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Sintered Diamond: A Synthetic Carbonado

Abstract. *Diamond powder is rapidly sintered into molded shapes at a pressure of about 65 kilobars and a temperature of 2500° Kelvin. Other conditions of pressure and temperature are also suitable. The product compares favorably with natural carbonado in its properties.*

A dozen years ago, I discussed the desirability and possibility of preparing a carbonado-type substance by bonding diamond particles together under conditions of high pressure and high temperature (*1*). I now report procedures for accomplishing this.

Natural carbonado is a rare polycrystalline form of diamond found primarily in Brazil. Because of its toughness it is sometimes used in diamond drilling bits for penetrating hard rock formations. Carbonado would have many additional uses if it were more readily available and if it were less difficult to shape. It has no regular cleavage, as does ordinary diamond, and it is also much more difficult to cut and polish. These problems are overcome in the synthetic carbonado, because it can be formed to shape in a mold. Several sintered diamond articles, including a cylinder with an axial hole strung on a wire are shown in Fig. 1.

The synthetic material is tough. It cuts, indents, scratches, abrades, and wears well. Compressive strengths ranging up to 58 kb and densities up to 3.48 g/cm^3 (the theoretical density of single crystal diamond is 3.51) have been measured.

I have experimentally delineated practical pressure and temperature fields, wherein pure diamond powder can be sintered within times ranging from several days down to about a second. This is illustrated by Fig. 2, where line 1 separates the thermodynamic region of stability for diamond (above line 1) from that of graphite (below line 1). Line 3 indicates a somewhat arbitrary lower temperature limit for obtaining a usable sintered body. Sintering times at this temperature are several days.

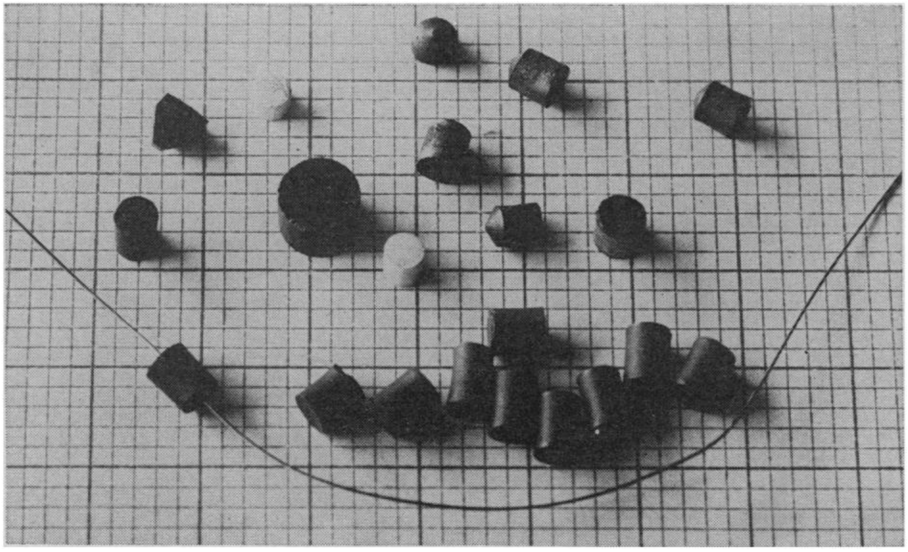

Fig. 1. Sintered diamond shapes (background: 5 squares per centimeter).

Line 2 delineates a practical region of stability where diamond powder is kinetically stable (metastable) with respect to conversion to graphite. This region is located between lines 1 and 2. The thermodynamic equilibrium (line 1) is, of course, time-independent. However, metastability (line 2) depends on time. The line shown is that determined for a temperature holding time of 30 minutes. For other heating times, the shape of the line is similar, but its position is shifted. The diamond powder is first subjected to pressure. Temperature is then applied and held constant for 30 minutes, after which the sample is quenched to room temperature before pressure is released. A longer temperature holding time reduces the area between lines 1 and 2, whereas a shorter time increases the area. Line 2 is also affected somewhat by diamond particle size, impurities, surface characteristics, and the surrounding atmosphere. A distinctive feature of line 2 is the large change in slope occurring near a pressure of 35 kb and a temperature of 1400°K.

Diamond powder can be sintered into a coherent body without any transformation into nondiamond forms of carbon in the region between lines 2 and 3. It is important to note that although this region includes and overlaps the region of diamond thermodynamic stability, it is not necessary to work within the region of thermodynamic stability to produce sintered diamond.

At a given pressure, sintering is most rapid at a temperature near line 2. Sintering time decreases rapidly with increasing temperature; consequently, the shortest sintering time is achieved at the highest pressure. It is possible to obtain a well-sintered product in only a few seconds at 85 kb.

For the particular heating time for which line 2 has been determined and a given pressure, line 2 determines the highest temperature than can be used to produce sintered diamond without any transformation to nondiamond carbon. Sintered diamond made under these circumstances is white or translucent in color.

Contrary to my former expectations, a sintered diamond product can be made at temperatures higher than those designated by line 2. At these temperatures some of the diamond will transform to nondiamond carbon. This product is black in color.

