

# Sintered Diamond

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## Introduction

During the past few years, researchers have learned to reconstitute diamond powder into useful products. These different products are proving to be useful with some applications being competitive with single crystal diamonds. For this reason, if for no other, anyone dealing with diamonds would be interested in the new materials.

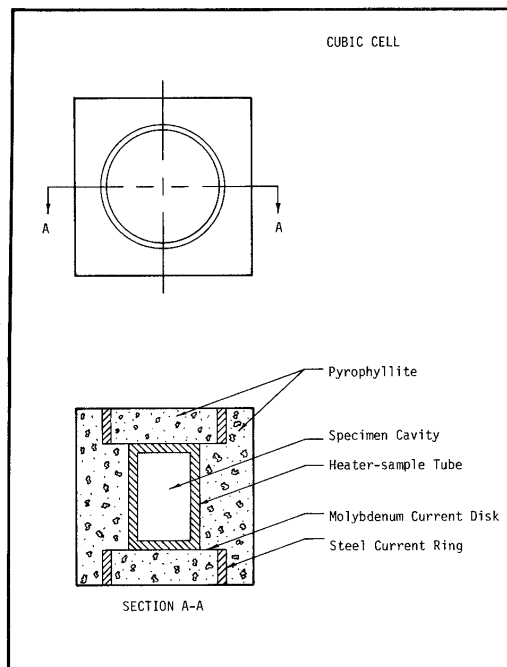
Large quantities of fine diamond powders are the by-product of sizing and shaping operations carried out on both natural and synthetic diamond crystals. Because fine diamond powders have relatively limited applications, there is a resultant large excess of supply over demand. This leads to the powders having a comparatively low value.

Extensive research has been devoted to developing a process that can take cheap diamond powder and reconstitute it. Several processes have been developed and, judging from the resultant materials, each process is different. Were information available, this article would deal with such materials in general. However, proprietary secrecy has severely restricted the available technical material. For that reason, the balance of the article will deal with a particular sintered diamond product called Megadiamond.

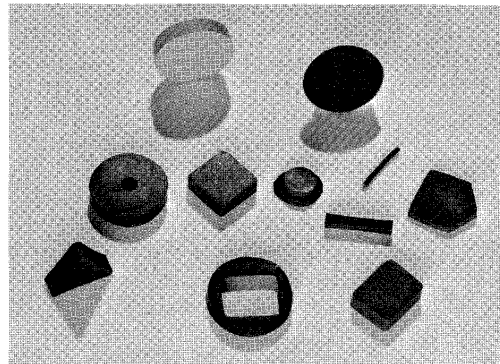
Described in the next section will be the bonding process whereby diamond powder can be fused together to produce large, coherent pieces of a desired shape. Subsequent sections will then describe the properties of the sintered diamond material.

## Process

The sintering of the diamond powders is accomplished by placing the suitably prepared powders in a mold. Then, the mold is placed in an ultra-high pressure press and subjected to a very high pressure. While in this high pressure field, the diamond powder is heated to such a temperature that it fuses together. Of course,



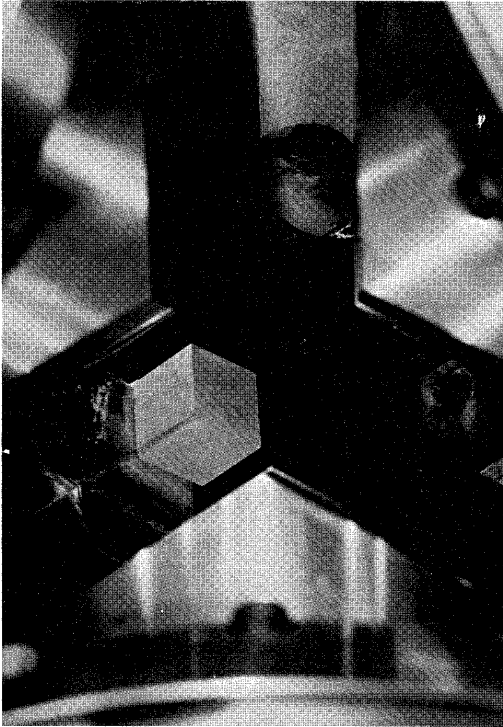
**FIGURE 1.** Typical mold used to make a sintered diamond.



**FIGURE 2.** Examples of sintered diamond shapes.

each of these steps is more complex than described but this is an overview of the process.

From the information presented thus far, the reader would conclude that only fine diamond powders can be used together. This is not so. Particles as large as 150 and as small as 0.1 microns have been tested and successful fusion



**FIGURE 3.** Cubic Press closing on mold shown in Figure 1.

was achieved. However, in all cases, it was necessary that the diamond be properly prepared.

