sp3's experience using Hot Filament CVD Reactors to grow diamond for an expanding set of applications

James Herlinger, President
sp3 Inc., Mountain View, CA, USA

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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. Prior to forming sp3 the founders spent four years working with both DC plasma and microwave deposition. Previous to this they developed CVD systems for the semiconductor industry.

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. Five years ago sp3 added the capability of growing freestanding sheets of CVD diamond using 100 kW DC torches in a second facility in Calgary, Alberta, Canada.

sp3's tooling effort has expanded into a full offering of diamond cutting tools. In addition to the coated tools, sp3 now offers fabricated tipped diamond tools using both its proprietary CVD thick film diamond (TFd™), often called "sheet" diamond, and traditional polycrystalline diamond (PCD). In summary, sp3 has experience in DC glow discharge, microwave, hot filament and DC torch grown diamond. The selection of hot filament as the reactor technology of choice for coated cutting tools has proved to be an excellent decision for sp3.

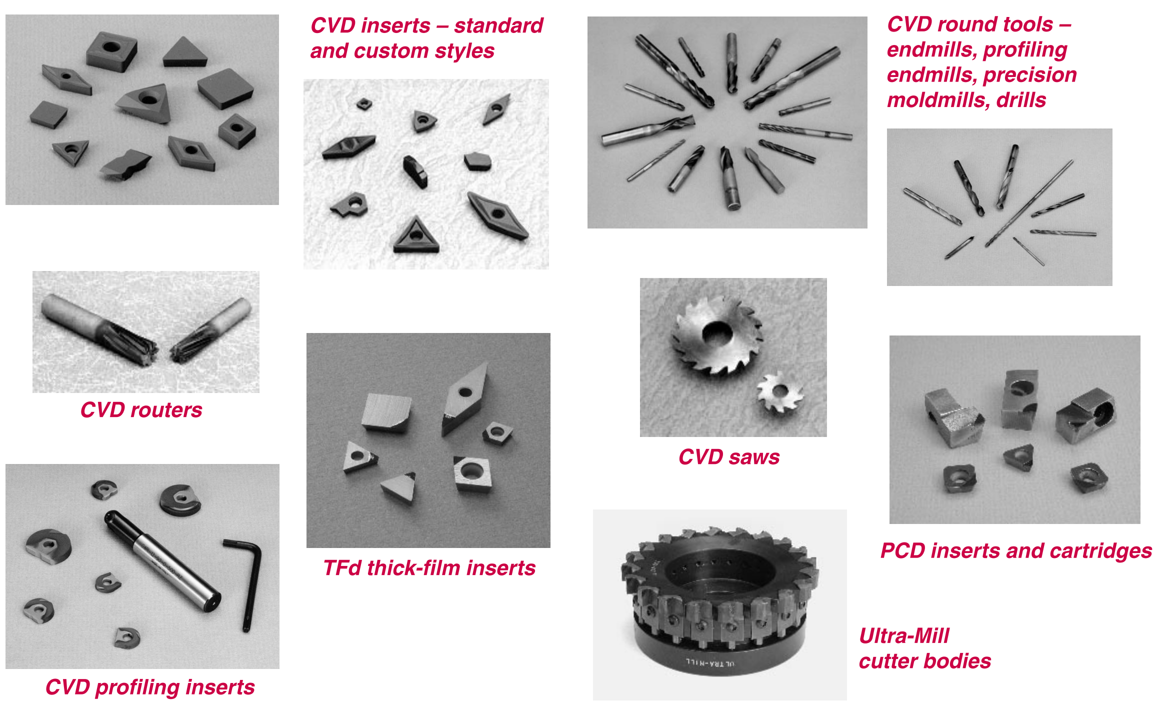


Figure 1. Range of sp3 diamond cutting tools

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. Figure 2 shows some of the details on CVD coatings for cutting tools.

