Evolution of Diamond for Optical Component Finishing

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Monocrystalline diamond, both natural and synthetic, has been widely used in the lapping and polishing steps of optical component manufacturing. The structural defects inherent in monocrystalline diamond will eventually limit usage in next generation products. Recently, diamond manufacturers have made some key morphology refinements to compensate for the limitations of monocrystalline diamond. As a result, engineers are achieving higher removal rates and finer, scratch free surfaces. We will examine, from a form-function perspective, the evolution of diamond into these fascinating new abrasive types.

Nanoparticle Technology

Nanoparticle technology will play a key role in diamond synthesis as the functional utility of all diamond types is increasing with decreasing particle size distribution. A diamond with an average diameter of 0.100 micron or less is now measured in nanometers. Nanodiamond has more pieces per carat than traditional micron diamond used in finishing (Table 1). How small is nanodiamond?

	One Carat 100 nm Diamond	One Carat UDD diamond
Pieces per Carat	1.1 x 1014	5×10^{17}
Equivalent pieces 3.0 micron diamond	44lbs	22 tons
30 micron diamond	22 tons	22,000 tons
Bermuda Beach Sand	5,000tons	22,000,000 tons

Table 1

One carat (200mg) of 100 nanometer (nm) diamond abrasive contains slightly over one hundred trillion pieces! Since the number of pieces per carat is proportional to the cube of the radius, the quantity of three micron diamond equivalent to the number of pieces in 100nm diamond jumps to 100,000 carats or roughly 44 pounds. The equivalent mass of 30 micron diamond, a size typically used in cutting wheels, is 22 tons.

Another diamond type that will be reviewed later, Ultra Dispersed Diamond, (UDD) contains about one hundred thousand trillion primary particles per carat. If we compare this number to 300 micron Bermuda beach sand at 4,000 pieces per 200 mg, the equivalent mass now becomes 22 million tons (Figure 1). This mass will fill a 50-yard beach one foot deep with dry-packed sand 550 miles long, or roughly 14 times around the entire Bermuda perimeter. Certainly, a carat of UDD may be added to the old adage "as much as the stars in the sky or grains of sand on the beach."

Monocrystalline Diamond

Monocrystalline diamond is a highly ordered crystalline solid. The carbon atoms are linked together in a regular fashion. Each atom shares one of its outer shell electrons with four other carbon atoms in an equally spaced tetrahedral environment. Monocrystalline diamond, both natural and synthetic, was the primary abrasive source of the finishing industry before the turn of the millennium. By volume, synthetic monocrystalline diamond (SMD) is used 30 times more than natural diamond for two reasons: cost and supply. In order to meet industry volume demands, artificial synthesis requires pressures exceeding 60 kilobars to emulate the production

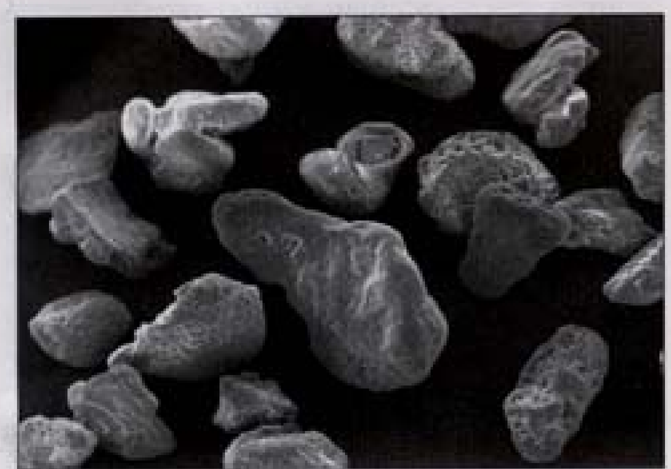

Figure 1. Size comparison of Bermuda beach sand with Ultra Dispersed Diamond. One carat of UDD has as many pieces as the surface of a beach 550 miles long.