Also, there are almost no restrictions on configuration of the mold into which the diamond is placed. Figure 1 shows the mold used to make a cylindrical sintered diamond and Figure 2 shows several of the shapes that have been made. In many cases where a complicated shape is to be made, the greatest difficulty lies not in sintering the diamond but rather in preparing the mold.

Figure 3 is a picture of a cubic press closing upon the sample of Figure 1. Once the press is closed, the hydraulic pressure behind the rams is increased until the desired pressure inside the mold is achieved. Then, an electrical current is passed through the heater tube to create the required temperature for the necessary length of time. Next, the pressure is released and the press opened. After this, the mold is retrieved then the sintered diamond product recovered.

A wide selection of pressure, temperatures and times can be used

although better results are obtained if the temperature is high enough that the diamond tends to revert to graphite and the pressure is as high as possible. Reference to the equilibrium diagram for the graphite-diamond system shown in Figure 4<sup>1</sup> will show that at any pressure a high enough temperature will cause diamond to convert to graphite. At room pressure and temperature graphite is more stable than diamond but the rate of conversion is infinitely slow. Above 1500°K however, the conversion rate becomes significant and above 2000°K very fast. Nevertheless, the sintering process works best when the temperature is slightly higher than the apparent equilibrium value.

Figure 5 shows a photomicrograph of the polished surface of a sintered diamond. It is visually apparent that the diamond particles have softened and the contact points fused together. This is astonishing since diamond has not been known to melt or even to soften. Temperatures that might be high enough to cause softening seem only to cause a rapid conversion of diamond into graphite, even at high pressures.<sup>2</sup>

Thus the bonding process contains two apparent peculiarities. First, the best bonding occurs when the temperature is such that the diamond ought to convert to graphite. Second, the diamond seems to soften, flow and fuse under conditions not known to make diamond behave in that fashion.

A more detailed consideration of the process suggests a rational explanation of these phenomena. Ordinarily, the pressure is estimated by considering the sample to be homogeneous

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Duane Horton was born in 1935 and raised in Salt Lake City, Utah, U.S.A., where he attended the University of Utah. There he obtained his B.S. and Ph.D. in Chemical Engineering, the latter degree being awarded in 1961. After graduation he worked in the field of solid propellant combustion at China Lake, California. In 1963 he accepted an appointment at Brigham Young University in the Department of Chemical Engineering. There he began work in the high pressure field under the tutelage of H. Tracy Hall. Subsequently, he became interested in diamond, its synthesis, uses, and properties. Ultimately, this interest caused him to join Tracy Hall and Bill Pope in forming a company to develop and exploit sintered diamond. Presently he occupies dual positions as Professor of Chemical Engineering and Vice President of Megadiamond Corporation.



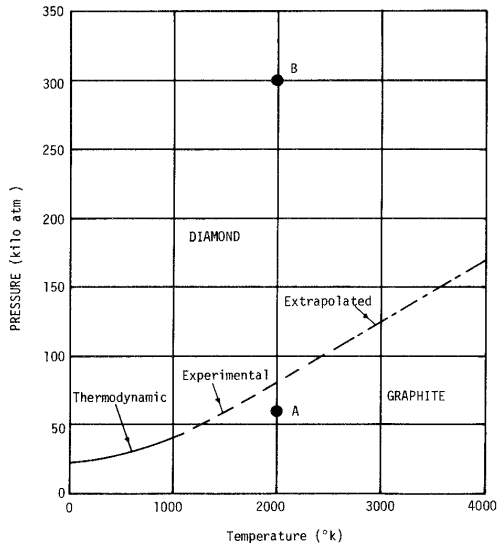
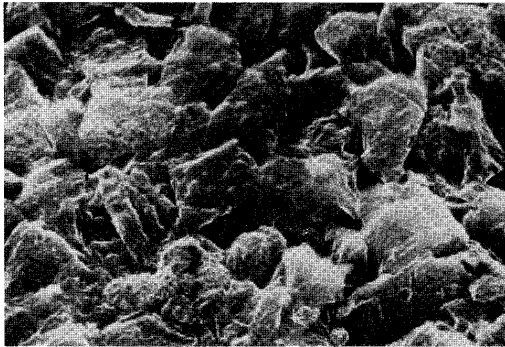
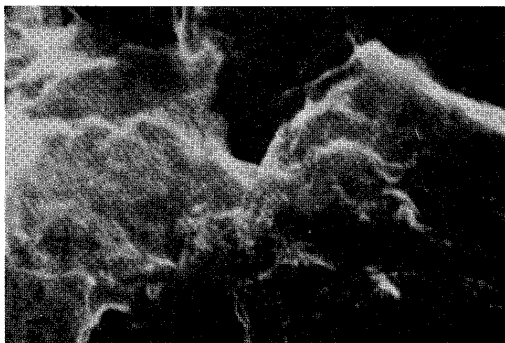


FIGURE 4. Solid carbon phase diagram.