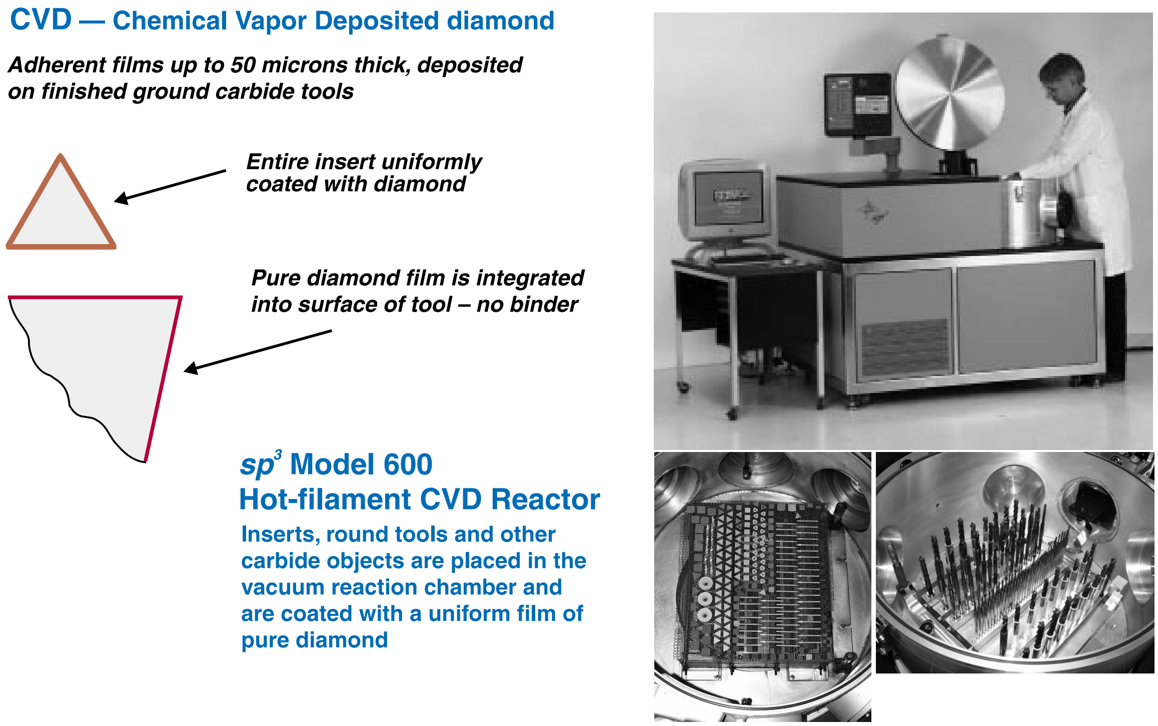


Figure 2. sp3 Model 600 Hot Filament Reactor with insert and round tool loads

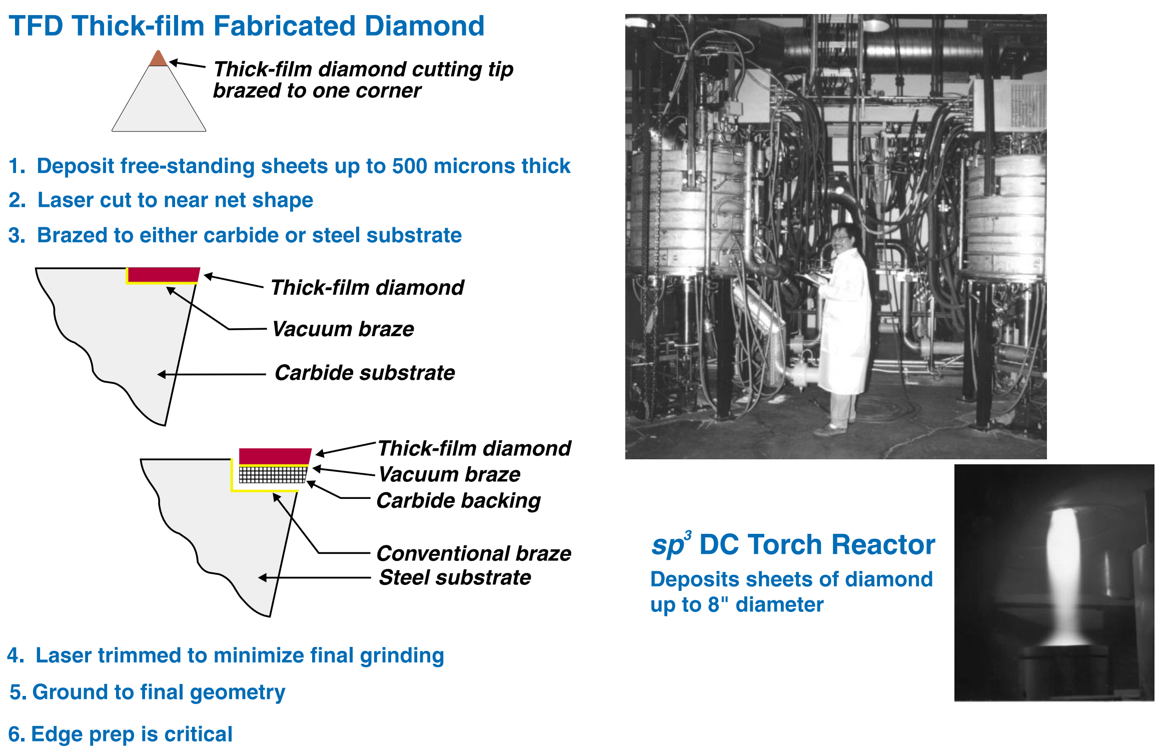


Figure 3. sp3 DC torch reactor

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[1], 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. (More system details are available at www.sp3inc.com.)