Figure 4. Sliver resulting from the cleavage of the 011 crystal plane of diamond.

conditions of diamonds found in nature. First attempts to convert graphite to diamond at low temperature and high pressure were unsuccessful. The reaction speed was too slow for the process to be considered cost effective. To increase the reaction kinetics, higher temperatures were needed, which, in turn, required even higher pressures. The reason for this dilemma is due to the positive slope on the P vs. T phase diagram along the graphite/diamond stability line (see Figure 3). The transition conditions certainly influenced the evolution of the modern diamond press that has 50 years of refinements since its birth in the 1950s by GE.

Nickel catalysts are now used to lower the temperature to about 2,000K and 60 kbar. Mechanically, presses differ slightly throughout the world, however, all SMD production will fall within the shaded region of the phase diagram (Figure 3). SMD production factories may have hundreds of presses since the reaction chamber is quite small. One synthesis may produce fewer than a hundred carats per run. The raw diamond then must be crushed, milled, cleaned and graded.

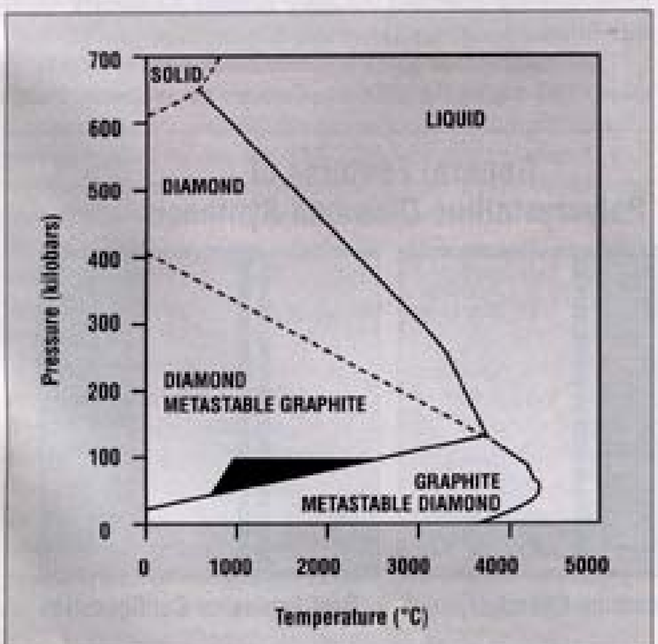

Figure 3. Phase diagram of carbon showing the shaded region of synthetic diamond production.

SMD has a good material removal rate, which increases with particle size distribution. Workpieces finished with SMD are the roughest compared to other forms of diamond outlined in this article since the edges are: large, in comparison to the total crystal size; sharp; not friable. It is this toughness that enables micron and submicron SMD to retain some of the shape characteristics, such as pyramids and trigons, of the parent crystals after the crushing and milling process. The probability of diamond cleavage increases along defect lines or impurity regions. However, SMD is resistant to acids and hot caustic solution so the surface can be cleaned to low impurity levels. Batch -to-batch quality from SMD suppliers is consistent. The cost per carat of SMD is the lowest. Prices are currently 0.25-0.75 per carat, depending on cleanliness and standard deviation.

The disadvantage to SMD is the inclusion of slivers (needle-like pieces) and plate structures (thin, flat pieces) in feed batches. Slivers or plates form when the 011 crystal plane cleaves during the crushing process (Figure 4). When the orientation of these elongated structures is perpendicular to the workpiece during finishing, sub-surface damage may occur from the excessive pressure at the point (Figure 5). Irregular finishing patterns and lower removal rates can also occur if a plate glides horizontally across the workpiece.