Therefore, careful regulation of temperature about those designated by

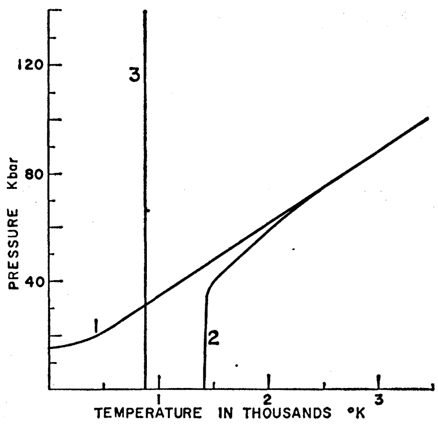

Fig. 2. Diamond sintering regions. Line 1 is the thermodynamic equilibrium boundary for diamond and graphite. Line 3 is a somewhat arbitrary minimum practical sintering temperature for diamond particles. Line 2 is the metastability line for conversion of diamond powder to nondiamond carbon. The location of line 2 depends on time, but the form of the curve for other time periods is similar to the 30-minute curve which is shown.

line 2 can yield a product ranging in color from white through gray to black, and synthetic carbonado of good quality can be produced throughout this color range. Debye-Scherrer x-ray diffraction patterns of the black product show lines characteristic of nondiamond carbons in addition to the diamond lines. The black product is a fair electrical conductor, whereas the white product is insulating. There has been some surface transformation of the diamond particles into nondiamond carbon in the black product. But when the amount of transformation is carefully controlled, a well-bonded black product with excellent physical properties can be produced. The nondiamond carbon is quite effective in bonding the mass of particles together. It always requires a longer sintering time and lower temperature to make an acceptable white product. Increasing the amount of diamond decomposition can produce a soft product that will wear away more rapidly in abrasive use. The softness and accompanying rate of wear can be controlled by manipulation of the pressure and temperature time variables.

The sintered products made from diamond powder thus far discussed are self-bonded. That is to say, the agencies responsible for bonding the mass of particles together come from the diamond itself. It is also possible to produce a synthetic carbonado from diamond powder, wherein a bonding agent or binder is added to the diamond powder. I have found that powders of hard refractory substances, including borides, carbides, nitrides, and oxides, will serve as suitable binders.

The time considerations for pressure and temperature discussed for self-bonded diamond also apply when binders are used.

Two examples for the preparation of synthetic carbonado are given below.

1) A cubic (hexahedral) press was used to generate pressure. The square faces of its tungsten carbide anvils were 0.953 cm on edge. The cubic pyrophyllite sample cell was 1.19 cm on edge and contained a graphite sample tube with sample space 0.254 cm in diameter by 0.475 cm long. This space was filled with natural diamond powder (1 to 5 μm particle size) and was heated by an electric current passed through the graphite tube. Pressure was increased to 85 kb (room temperature calibration; no correction for elevated temperature) then temperature was increased to about 2440°K and held for 3 minutes. Heating current was then switched off, whereupon the sample cooled to near room temperature in about 10 seconds. Pressure was released, and a near-white synthetic carbonado was removed. It was cylindrical in form, being 0.218 cm in diameter by 0.376 cm long; it weighed 0.25 carat (5 carats = 1 g). Its pycnometric density was 3.48 g/cm^3.

2) A cubic press with anvils 1.27 cm on edge was used. The heater and sample container loaded with 1- to 5-μm diamond particles was a molybdenum tube with interior sample space 0.775 cm in diameter by 0.525 cm long. This was centered within a pyrophyllite cube 1.58 cm on edge. Pressure was 65 kb (room temperature calibration) and temperature was held at about 2500°K for 21 seconds. The product was a dark steel-gray cylinder 0.498 cm long by 0.643 cm in diameter, weighing 2.5 carats and having a pycnometric density of 3.09 g/cm^3.

If, in a run like example 2 above, some of the 1- to 5-μm diamond powder is replaced with 5- to 40-μm particles, the product density can be increased significantly. Synthetic diamond powders produced by explosive means as well as by static pressure methods can also serve as starting material for the production of synthetic carbonado.

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Hemolysis Near an Ultrasonically Pulsating Gas Bubble

Abstract. *A small volume of an erythrocyte suspension was subjected to the action of a manipulated gas bubble set into stable oscillation at 20 kilohertz. Release of hemoglobin occurred when the oscillation amplitude exceeded a critical threshold. Hydrodynamic stresses resulting from acoustically induced small-scale eddying motion near the bubble may be the mechanism of hemolysis.*

The sonic interactions with biological suspensions characteristically occur by means of cavitation, a complicated phenomenon involving sonically activated bubbles. To understand the mechanism, the situation may be simplified by maintaining a single stable oscillating gas bubble in a suspension of cells within a Plexiglas vessel attached to a vibrating bar. Observations in this system led to two primary conclusions: (i) stresses associated with acoustic streaming constitute an important mode of action for sonic effects on cell suspensions, and (ii) measurement of the critical threshold for release of cell contents provides a method for determining the mechanical strength of cell membranes.

The gas bubble is formed in a stainless steel tube (260 μm inside diameter by 2.0 cm) which is connected to a gas reservoir through a 30-cm length of stainless steel tubing (50 μm inside diameter). The latter small tubing prevents large volume changes of the bubble by providing resistance to rapid changes in pressure. By observing with a microscope the operator adjusts the pressure in the gas reservoir so that the gas-liquid interface is hemispherical. Upon application of the sound field the bubble starts to oscillate and also flattens as the enclosed air tends to retreat up the tubing. The reservoir pressure is then increased until the bubble is again hemispherical. In this system the increase in reservoir pressure can be