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[2] 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 quite easy to vary the grain size as well as other properties of the grown films.

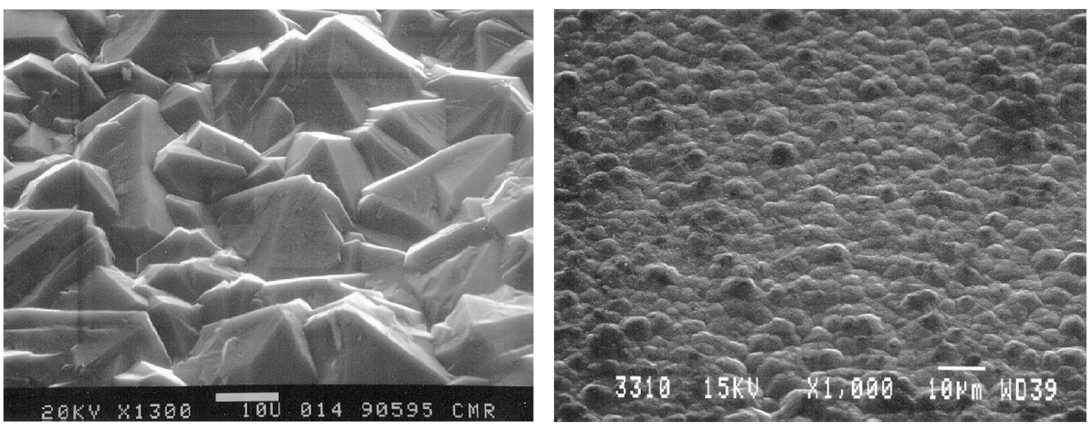
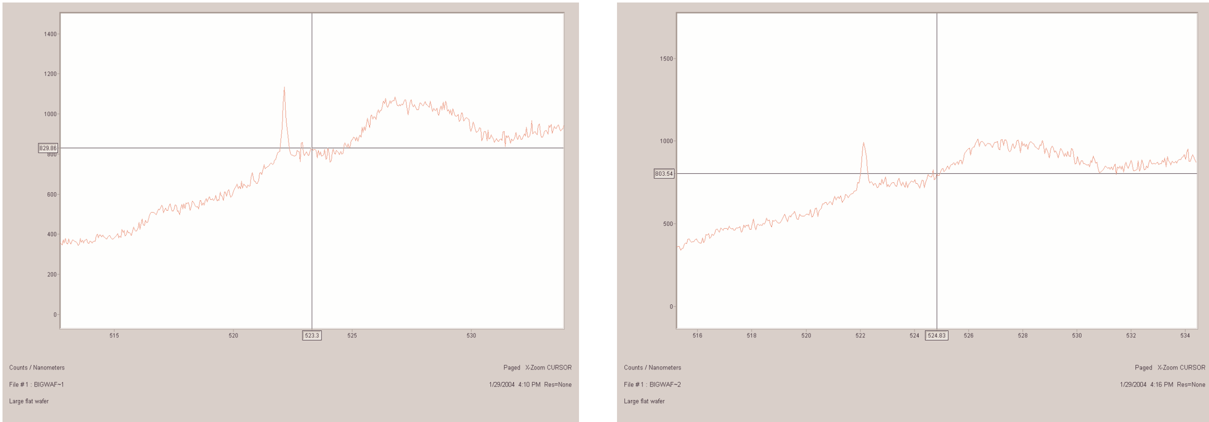


