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SYNTHETIC DIAMONDS FOR THE INDUSTRY: THEIR OUTSTANDING PROPERTIES AND FUTURE DEVELOPMENT

by

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ABSTRACT

In this contribution, an overview is given of the industrial uses of diamond. The major part of these diamonds are synthetic. The Ultra High Pressure synthesis of diamonds is explained first and related to their mechanical and thermal properties. In a second part, the advantages, manufacturing and characterisation tests of polycrystalline diamonds (PCD) are presented.

In a third part, the CVD diamond synthesis technology is described and a prognosis of future applications in high-tech industries such as optronics and micro-electronics is presented. He concludes that y diamond is certainly the material of the future. The 21st century might well become the "Diamond" century !

RESUME

Dans cette contribution, on donne un aperçu sur l'usage industriel du diamant. La majorité de ces diamants sont synthétiques. L'auteur explique d'abord la synthèse du diamant sous ultra haute pression et la met ensuite en rapport avec ses propriétés mécaniques et thermiques. Ensuite, la production et la caractérisation des diamants polycristallins sont présentées en relation avec leur application industrielle.

Enfin, l'auteur décrit la technologie de synthèse CVD du diamant, et donne prognose pour de futures applications dans les industries de haute technologie comme l'optronique et la micro-électronique. Il conclut que le diamant est certainement le matériel du futur. Le 21ième siècle pourrait bien devenir le siècle du "Diamant" !

KEY WORDS

Diamonds, synthetic, properties, manufacturing, thermal damages, polycrystal, test, CVD.

MOTS CLE

Diamant, synthétique, propriétés, fabrication, dégâts thermiques, polycristal, test, CVD.

1. THE SYNTHETIC DIAMOND

1.1. PRODUCTION

The world consumption of industrial diamond has grown at the rate of more than 9 per cent per year during the last 20 years.

Starting from zero in 1957, the share of the synthetic diamond has now reached 89 per

¹ Diamant Boart, Avenue du Pont de Luttre 74 - B-1190 Bruxelles.

² An abridged version of this article has been published in Antwerp Gems, vol. 1, nr 2 (1990), pp. 8-15.

³ paper based on communication presented at Diamond Symposium, Antwerp, 24.11.1989.

cent. It means that the growth of the consumption of the synthetic diamond has been much higher, 13 per cent per year during the last 20 years (figure 1).

CONSUMPTION OF SUPERABRASIVES

WORLDWIDE

Figure 1: World consumption of industrial diamond. Whereas consumption of synthetic diamond grows almost exponentially, consumption of natural diamond remains stable.

Ten years ago, the consumption of the natural diamond was still limited by the mined production. A shortage has even been experienced in several sizes of crushed industrials. Nowadays, the availability exceeds the needs. But the industrial world does not like a natural product anymore. For technical reasons connected to the reproducibility and the sophistication of a high-tech product, the synthetic type is now preferred, although more expensive in many cases.

1.2. HOW CAN DIAMOND BE MADE ?

The first analysis of diamond has been made around 1770 by the French scientist Lavoisier, who proved that diamond is pure carbon. In natural diamonds, some impurities like nitrogen, aluminium, silicon can be found at a relatively low level.

Diamond is not the only crystalline phase of carbon : graphite is most common. The atom model of graphite shows a structure made of planes, opposed to the isotropic structure of diamond.

Due to its laminated structure, with comparatively weak atomic forces holding the planes (Van der Waals bonds), graphite is often used as a lubricant. Quite the contrary, the diamond is made of carbon atoms squeezed together and tightly hold on each four neighbour atoms (covalent bonds). It is hard enough to be used as an abrasive, in fact it is the toughest one. The dense arrangement of diamond is the highest number of atoms per volume of all known materials.

Diamond is stable at high pressures, above 30.000 atmosphere, and metastable in room conditions. At first sight, it seems paradoxal that the hardest and most durable materials known to man is metastable ! This point will be discussed later when the thermal damage will be examined.

