.** Films can be either undoped or doped with boron for those applications requiring electrical conduction.

Electrodes

Many applications in electrochemistry require electrodes to conduct the appropriate electrical power in the reaction. In many instances, electrodes are formed from titanium mesh and then coated with various noble metals. These electrodes come in a wide variety of sizes and shapes with maximum sizes approaching 1m by 1m. Hot filament deposition is clearly superior for this technology because of the ease of scaling to these large areas.

Electrodes fabricated from titanium mesh and coated with hot filament CVD diamond have clearly demonstrated longer life over the typical noble metal coatings when used in a wide variety of electrochemical applications. sp3 has successfully coated titanium electrodes up to 300mm by 300mm with conductive diamond using hot filament technology.

Thermal Management

Films up to 50 microns thick have been grown on 200mm wafers in hot filament reactors for experiments in thermal management of integrated circuits. Flatness, fixturing in the reactor and the thermal performance of the diamond are all considerations. Using hot filament reactors, sp3 has grown 20-micron films on 200 mm, 750-micron thick silicon wafers while maintaining a flatness of 50 microns.

Diamond on Silicon

sp3 has successfully coated silicon wafers using hot filament technology for the past seven years. Wafers as small as 50mm up to 300mm have been coated for a wide variety of uses, in addition to prototypes for electronic applications.

Mechanical Applications

In addition to cutting tools, sp3 has experience in coating cemented carbide, silicon carbide and silicon nitride in a wide variety of shapes when extreme wear resistance is required. These films typically require smooth surfaces, either as grown or polished after growth. Hot filament technology allows the uniform growth of these smooth films with surface finishes better than 0.2 micron Ra. Applications are guides, wear surfaces, pivots, and seals

Film Properties

For thermal applications, hot filament reactors typically grow films in the range of 10 to 15 W/cm/°K. Natural IIa diamond is over 20W/cm/°K. However, this rare form of diamond is much too expensive to be considered for fabrication into heat spreaders. Various laboratories have demonstrated diamond growth that approached type IIa diamond in its ability to spread heat. Moving these lab experiments into cost effective production has proved to be very difficult.

Most thermal engineers are coming to realize that a slightly lower measure of heat diffusivity is a reasonable trade-off if the cost drops by a wide margin. In many cases the thermal engineer can choose to use more of a lower performing diamond to arrive at the most cost-effective solution for a given amount of heat dissipation. For example, it is often less expensive to use twice as much diamond with a thermal conductivity of 13W/cm/°K (cost of $1.00 per cubic mm), than IIa diamond with a thermal conductivity of 20W/cm/°K (cost of $10.00 per cubic mm).

sp3's experience has shown that its hot filament grown diamond at 10 to 15W/cm/°K meets an important price performance plateau. Thermal systems engineers who rejected diamond in the past as too expensive are now considering it in large quantity.

Processing – Why Hot Filament?

Ease of Scaling

The deposition area of a microwave reactor is, in part, limited by the frequency of the plasma generator. The maximum deposition area in state-of-the-art microwave systems is limited to about 150mm in diameter. Some DC torches are capable of deposition over areas up to 200mm in diameter. The present sp3 hot filament reactor deposits over an area 350mm by 375mm. There are hot filament reactors in Europe depositing over an area about 400mm by 800mm. sp3 is currently considering a reactor that will deposit over an area 1000mm by 1000mm.

Lowest Cost Deposition

Hot filament reactors are straightforward in their execution. DC power is used, the reactors are simple mechanically, and control is well defined. Control mainly comprises gas ratios and flow rates, vacuum level, and the amount of DC power in the filaments. Care must be taken to fixture the reactor correctly to insure that substrates run at proper temperatures. In sp3's experience, careful fixturing design can usually achieve optimum substrate temperatures without the need for substrate heaters or coolers.

When looking at the cost of deposition one must consider the capital investment, operating expense (utilities required such as power and gasses), ease of use, and reactor reliability and availability.

Growth Rate

Hot filament reactors are often perceived to grow diamond at a slow rate. Diamond does grow slowly in any CVD reactor. Hot filament reactors typically grow diamond at 0.3 to 2.0 microns per hour, microwave systems from 1.0 to 5.0 microns per hour, and sp3 experience with DC torches has demonstrated growth rates exceeding 20 microns per hour. It is important to remember that in a hot filament reactor the diamond is being grown over a large area. The definitive measure of growth is carats per hour for a given capital investment, cost of operation such as maintenance and utilities, labor, overhead and power consumption. When measured in these terms, hot filament systems have about twice the efficiency of typical microwave or DC torch systems.