Figure 4. Typical coarse and fine CVD diamond films used in cutting tools.

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 cutting film shown above in Figure 4. 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.

The Raman signature of a 1 micron film grown on a 200 mm wafer is shown in Figure 5.



5a. Center of wafer **5b. Edge of wafer**

Figure 5. Raman signature of a 1 micron film on a 200mm silicon wafer [3].

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.

It should be noted that sp3 also supplies free standing diamond for heat spreaders up to 2mm thick. These thicker segments are grown in sp3's DC torch systems and typically are from 250 microns to 2mm thick, depending on the application. Thermal conductivity exceeds 13W/cm/°K. Some of these typical shapes from sp3's DC Torch system are shown in figure 6.

More information about these products can be found at www.sp3inc.com.

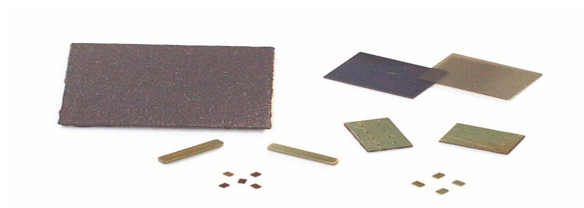


Figure 6. Examples of thick-film sheet diamond products.

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.

sp3 developed a CVD diamond based pad conditioner for preparing the pads used in the chemical mechanical planarization of semiconductor wafers. A thin layer of CVD diamond was grown on a silicon wafer; diamond grit of a specific size was then arrayed on the wafer and an additional layer of CVD diamond was grown, epitaxialy bonding the grit between two layers of CVD diamond. The pad conditioners are supplied in both 50mm and 100mm diameters. This product line was sold to Diamonex (a Morgan Crucible company) and has had some significant market success. It should be noted that this product could only be produced in a reactor with horizontal filaments.

Some typical pad conditioners are shown in Figure 7.

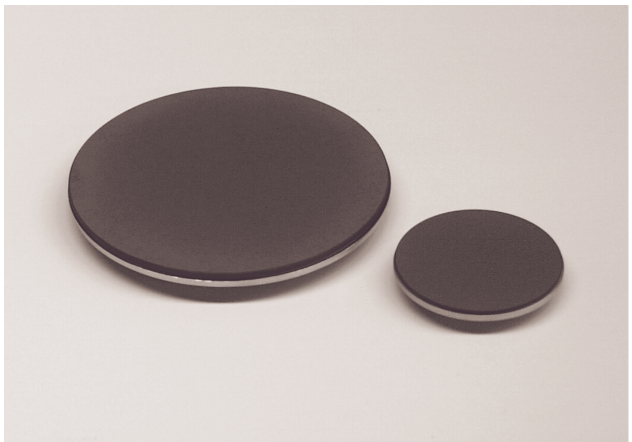


Figure 7. Examples of CVD diamond pad conditioners.