The usual synthesis of diamond, converting graphite to diamond, requires ultrahigh pressures, more than 50.000 atmosphere (5 GPa), and very high temperatures, more than 1200°C, in order to reach the region where diamond is thermodynamically stable (figure 2) (Bovenkerk *et al.*, 1959; Bundy *et al.*, 1961; Bundy, 1963, 1964).

This process needs a sophisticated tooling in order to withstand this tremendous pressure, which is so high that it exceeds the compressive strength of all know materials. The breakage of the expensive tooling used is the major part of the process cost.

The first ultra-high pressure synthetis of diamond has been made by the team of ASEA in Sweden in 1953 but has been kept secret until the success of General Electric in USA, end of 1954. Three years later, the first synthetic diamond type for grinding applications has been introduced by GE on the market. Since the pioneers' time in the fifties, many laboratories like the De Beers Diamond Research Laboratory have succeeded in repeating this exciting experiment, an old dream of the alchemists. Today synthetic diamonds are produced by at least thirty factories.

The transformation takes place in a reaction chamber containing the graphite as the carbon source and a solvent-catalyst. The mix is placed in a ceramic capsule in charge of insulating the tooling from the dangerous heat and of transmitting the needed pressure.

The heating by the Joule effect (generally AC, sometimes DC) of the reaction chamber can be made in three ways :



Figure 2 : Carbon Phase Diagram Showing the Diamond Graphite boundary (after Bundy, 1963).



Figure 3a : Diagram showing the loss of weight of natural and synthetic diamond when heated in pure oxygen and in stagnant air. Natural diamond loses more weight than synthetic diamond.

- direct heating where the electrical resistance is the mixture solvent and carbon itself. This is not stable because the liquid is infiltrating graphite and during the



Figure 3b : Diagram showing the thermal stability of natural (in pure oxygen, and without oxygen) and synthetic diamonds. Natural diamond shows to be more stable than synthetic diamond.

conversion of graphite to diamond, the resistivity is becoming higher.

- indirect heating where a tubular furnace is put around the reaction volume. This

electrical resistance can only be modified by geometrical changes.

- semi-direct heating (Diamant Boart patent, ref. Belgium 706.964) where the furnace is flat along the piston noses. In this arrangement, no electrical insulator is needed between the reaction and the resistances.

At high temperature, the carbon is dissolved in a solvent which is a liquid alloy, generally a mixture of nickel, iron, cobalt and sometimes chromium, manganese,

When saturation occurs, the precipitation of some excess carbon can only be made in the dense phase (diamond), due to the ultrahigh pressure. The diamond growth is carried on until the required crystal size is reached. Then the temperature drops, due to the power cut (Plate 1, photographs 1-4).

After cooling and depressing, the reaction cell is removed and the crystals are chemically cleaned, by acid leaching of metal and graphite, diamond being resistant to most chemicals (Plate 1, photograph 5). The sorting can be made automatically by size, shape, purity and toughness.

The tooling is basically made of cemented tungsten carbide and consists of a ring (die) with an anvil (piston) on each side. The die set with both anvil arrangements are put inside a big hydraulic press (Plate 1, photograph 6). Many different diamond types can be achieved from that process :

- Top quality, like gem-like, can be obtained when everything goes well. These stones can even be cut into small brilliants but their cost is far above that of natural diamonds which makes them unattractive for jewelry applications at the current prices (Pl. 2, photograph 1).

The top quality for industry is used for granite sawing.

- A near top quality is preferred for mining bits, for marble saws and for construction saws and drills. These crystals have generally a cubo-octahedral habit. It means that you can recognize the square faces of the cube morphology (crystallographic (100) planes) and hexagonal or triangular faces of the octahedral morphology (crist. (111) planes) (Pl. 2, photograph 2). - Less regular crystals are used in less severe conditions for soft marble sawing, dry cutting, wire sawing, glass and mechanics (Pl. 2, photograph 3).

These are only a few among many types, each of them is tailor-made for an industrial application, therefore they are better than the mined diamond for the same usage.