Reducing excessive slivers and plates from a SMD feed batch presents a considerable challenge to diamond suppliers. The reason is that the plates and slivers are hydrodynamically equivalent to smaller, more regularly shaped pieces. During elutriation, slivers gather momentum from the added frictional component and carry over into the graded batch. This phenomenon is similar to drop-

Figure 5. Diamond-workpiece interactions during the lapping or polishing process. Synthetic Monocrystalline diamond slivers that interact perpendicularly with the surface can lead to sub-surface damage from excessive pressures at the point. If the direction is parallel to the workpiece, removal rate will decrease. Polycrystalline diamond of the same size as monocrystalline diamond will contact the workpiece with "microcrystallites" instead of flat planes or sharp points. The result is an increase in material removal rate compared to SMD. Heat treated monocrystalline diamond has excellent removal rate with less scratching since sharp points are lubricated with a graphite shell. Ultradispersed diamond is used for ultrafine finishes. There are no large irregularly shaped pieces present in UDD.

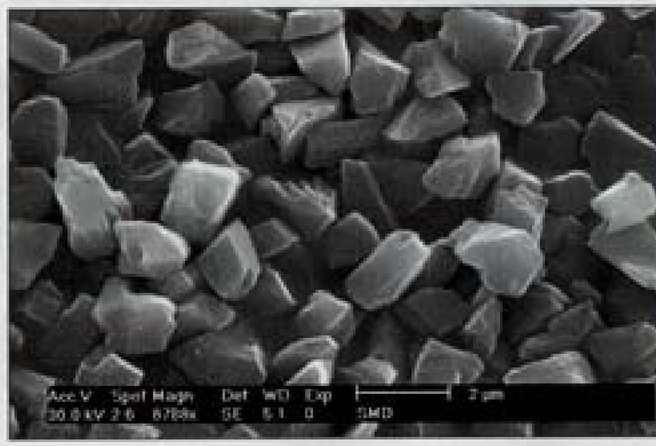

Figure 6. SEM of milled monocrystalline diamond with a reduced sliver concentration.

ping two pieces of paper, one crumpled and one flat, from shoulder height to the ground. The crumpled paper will hit the ground faster since the frictional force of air "floats" the flat paper. Slivers and plates may be removed in milling if the energy selected is above the cracking energy of the thin pieces, and below the cleaving energy of regular-shaped pieces (Figure 6). The smaller fragments will then be removed in the grading process. Extra tight distributions add significant time and cost to SMD powders.

Polycrystalline Diamond

Polycrystalline Diamond (SPD), unlike SMD, is produced via an explosive shock synthesis (Figure 7). Several tons of explosives are used to generate about 250 kilobars (equivalent to about 3 million psi) of pressure on the graphite feed. Each SPD piece contains smaller diamond "microcrystallites." The microcrystallite planes are oriented in different crystallographic directions every 10-50nm, regardless of the particle size distribution of the parent pieces. The hardness of an individual microcrystallite is comparable to that of SMD. The difference between SPD and SMD is discernable in SEM, however, transmission electron microscopy (tem) allows the operator to clearly see the microcrystallite structure (Figure 8).

SPD has a higher material removal rate (mrr) than SMD. Most studies show that the mrr is up to 10 times that of SMD. The reason is that SPD has more cutting edges and higher surface area than SMD. Multiple edges and corners are in contact with the workpiece simultaneously, thus reducing the probability of sub-surface damage from excessive pressures (Figure 5).

The most obvious disadvantage is the sticker shock. SPD costs on average 10 times as much as SMD (2.10 to 8.00 per carat). However, dollar losses up front can be easily recovered by using less diamond in the finishing process, not to mention the labor savings for shorter mrr times.