5300 X



35000 X

FIGURE 5. Enlarged views of sintered diamond surface.

and to transmit the pressure hydrostatically. This estimated pressure is low enough that the diamond should transist to graphite. However, the sample is not homogeneous nor is the pressure within the diamond powder transmitted hydrostatically. Instead, the externally applied force is concentrated at the diamond contact

points and the resultant pressure at these points is so high that diamond is stable.

This can be understood better if a specific case is examined. For example, one where the pressure applied to the exterior of the diamond powder sample is 60 kilobars and the temperature is 2000°K (Point A on Figure 4). Figure 6 depicts an idealization of a cut-away view of the sample under these conditions. The forces on any segment of the sample must be balanced so that the "hydrostatic" pressure times the end area of the sample must be equal to the sum of the forces exerted on the diamond contact points. During the compression and packing process, these contact areas become as large or perhaps twenty percent of the cross sectional area. Therefore

$$\begin{aligned} (\text{Applied pressure}) \times (\text{End area}) &= (\text{Contact pressure}) \times (\text{Contact area}) \\ \text{or } (60 \text{ kbars}) \times (\text{End area}) &= (\text{Contact pressure}) \times (0.2 \text{ end area}) \end{aligned}$$

and the pressure at the contact points would be 300 kbars. Thus, point B on Figure 4 is actually representative of the conditions at the contact points. It can be seen that this is well within the diamond stable regime so at the contact points where the fusion takes place, diamond is the stable species and no graphitization occurs to hinder the fusion process.

Also, we suggest that diamond does achieve a degree of plasticity at 2000°K but it is not ordinarily observed because the formation of graphite obstructs observation. Only when graphite formation is prevented by the special and unexplored conditions described above can the softening be observed or inferred. More particularly, only under these conditions can the slightly softened diamond be forced together for a period long enough that distortion and fusion can take place. By this explanation, the

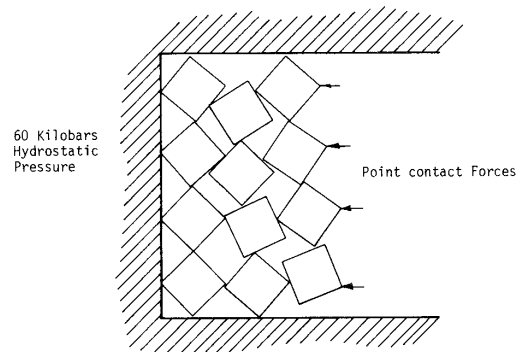
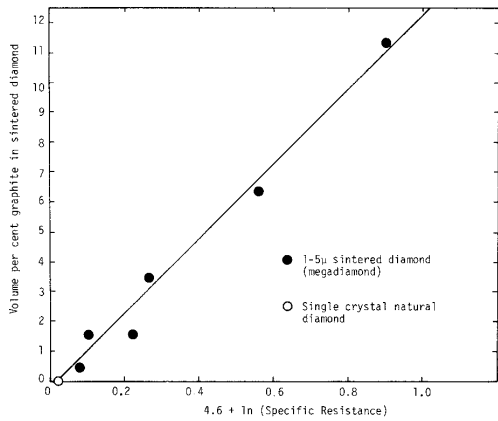
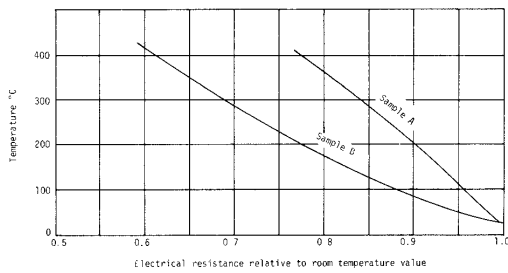


FIGURE 6. Idealized depiction of sintered diamond cross section.



**FIGURE 7. Specific resistance (in ohm-cm) versus graphite to diamond ratio in sintered diamond.**



**FIGURE 8. Sintered diamond electrical resistance as a function of temperature.**

peculiarities of the bonding process disappear and are replaced by ordinary sintering behavior that is often observed with other materials.