A similar superabrasive that does not exist in nature can be manufactured : cubic boron nitride. Made of boron and nitrogen, the hard phase is obtained in the same way as for synthetic diamond, but starting from the hexagonal boron nitride, which has a graphite structure (Pl. 2, photograph 4).

Cubic Boron Nitride (CBN) is chemically different from diamond and has not the same reactions with iron, thus not producing iron carbide (cementite) when the abrasive wheel is grinding steel, which reduces the agressivity of such wheel. Therefore CBN wheels are superior to diamond wheels for steel grinding.

2. THE OUTSTANDING PROPERTIES OF DIAMOND

Diamond is the hardest material known.

It has the highest compressive strength, the highest heat conductivity and it is an excellent electrical insulator.

Its optical transmissivity is exceptional and its corrosion resistance is very high.

Diamond	8800 kg/mm2
Cubic Boron Nitride	4500 kg/mm2
Boron Carbide	3700 kg/mm2
Silicon Carbide	3000 kg/mm2
Titanium Carbide	2900 kg/mm2
Tungsten Carbide	2300 kg/mm2
Alumina	2200 kg/mm2
(Alumina, Corundum	,
Ruby)	
Quartz	820 kg/mm2
Glass	530 kg/mm2
Steel	200 to 1000 kg/mm?

Table 1 : Knoop hardness in kg/mm2

Diamond is unique by being the champion in many categories but what makes it the most

interesting material is the combination of those advantages. For instance, to be a very good electrical insulator and at the same time a better thermal conductor than metals is exceptional and of outstanding importance for the industry (Berman, 1965; Field, 1979; Wilks & Wilks, 1991) (Tab. 1-2).

Diamond	600 to 2100
Copper	400
Aluminium	200
Tungsten Carbide	100
Graphite	65
Nickel	60
Silicon Carbide	10 to 100
Alumina	9 to 25
Granite	2 to 5

Table 2 : thermal conductivity in $W/(m^{*\circ}K)$

All diamonds are not the same. Although falling in the same range, the mechanical properties can be different enough to make the diamond tools more or less effective. The know-how of the toolmaker is to establish the correlation between the tool performance and the diamond characteristics. When this is achieved, laboratory measurements can be used the predict the final performance or at least to know the potentialities.

The usual characteristics measured on diamond grits are : distribution of sizes, shape factor, toughness index, microchipping resistance, wear or abrasion resistance, same mechanical characteristics after heat treatment, ... The examination by a scanning electron microscope with of resolution of 5 nm (0.005 micron) is useful to understand how some defects are responsible for the mechanical characteristics.

Natural and synthetic diamonds enjoy very high mechanical and corrosion resistances until 600°C, but they can be thermally damaged at higher temperatures. It has been said above the diamond is metastable. This statement is not easy to visualize but can be illustrated in the following way : "If you are above the second floor of a building, you may feel a little bit less 'stable' than when you are on the pavement outside. Especially in case of fire, any jump through the window could give you a good idea of what is a metastable state, due to heat, compared to a stable and solid state in the street...." Similarly thermodynamic instability affects diamond at high temperature (Tab. 3).

THERMAL DAMAGES IN DIAMOND
1. Thermochromic effects :
amber colour, reversiblepermanent black colour centers
2. Differential thermal expansion :
inside (inclusions)outside (Co from PCD, Bond)
3. Reactions with the environment :
with the gaz (oxygen)with the metals or alloys
4. Phase conversion, recrystallisation :
conversion to graphiteor to an intermediate structure

Table 3 : thermal damages in diamond.

When subjecting diamond, natural and synthetic, to progressively higher temperatures, natural diamond is disappearing first. Most of impurities of natural diamonds do not prevent the reaction $C + O_2 \rightarrow CO_2$. Synthetics are more resistant because the impurities are mainly the metals used as solvent or catalyst. These atoms are not volatile and act as inhibitors to the CO_2 -reaction. When oxygen is rare, synthetics and natural behave more or less the same with respect to the loss of weight at high temperature (Fig. 3a). When the damages are measured by mechanical ways, synthetic diamonds appear to be more sensitive than naturals (Fig. 3b).