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 tend to 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

Some typical mechanical wear parts are shown in Figure 8 and some seals in Figure 9.

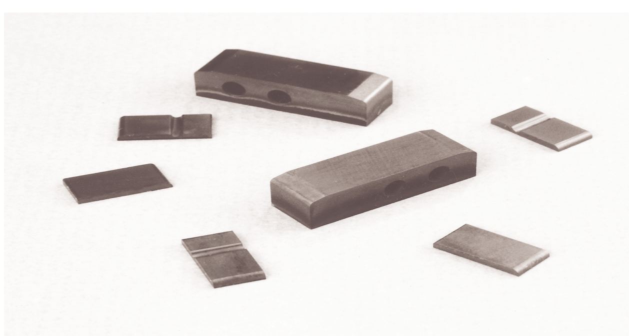


Figure 8. Diamond-coated mechanical wear parts.

Figure 9. Diamond-coated seals.

Film Properties

The films grown are polycrystalline diamond and they exhibit the properties expected of diamond. In terms of wear resistance in cutting tool applications, the tools exhibit wear rates equal to or exceeding that of traditional PCD tools. A great deal of test data showing the relationship between cemented carbide, CVD diamond and PCD diamond is available at www.sp3inc.com.

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 however.

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 a very important price performance plateau that has thermal systems engineers now considering diamond in large quantity where they had rejected it in the past as too expensive.

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. There is no reason this can't be done other than the market has not yet demanded a reactor this large.

Lowest cost deposition

Hot filament reactors are reasonably straightforward in their execution. DC power is used, the reactors are simple mechanically, and control is straightforward. 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. It has been sp3's experience that careful fixturing design can usually result in achieving 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.

sp3's experience is that the capital investment of a hot filament system for a given quantity of diamond grown is about 1/3 to 1/2 that of microwave systems on a cost per carat basis. Only the large DC torch systems grow diamond in bulk at a cost per carat approaching hot filament deposition for a given capital investment. sp3's experience has clearly shown reactor up-time over 96% and reactor availability (defined as deposition time as a percentage of total time) of over 90%. Disposable parts are minimal and the power generating systems are simple DC power supplies.

The sp3 reactor has been designed so that filaments can be easily replaced on every cycle, a procedure which takes about 10 minutes. A set of filaments can be fabricated for less than $10.00. This approach has eliminated the need for a costly and unreliable load locked system. There is an additional benefit of fresh filaments for every cycle—it allows for fine-tuning a given recipe to achieve a specific deposition goal. Load locked systems tend to be less flexible as they are running steady state. This often eliminates some important possibilities. For instance, the nucleation operating conditions may be quite different from the conditions established for primary growth.

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. With respect to growth rate, 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 systems 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 Model 600 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 some 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 try to compensate for these effects by rotating the substrate in an attempt to normalize deposition temperatures. This poses an additional complexity as 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. Another phenomenon observed is that substrates in microwave reactors and torch reactors tend to get hot spots on sharp corners of the objects being coated. This causes an unstable situation that often results in a thermal runaway condition at these sharp corners. The hot corners will accelerate film growth at the corner, with the result that uniformity within the part suffers. The common term on flat tools is "dog boning." 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.

Figure 10 is a schematic representation of some of the uniformity issues.