It has been found that (See Figure 5) if pure diamond powder is used to make a sintered piece, there are many minute holes left in the matrix with the resulting apparent density ranging from 3.1 to 3.48 g/cm<sup>3</sup>. A stronger sintered product can be made if these holes contain a filler material. This is done by mixing diamond powder with another powder such as boron carbide or silicon carbide prior to placing the mixture in the mold. When the mixture is sintered, the diamond-diamond contact points fuse as described above. However, additional bonds between the filler and the diamond contribute further strength to the composite material.

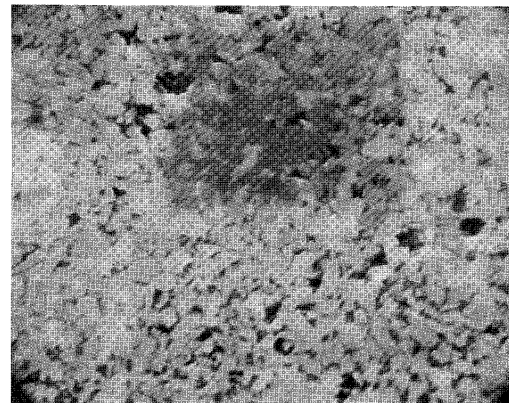
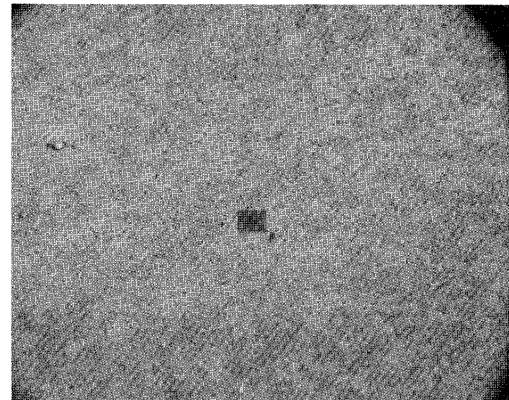
### Electrical Properties

From a scientific point of view, the unusual electrical properties of some sintered diamonds are perhaps the most interesting. These properties are the result of a thin graphite film that forms on the diamond powder surfaces adjacent to the microscopic holes. Such a film

forms in the manufacturing process because at these surfaces, there is not a pressure intensification effect. Therefore a high temperature causes some graphitization of these diamond surfaces.

The most obvious electrical property of the reconstituted diamond is that it is conductive, and the conductivity correlates with the graphite content as is shown in Figure 7<sup>3</sup>. The amount of graphite and hence the electrical conductance can be controlled by proper selection of manufacturing parameters. Use of either a lower pressure or a longer heating time increases the thickness and hence the total amount of graphite film covering the particles. Interpolation of Figure 8 indicates that the electrical resistance (the inverse of conductance) is linear over the extraordinarily large temperature range of 400°C. Thus the material has the properties of an excellent thermistor.

Curiously enough, the sintered diamond can be either a P or N type semiconductor with the amount of graphite present determining the type. Regardless of the amount of graphite present, the



2000 X

**FIGURE 9. Photomicrograph of Electron Beam Image Effect.**

material luminesces with a blue light when subjected to a high voltage.

An even more peculiar property is illustrated in Figure 9 which is a photomicrograph of a polished Megadiamond surface upon which an electron scanning microscope beam had focused. The larger dark image represents a discoloration produced at a lesser magnification while the darker, inner image resulted from the beam of a greater magnification. The surface retained the "photograph" of the beam but the cause of the retention is not known.

### Other Properties

Not only the electrical but also the physical properties of sintered diamond are interesting. In the preferred form, micron-sized diamond particles are fused together. Consequently, on a macroscopic scale Megadiamond is homogeneous and isotropic. In this aspect the material is similar to carbonado. By contrast, single-crystal diamonds have different properties along different crystal axis. Being composed of randomly oriented particles, sintered diamond has a hardness and wear resistance representative of the "average" properties along the various axis of a diamond crystal. This causes it to be, like carbonado, extremely wear resistant if the wear forces are not too great. Another similarity is that, like carbonado, sintered diamond is much tougher than single crystal diamonds and does not fracture readily. When it does fracture, a rough, irregular surface is formed. Also, like carbonado, the material usually has a dark, almost black, color. If the manufacturing parameters are carefully controlled a light gray color results but this result is achieved with difficulty.

Probably due to the graphite present in sintered diamonds, they are wetted by some solders\*. This property contrasts with that of other diamond which is not wetted by conventional solders.