3. POLYCRYSTALLINE DIAMONDS (PCD)

3.1. Polycrystalline natural aggregates or carbonados and their drawbacks

A polycrystalline natural aggregate of diamonds was already known before the first achievement of synthetic diamonds. This material, also known as Carbonado, can be found in Brazil or South Africa. It has been used in drilling and sawing and has been economically successful in several cases. The advantage of a polycrystalline product, already recognized during the first two decades of the 20th century, is its isotropic strength.

This particular point could be of interest to understand the present success of polycrystals. The weakness of monocrystals with respect to shocks is the cleavage. This property to split when stresses are applied in a particular direction is well known by the diamantairs who have even made the first cuts of the future "brilliant" this way. But for the industry this monocrystal characteristic is a dangerous weakness. On the contrary, a cleavage cannot happen in polycrystals because crystal grains are randomly oriented and the splitting of each grain is stopped at the grain boundary.

But polycrystals have a weakness too, which depends on the joints between grains. In a natural product like Carbonado, mother nature has not taken particular attention to that point. In many cases, carbonados are thus too friable as the grains are splitting from each other.

Therefore the industry prefers synthetic products exactly meeting their requirements. What was already true for monocrystals is of course valid for polycrystalline products. The synthetic PCD is tailor-made for drilling, cutting, chiseling, sawing or whatsoever. Particularly in drilling explications, PCD has largely contributed to fast and economical drilling (Delwiche, this volume). The drawback of this extreme suitability is the wide range of different products. Each type has its specific function and any attempt for standardisation is kept in check.

Particularly, the range of available geometries is almost unlimited (Pl. 2, photograph 5). This causes an additional challenge to the industry developing PCD applications. In order to explain how to select the right PCD for each application, we have to explain how they are manufactured. The apparatus needed is the same as for diamond synthesis, that is basically a hydraulic press with a tooling capable of ultra-high pressure and high temperature. To reach the necessary exceptional strength, the PCD body has to be sintered in the conditions of diamond synthesis. Only in the environment of diamond crystal growth real diamond bridges between monocrystalline grains can be expected.

3.2. Manufacturing PCD

The start material consists of very fine and randomly oriented diamond grains. The grade of the particles can be 1, 10 or 100 microns, in fact any size or any combination of sizes. The size of the grains is the first characteristic of the future PCD blank. As only diamond growth by a solvent-catalytic process appears to be economical, a liquid metal allov is needed in the reaction cell. Cobalt is often used as the solvent. The final concentration in metal is the second characteristic of the PCD. Other ingredients may be added and new composite products are now investigated. During the high pressure-high temperature process, that is during sintering, any specific condition acts on the structure of the final product.

The main difference between Diapax and Tripax ⁴ types is that Diapax are backed by a substrate in tungsten carbide. In that case, the characteristics of the carbide have to be added to those of the PCD in order to determine its properties.

However, after the sintering process, other treatments can take place and may change considerably the PCD specifications. The known treatment is the leaching of the PCD without substrate (Tripax type) in acid in order to remove any trace of metal. The problems due to thermal expansion of the inside metal are so avoided. But a high level of porosity is induced allowing the oxygen to make some damage inside the PCD. As usual, any improvement has its drawback.

Research is now concentrated on new types which are no more pure diamond. Polycrystalline cubic boron nitride (CBN) is already well known by its mechanical applications in machining steel and alloys. Composite materials of diamond or cubic boron nitride and ceramics are now under investigation. We may expect a prolific range of new composites in the future.

⁴ Diapax and tripax are registered trade names for the PCD (polycrystalline diamonds) used by DB Stratabit.

3.3. PCD CHARACTERIZATION

3.3.1. Basic philosophy

"To test the PCD blank itself and alone". That means to involve as few parameters as possible. The shape itself has to play a minimum role in the test. We are interested in mechanical strength only. All results will be figures estimating how far the PCD resist to different types of stresses in different conditions. The higher the value, higher the strength of the blank.