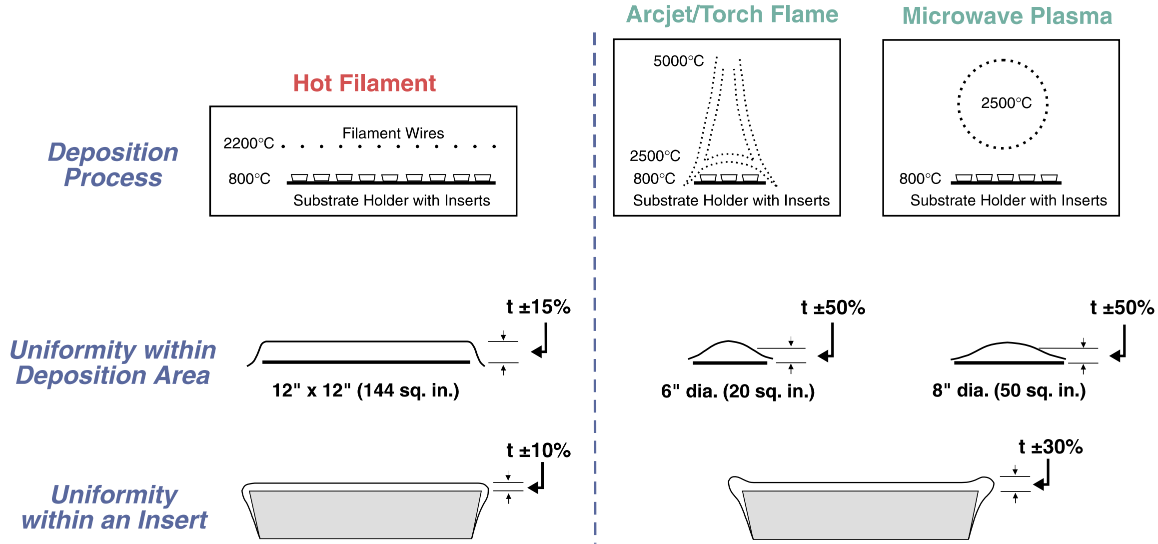


Figure 10. Deposition area and uniformity.

Safety

As systems get larger, safety becomes an increasing concern. The typical sp3 hot filament system uses 30 kW of DC power. This is an easy power level to manage and control when using DC power. 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 maiximum 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. In addition, the system protects against software failures with hardware interlocks. The cabinet and chamber doors are interlocked to protect the user and the system cannot be energized if the doors are not fully closed and the system is at the correct operating conditions.

Accurate temperature control

Temperature control is critical to consistent and uniform diamond deposition. It is much 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.

With a hot filament system, accurate control of deposition temperature provides the ability to coat in both two-dimensional and three-dimensional arrays, as illustrated earlier for inserts and round tools in Figure 3.

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. Please see the sp3 Model 600 Reactor specification for a complete description of the control system. 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[4]. 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.

An example of a fine-grain, graded layer film is shown in Figure 11. This is a smooth, contiguous one-micron thick layered nanograin film deposited using sp3's patented Graded Layer technology. Process conditions were varied to generate a film composed of ten 1000 angstrom thick films, each composed of a graded layer structure. The result is a very smooth, fine grain layered contiguous film.