In comparison to SMD, batch-to batch consistency is more challenging for SPD suppliers for two reasons. First,

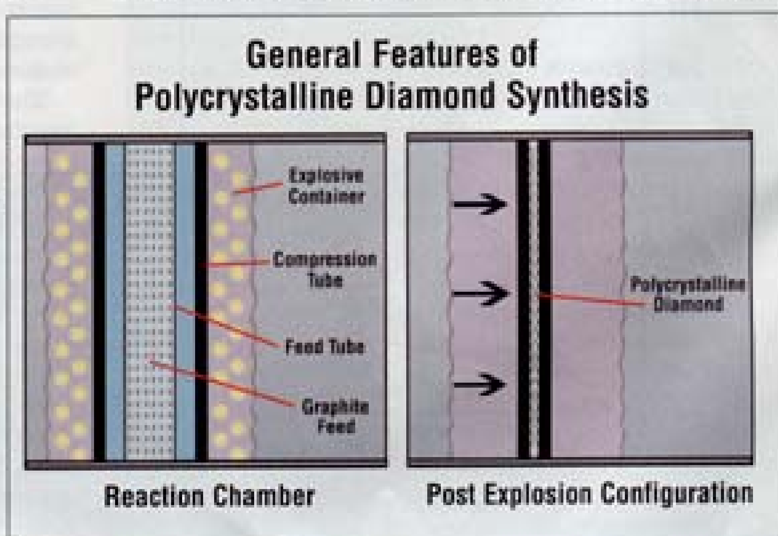

Figure 7. General features of polycrystalline diamond synthesis. The transition occurs via an explosive compaction process.

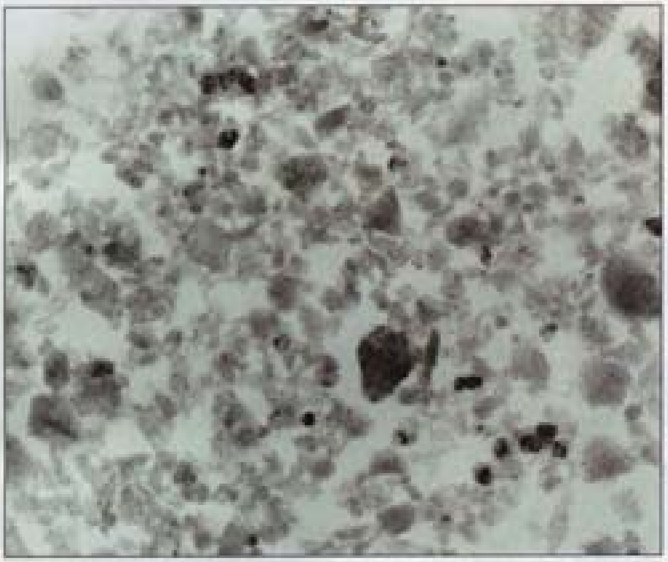

Figure 8. TEM of polycrystalline diamond showing the microcrystallite structure of the parent piece. The bar represents 100nm.

Figure 9. TEM negative of transitional carbon that is present in low concentrations in polycrystalline diamond batches. Transitional carbon is SPD oriented in one direction. The piece lacks the microcrystallite structure of polycrystalline diamond.

unlike SMD, the edge sharpness and crystallinity can vary with milling conditions. Under-milled material tends to produce a rougher surface with a relatively (in comparison to SPD, but far fewer than SMD) higher scratch rate. Over-milling dulls edges and lowers mrr. Secondly, impurity pieces of transitional carbon often reach unacceptable levels. The transitional carbon can be thought of as SDP oriented in one direction (Figure 9). The only drawback of transitional carbon is lower mrr. Transitional carbon can easily be spotted in the SEM, while TEM analysis is required to control and select batches with consistent edge definition.

Heat Treated Synthetic Monocrystalline Diamond

Heat Treated Synthetic Monocrystalline Diamond (HTSMD) is manufactured by heating well-graded and cleaned SMD in an inert atmosphere to about 1,200°C. Under these conditions, the powder darkens from light grey to black (Figure 10). The color change represents a reorganization of the surface into a more disordered, graphite-like layer (Figure 11). The particle size distribution of HTSMD is often tighter than SMD and SPD since unwanted pieces on the fine end tend to fuse into larger clusters or disappear completely in the heat treating process.