Obviously, other parameters are important also for the final performance in the field. Thus, the laboratory test cannot predict the field result. That is normal and that is fine. Like for every tool, a good workman and a good usage are necessary to achieve the success. In our philosophy, the basic properties of the tool have to be measured independently. So, the conclusions can be more easily generalized.

Nevertheless, in order to estimate the suitability of the tests to the field, the stresses used in the test must be in close relation with those induced by the application. The test procedure is different for Diapax and Tripax because both types are supposed to be used in different ways.



Figure 4: The diapax tests show the relationship between the feedstock particle size and the mechanical resistance. The latest improvements in the manufacturing process have reduced these differences.

3.3.2. Diapax Tests

3.3.2.1. Standard test of characterisation

Abrasion resistance : this procedure is now very classical. The blank is supposed to machine a rod of hard stone like granite for example, on a lathe. The wear on the chisel can be measured easily.

Shock resistance : many impacts can be generated by a mass falling on the edge of the sample. The relative speed and the number of impacts can be choosen in relation with the final purpose.

Fatigue resistance : if the impact energy is low enough, a big number of shocks will be necessary to cause some damage. Although the basic phenomena observed in this way are not the same as fatigue in metal. Some very interesting information is obtained when analysing this type of "brittle fatigue".

Wear resistance : when taking care to avoid any shock in a smooth trajectory of the sample on a surface, pure wear is induced and can be measured, by weight loss for example.

Resistance with respect to wear with shocks : if the surface used in the preceding experiment is ruguous or if there are obstacles on the path followed by the sample, shocks will be induced and will change the wear process.

Crushing resistance : after some time in a ball mill, some fragments will be lost for the diamond layer and also from the carbide substrate. Measurements of lost material give us information on diamond and carbide crushing strength.

Thermic stability : after heat treatment, the same mechanical tests as described before, can be repeated. Results are compared and the thermic degradation can be computerised. Different temperature and time can be tested, according to the purpose.

These seven points are the standard tests of characterization used at Diamant Boart for each Diapax type. But other tests are often performed to get further information, like grain sizes, metal content, transverse rupture strength, hardness...

Non-destructive techniques are of course used as often as possible, like ultrasonic methods, for testing the risk of delamination for instance of scanning electron microscope, for examination of homogeneity and presence of cracks (Pl. 2, photograph 6).

3.3.2.2. Comments on test procedures

The most informative tests are destructive, the test cost could thus be a problem.

We must take care of another problem : the sampling. We must be sure that the supplier does not present his best pieces, sorted by ultrasonic or SEM methods for laboratory evaluation. The best, which is only possible for big companies, is to take random samples from big batches.

An obvious characterization is the geometry. We can only compare bodies of same size. We have to pay a particular attention to the chamfers that can change the results a lot, praticularly when testing the shock resistance of the edge.

The first relation that will appear is the one between grain size and basic properties (Figure 4) :

small grains sizes give a higher abrasion resistance ;

larger sizes give more shock resistance.

The big skill is anyway a question of compromise. In recent products, we have observed that improvement has been achieved for a typical group of characteristics which was not matched for other properties.

3.3.3. Tripax tests

Two basic differences between Diapax and Tripax have resulted in redesigning the tests for PCD without substrate :

- Tripax bodies are usually smaller, thus preventing us to clamp the sample for shocks on the edge, for example.

- All results can be measured by weight loss because there is no heavy tungsten carbide.

The prefered test procedures are related to crushing machines in one way or another. The tests used can be summed up in two groups : wear and crushing resistances.

The different generations of Tripax are hardly comparable.

- The first generation still contains the metal or metal alloy used for the crystal growth process. Tripax of that kind are no more used (that product can only be found in Diapax).

- The second generation is obtained by leaching the material of the first generation. The result of this process is a porous material, consisting of pure diamond or pure CBN.