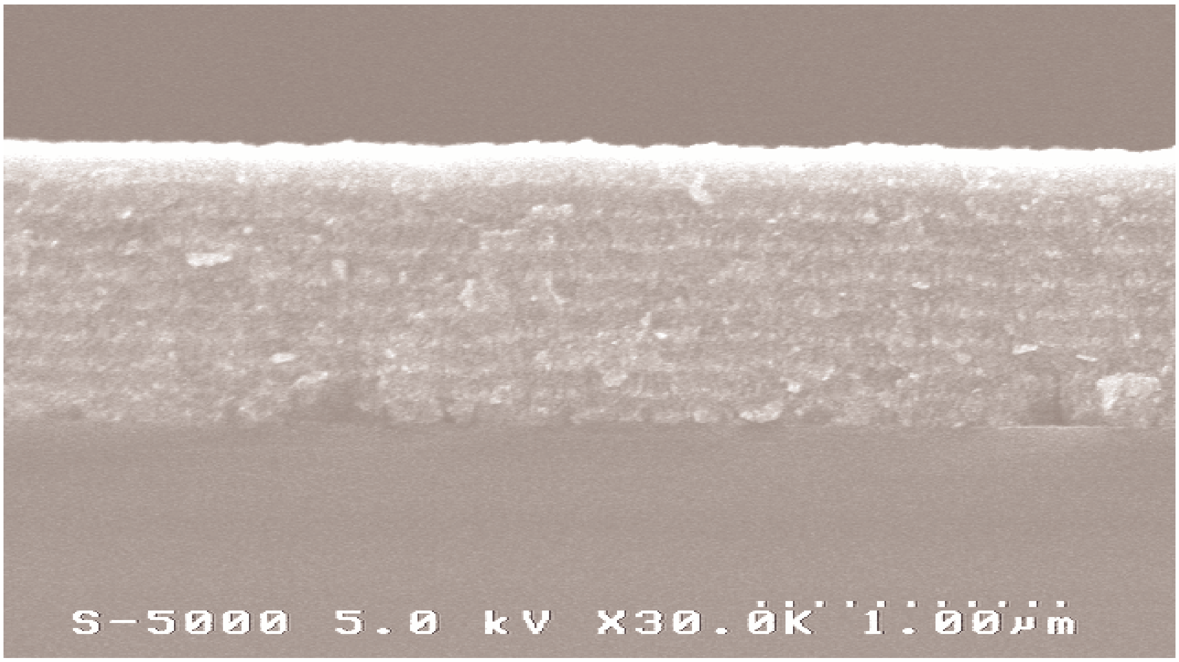


Fig 11. Multi-layered fine grain film.

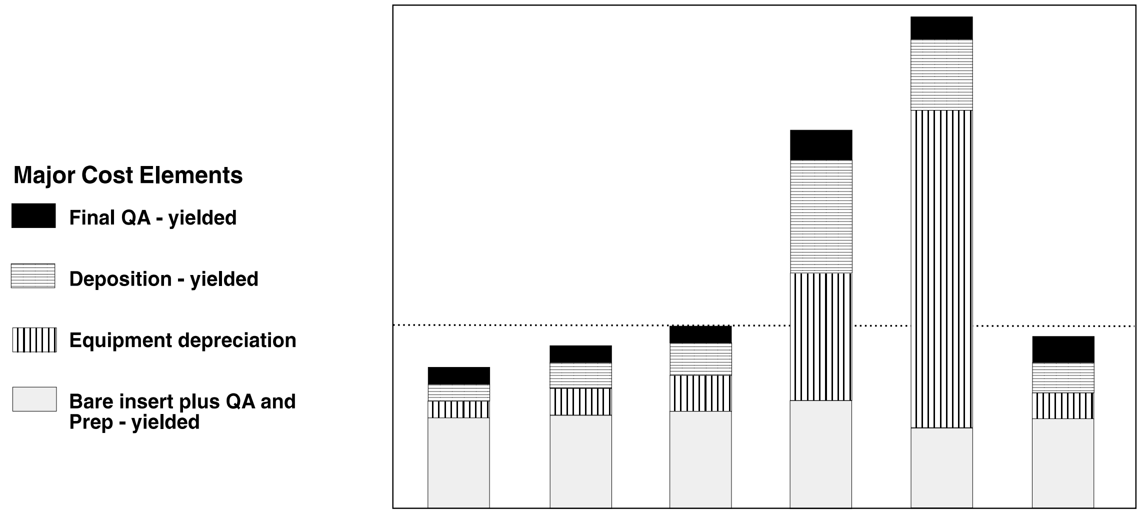
The economics of hot filament reactors

The costs to coat typical cemented carbide cutting tool inserts are compared in Figure 12. The costs are normalized for three different thicknesses of films grown in hot filament reactors and compared to the normalized costs to grow similar films in microwave, laser and arc jet systems.