In one way, sintered diamond is similar to single diamond crystals. Diamond has the highest known thermal conductivity (9 watts/cm°C for type I and 25 for Type IIa at room temperature) and that of sintered diamond can be nearly as large (8 watts/cm°C).<sup>4</sup>

The chemical properties of sintered diamond are essentially the same as those of diamond crystals. The only two known differences are

slight. First, the graphite film can be leached from the sintered piece by the use of a powerful oxidizing solution. This leaves a mechanically strong, white, electrically resistive sample. Second, the sintered diamond oxidizes more readily than natural diamond; probably because of strains resulting from the sintering process.

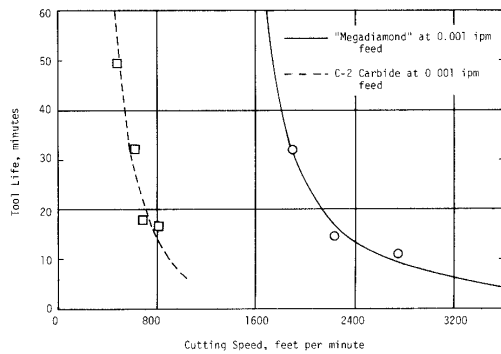
### Wear Resistance

In still another way this material differs from most single-crystal diamond. A large diamond can be lapped so that it has a sharp edge and can then be used as a cutting tool. After a certain amount of use the cutting edge of a single-crystal diamond becomes worn and rounded. Then the cutting forces become high and a poor finish is left on the workpiece so the tool must be refinished. In contrast to this, the cutting edge of a worn and rounded sintered diamond will continue to cut with relatively smaller cutting forces while the workpiece finish is still relatively good. (This property is more apparent with the filled type of sintered diamond.) Apparently, this is because each microscopic particle acts as an individual cutting point. As one particle becomes worn away another is exposed and the edge continues to cut well. Furthermore, the wear rate is not dependent upon the tool orientation as it is with a single-crystal. This is because as was mentioned earlier, sintered diamond is macroscopically homogeneous.

The above result is somewhat dependent on the material being machined as sometimes the sintered -diamond tool wears just as fast as conventioned diamond tools. This was the case when sintered diamond was used to cut the hardest grades of carbon and also some fiberglass-filled phenolics. Here also, sintered diamond that does not contain a filler usually performs more poorly than the filled material.

The comparative wear rates of sintered diamond and tungsten carbide have also been measured in several tests. A sintered diamond cutting tool insert examined under comparable conditions lasted fifty times longer in cutting melamine plastic<sup>5</sup>. When the material cut was on 88/12 Al/Si alloy, the sintered diamond had a negligible wear after cutting an amount of alloy that wore out a tungsten carbide insert. Still other results of this nature are shown in Figure 10, where data for machining 390 aluminum alloy are shown.<sup>6</sup> This very abrasive alloy would wear out a tungsten carbide cutting tool much more rapidly. For example, at a cutting speed of 1,000

\* An example of such a solder is Handy and Harmon 1175-1225.



**FIGURE 10. Cutting speed/tool life comparison for sintered diamond and carbide tool cutting AA 390 aluminum alloy.**

surface feed per minute, the sintered diamond tool life was 140 times as great as that of C-2 carbide.

### Conclusion

From the discussion presented above, it can be seen that sintered diamond is a new and unique material. Of all types of diamond, it is most closely related to carbonado. However, it differs in many ways. One is that a desired shape is achieved more easily because the diamond powders can be sintered into almost any desired shape. Also, it has electrical properties that are dependent on manufacturing parameters and differ from those of other diamonds. Furthermore, its mechanical and chemical properties can be improved by the use of select fillers incorporated into the powder. Thus, this new technology adds a new dimension to the use of an already versatile substance—diamond.

<sup>1</sup> Bovenkerk et al., "Preparation of Diamond", Nature 184, p.1094, 1959.

<sup>2</sup> Horton, R.M. and M.D. Horton, "The High-Pressure Graphitization of Diamond", High Temperatures-High Pressure, 4, p. 39, 1972.

<sup>3</sup> Wheeler, R.L., "Electrical, Mechanical and Thermal Properties of Sintered Diamond Particles" Unpublished Ph.D. Thesis, Brigham Young University, Provo, Utah 1973.

<sup>4</sup> Pope, B. J. et al., "High Thermal Conductivity Microwave Substrates," Final Report to the U.S. Army Electronics Command on Contract DAABO7-73-C0289, in press.

<sup>5</sup> Morris, D., "Diamonds Glitter in High Wear Applications", Tooling and Tool Design, p. 82, Jan. 1974.

<sup>6</sup> Olofson, C.T. and F.W. Boulger, "Tool Life Evaluation of Megadiamond Disks", Battelle report to Diamond Distribution, Inc., Oct. 1973.