The graphite sheets provide a lubricating region between a sharp diamond edge and the workpiece (Figure 5). This shell coating lowers the scratch and defect rate compared to SMD. Recall that slivers in SMD are difficult to completely remove from a batch since they are hydrodynamically equivalent to well-shaped pieces. Since the entire batch is engulfed in the high temperature oven, this process shapes and lubricates all slivers that would otherwise scratch or damage a surface. MRR is comparable to SPD. However, HTSPD has one distinct advantage over SPD: reduced diamond embedding in the workpiece. Customer feedback indicates that work-

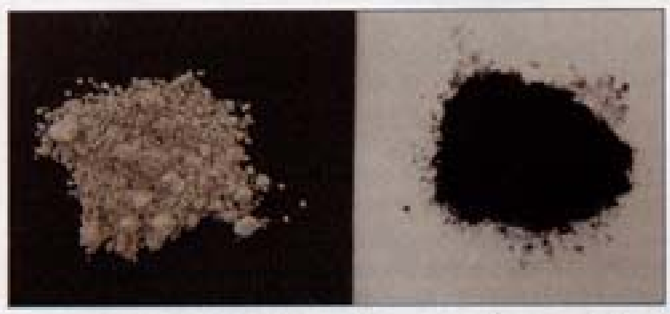

Figure 10. Color comparison between SMD (white) and HTSMD (black).

pieces have up to 33% less embedding when using HTSMD compared to other diamond sources.

Great care must be taken when preparing dispersions of HTSMD. The heat treating process decreases the surface polarity resulting in a tighter stability region. In fact, the heat treated diamond must undergo a proprietary surfactant soak to add polarity to the surface or the material will settle clear in an aqueous environment in a matter of minutes. The cost is about 10% less than comparable SPD, but the benefits, once again, more than compensate for the price.

Ultradispersed Diamond

Ultradispersed Diamond (UDD) is produced in the diamond stability region at T 3,000K and p 100kilobar. However, there is one critical process difference that distinguishes UDD from other diamond types. The feed, explosives and UDD product are contained within the explosion chamber. The chamber and explosives must be oxygen deficient (17 pieces per carat (Figure 12). The diameter of the pieces falls within a 2 to 10 nm range with the predominant size being 3-7nm. This incredibly small size range has led to some nomenclature inconsis-

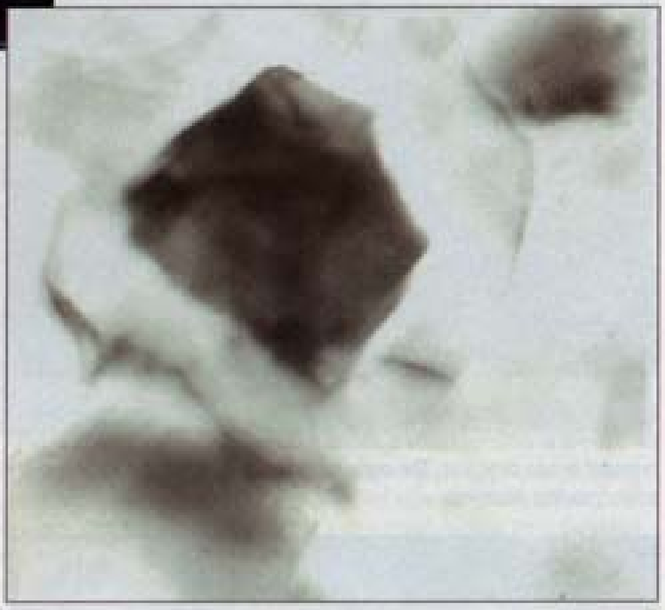

Figure 11. TEM of HTSMD showing the diamond core and graphite outer shell. The bar represents 10nm.