For the sake of comparison an sp3 Model 600 Hot Filament Reactor is compared with a large microwave system depositing over a 6 inch diameter, a spot laser system such as the one offered at one time by QQC, and sp3's experience with its own DC arc jet systems.

One of the first observations that can be made is that costs for the final parts are not dramatically reduced by making the films thinner. Diamond deposition is less than 30% of the overall cost and it is probably a false economy to grow thinner films as an attempt to reduce cost. The film thickness should be selected based on criteria such as adhesion, edge sharpness and life expectancy, not minimal cost.

Hot filament is definitely the lowest investment, the highest performance, and lowest cost method of deposition. Technical risks are lowest. Microwave is about two times more expensive, it does not deal well with three-dimensional arrays such as end mills, and the systems with their increased complexity (rotation, microwave power generation) are not as robust and reliable as hot filament. The spot laser systems that were discussed several years ago have not worked out. They are expensive and difficult to operate. No demonstration of reliable production has ever been completed. Very complex robotics are required to raster the substrate under the laser beams. Arc jet systems are a close second to hot filament; however, the increased difficulty with temperature control, the larger initial investment, lower reliability and poorer product uniformity tend to rule out this technology for cutting tool applications. These systems are more suited to growing bulk or sheet diamond.



Reactor Type	Hot Filament	Hot Filament	Hot Filament	Microwave	Spot Laser	Arc Jet
Diamond film thickness (microns)	7	20	35	35	35	35
Capital investment incl. installation ($ x 1000)	600	600	600	850	3000	1500
Deposition area (sq. in.)	144	144	144	50	0.04	100
Growth rate (microns per hour)	1.1	1.1	1.1	1.5	1200	10
SPG-322s (3/8-inch IC) per year x 1000	154	87	57	25	35	188
Technical risk	Low	Low	Low	Med. Low	Very High	Med. Low
Overall product performance - scale of 1 to 10	4	6	8	5	1	5

Figure 12. Normalized CVD insert costs compared by deposition type.

sp3's experience in the production environment

Over its production history sp3 has successfully completed more than 6000 individual reactor runs, coating over 1 million tools. Typical reactor availability exceeds 95%, time between runs is usually less than one hour, and more than 99% of runs are completed successfully. After initial setup and start, the systems run unattended, often for over 24 hours when thicker films are required.

The primary reason for any aborted runs results from power outages caused by the local power provider, accounting for over 75% of these aborted runs. It should be noted that the process is very forgiving and the reactor always aborts to a safe condition, with very little scrap. Deposition can be resumed (correcting for total time) in 95% or more of the aborted runs.

Filament Replacement

The Model 600 was designed to have the filaments replaced every time the system is vented to atmosphere. A typical filament is fabricated from 0.005 inch (0.12mm) tungsten wire. The filaments are a simple straight wire with a 1cm 90° bend at each end for tensioning. The wire cost is $250 for 1000 meters. A set of 30 filaments can be fabricated in about 10 minutes with a wire cost of less than $3.00 for the set. It takes about ten to 15 minutes to install the filament in the stretcher frame and position it in the reactor.

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. Hot filament reactors successfully coat both flat and round cemented carbide cutting tools, silicon carbide seals of various shapes and sizes, and silicon wafers for applications from as small as 50mm up to a maximum of 300mm.

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.

sp3 made a fundamental decision many years ago to develop and refine hot filament reactors for CVD diamond deposition, and this has proved to be a sound decision. 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. This important technology has been the cornerstone that has helped sp3 become a leader in the development and marketing of a wide variety of CVD diamond products. sp3 will continue to develop, improve and refine the use of Hot Filament Reactors. They are the best choice for almost all applications of CVD diamond.

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[1] Reference sp3 patents 5,883,753 and 5,997,650.
[2] Reference sp3 patents 6,063,149, 6,319,610 and 6,533,831.
[3] Raman analysis courtesy of Jim Butler at the Naval Research Laboratory.
[4] Reference sp3 patents 6,063,149, 6,319,610 and 6,533,831.