On cutting tools, typical growth rates are 0.8 to 1.2 microns per hour using both two-dimensional arrays for flat tools and three-dimensional arrays for round tools. The sp3 reactor can coat 250 or more flat tools or up to 150 round tools in a reactor load. These large quantities per run help offset the slower growth rate. The typical approach is to turn the reactors once a day and add reactors incrementally as the business grows. On large surfaces such as a 200mm wafer, growth rates drop to about 0.4 microns per hour.

sp3 is currently working on enhancements to bring the growth rate to about 1 micron an hour on large (greater than 100mm) surfaces. Typical substrate temperature during deposition is about 800–900°C. Higher temperatures can increase growth rate but then a whole new set of problems is introduced. Cemented carbides will deteriorate if held at temperatures over 900°C. Higher temperatures also amplify the problems of dissimilar coefficients of thermal expansion. If anything, CVD diamond technologists should be working toward lower deposition temperatures while maintaining present growth rates.

Uniformity

Uniformity is an area where hot filament reactors have clearly demonstrated their superiority over both microwave and DC torch approaches. A typical reactor load in a hot filament system may comprise several hundred cutting inserts, over 100 round tools, or a 300mm silicon wafer. A hot filament reactor has the advantage of uniform temperature across the entire deposition area.

In contrast, microwave reactors and DC torches create a sphere or plume of energy that is hotter at the center than at the edge. System designers compensate for these effects by rotating the substrate to normalize deposition temperatures. Substrates must rotate in a near vacuum, requiring ferrofluidic feed throughs or mechanisms that must move in a near vacuum. Rotation adds complexity and cost, reduces reliability and often is a source of contaminants.

The sp3 hot filament reactor achieves its uniformity performance without rotating the substrate. Substrates in microwave reactors and torch reactors tend to get hot spots on sharp corners of the objects being coated. The hot corners accelerate film growth at the corner, with the result that uniformity within the part suffers. These high corners create problems in tool clamping, and maintaining a consistent edge radius or hone is nearly impossible. Hot filament reactors have clearly demonstrated uniform temperature across large deposition areas and, equally importantly, uniform coatings across an individual substrate.

Safety

As systems get larger, safety becomes an increasing concern. The typical sp3 hot filament system uses 30 kW of DC power, which is an easy power level to manage and control. If there were to be a runaway situation the wires will overheat and break, creating an open circuit. On the other hand, microwave systems operating at 30 kW power levels can be dangerous. If the plasma ball should jump to the walls of the chamber a catastrophic event is the probable outcome.

High power microwave systems also have shown evidence of benzene and benzene byproduct formation during the deposition process. Some of these complex hydrocarbon chains have carcinogenic properties. We have seen no evidence of benzene type hydrocarbons in hot filament systems.

The sp3 systems have both hardware and software interlocks to provide maximum protection to both the operator and the system itself. The controller allows for the setting of limits that, when exceeded, will cause the system to shut down and the event will be logged. The cabinet and chamber doors are interlocked to protect the user and the system cannot be energized if the doors are not fully closed.

Accurate Temperature Control

Temperature control is critical to consistent and uniform diamond deposition. It is easier to govern the voltage on a hot wire for temperature control than to try to govern the proximity of a plasma ball to a substrate in the typical microwave system, especially if temperature control over a large area is critical. Substrate temperature control within +/- 5°C is common in hot filament systems. Accurate control of deposition temperature provides the ability to coat in both two-dimensional and three-dimensional arrays.

Process Control

The combination of a sophisticated control system and a technical approach that lends itself to control has allowed sp3 to develop a reactor with a great deal of flexibility. The system incorporates a process controller developed specifically for the control of plasma CVD depositions in the electronics industry. The controller governs pressure, power to the filament wires, vacuum levels, safety interlocks, etc. The controller can store individual recipes that may have up to 58 process steps and may ramp between steps.

sp3 used this level of control to develop its fine-grain graded layer films. Varying the ratio of methane and hydrogen along with vacuum as a function of time produces these films. The morphology of the films is varied as a function of time. A smooth, contiguous one-micron thick layered nanograin film may be deposited using sp3's patented Graded Layer technology. Process conditions are varied to generate a film composed of multiple 1000 angstrom thick films, each composed of a graded layer structure.

In Summary

For sp3, hot filament reactors have proven to be efficient and cost-effective diamond deposition systems for an ever-widening set of applications. The systems are easy to operate and have proven to be very reliable.

With respect to manufacturing cost, diamond deposition represents only a secondary portion of the total cost of almost any product. In cutting tools, for example, the diamond deposition cost is less than 30% of total product costs.

It is important to choose a deposition approach that provides proper uniformity, control, repeatability, and ease of scaling. Deposition over large areas is often more useful than a focus on microns per hour when evaluating overall growth rates.

The decision made by sp3 many years ago to develop and refine hot filament reactors for CVD diamond deposition has proved to be sound. Experience has shown, time and again, that this approach has provided a reliable, safe and cost effective path to CVD diamond product development and long term production. sp3 will continue to develop, improve and refine the use of hot filament reactors as the best choice for almost all applications of CVD diamond.