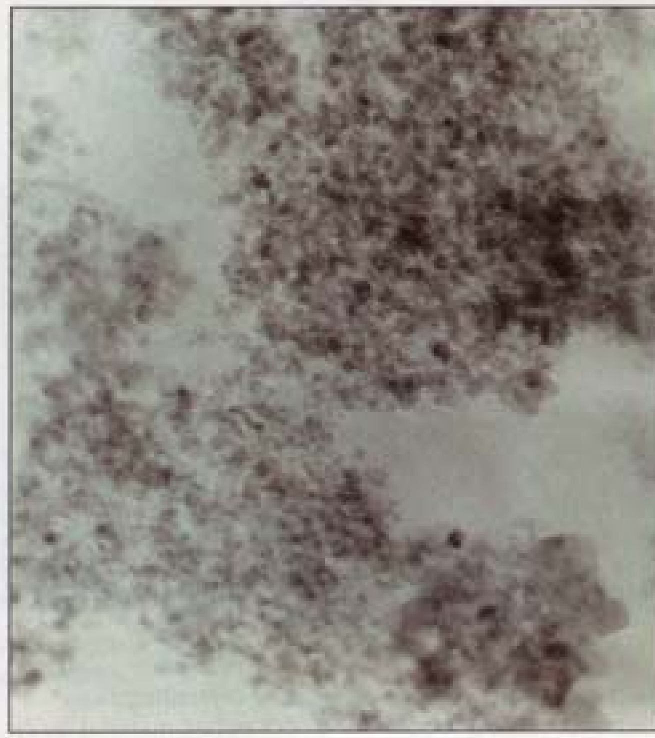

Figure 12. TEM image of UDD diamond. The size of the primary particles ranges from 3-7 nanometers. The bar represents 20nm.

tencies. UDD is often referred to as "nanodiamond." The term nanodiamond is a much broader term that encompasses all diamond types. Diamond that is nano-sized is readily available from SMD, SPD and HTSMD suppliers. UDD is a particular class of nanodiamond. Another term referring to UDD that is gaining popularity is "cluster diamond." This description is unique to UDD as long as the terminology reflects a clustered nanodiamond and not nanocluster diamond. The reason for the distinction is that UDD is composed of primary and secondary particles. The primary particles cannot be broken down to smaller sizes. The primary particles exist in suspension as clusters. The cluster size can range from a few primary particles to a few microns.

Since UDD is relatively new on the open market, performance data - compared to other diamond types - is a work in progress. However, UDD is expected to be the most tested diamond type in 2005, so the data gap will quickly close. Initial results indicate that mrr is relatively low, but this is expected when finishing to angstrom or sub-angstrom roughness with nanoparticles. Finishes are relatively scratch-free because rogue slivers are not produced in the UDD manufacturing process (Figure 5).

Another characteristic advantage of UDD particles is that the edges are not subject to the attrition variability inherent in all other diamond types. UDD particles are produced directly in the explosion process, unlike all other diamond types, which are chips from parent pieces. Control of cluster size appears to be the primary critical to quality parameter for UDD products. Finishing quality will change with cluster size. We are currently working on many development projects including product characterization for quality control purposes at our Northboro MA research and development facility, and a new "grow and control" technology at Warren/Amplex Superabrasives. Although the details are beyond the scope of this article, one of our technical experts can expand on this new technology by contacting an "Ask the Expert" on the Warren/Amplex Web site.

Clearly, all diamond types have advantages as well as challenges. There is a fine line between optimum performance and cost effectiveness. Whether considering process improvements or new state-of-the-art finishing applications, it is best to gage the above performance comparison by assuming that all other conditions including machine, work pressures and slurry are constant. Diamond performance can also be controlled with the slurry chemistry. A slight change in dispersion or slurry viscosity can reduce scratch rate and/or increase mrr. The performance summary in Table 2 can be used as a guide when selecting diamond for new or next generation finishing processes.

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