- In the third generation, the porosity has been filled by an exotic material, most often a ceramic. This composite is not obtained from the first or second generation, but directly synthetised as such. The interesting thing in the last generation is the intergrowth not only between the diamond grains, but also between the foreign material grains. One can imagine how productive the research on his type of combination can be. The two interlocked structures give a very interesting thoughness and other attractive properties, including the thermal stability of non-porous diamond in presence of oxygen.

4. THE FUTURE : CVD DIAMONDS

The ultrahigh pressure process is no more the only way to make diamonds. A new feasability is born by the technique of Chemical Vapour Diamond (CVD). Today,



Figure 5 : SEM image of isolated diamond twin crystals produced by CVD synthesis at the Diamant Boart Laboratory in Brussels (bar length $10 \mu m$).

research is very active in this field (Tab. 4) (Fukunaga, 1988; Kamo, 1988; Sato, 1988).

As early as the seventies, research into the feasability of diamond coatings was initiated in the Soviet Union. Using essentially the chemical vapour deposition process, the scientists investigated methods to deposit a continuous film of diamond on a substrate, but obtained low growth rates and layers of a few microns only.

The pioneering Russian work was followed up in the early 80's by the Japanese who concentrated on developing feasible and industrial processes to coat with diamond : cutting tools, lenses, optical windows and loudspeaker diaphragms.

Crystallume, in California, was formed in 1984 to produce diamond films, with the idea of taking advantage of the material's hardness and wear resistance, with an annual budget of 3 million US\$. In 1986, the United States began a massive research programme on CVD diamond growth. Pennsylvania State University has established a Diamond and Related Materials Consortium which has attracted 31 industrial sponsors and receives substantial government funding administered by the Office of Naval Research.



Figure 6 : SEM image of polycrystalline diamond coating produced by the CVD-synthesis (bar length 10μ m).

1953	USA : first success (traces)	
1970	USSR : diam. by epitaxy on diam.	
1976	USSR : diam. on various substrates	
1982	Japan : continuous films	
	(the very start !)	
1984	Sweden : first sign in Europe by	
slow development		
Intensive research in Japan		
1985 - The USA start research		
1987 - Intensive research in USA		
1988 - Commercialization in Japan !		

Table 4. : CVD History.

The results are still very limited, due to the slow rate of crystal growth, but encouraging (Tab. 5). In most techniques, the CVD process is enhanced by a plasma. This is not an easy process owing to much instability in the plasma, because the result is the consequence of three steps :

electron-impact dissociation of the precursor gases to form the plasma, by supply of energy (microwaves, laser, arc discharge, direct heating, ...) (Tab. 6).
radical and ion transport to the substrate (provided the lifetime of these radicals or ions is long enough).

3. film and crystal growth with the occurrence of many chemical reactions.

Source gas	Growth rate		
Hydrocarbons Alcohols CO	0.3 m/hr 0.8 m/hr 1.5 m/hr (small particles)		

Table 5 : Chemical Vapour Description by microwave plasma, diamond growth rate according to source gas.

The mechanical applications could be :

a) reinforcement of anti-wear materials and cutting inserts, like tungsten carbide and refractory metals. It has been shown that a diamond coating on such inserts greatly improves the turning of non-ferrous metals, mainly aluminium.

b) protective layers on existing diamond tools against lateral erosion, which is destructive for the core itself of diamond disks and saws.

c) high thermal conductivity diamond layers can eliminate the need for cooling the tools,

now required to prevent damaging graphitization.

d) new concepts of tools where deposition of diamond grit is rather difficult or impossible: concave grinding wheel, ultra-thin blade saws, etc..

The industrial applications of CVD diamonds are not only in mechanics. The new opportunity to make diamond films could emerge on protective coatings against wear and corrosion. Particularly in optics, some diamond properties can now be used like the excellent transparancy even to UV-waves. A diamond coating could act as an antireflection film on a window, due to its high refractive index. Above all, the most promising applications will be in electronics. The characteristics required for a good semiconductor can be found in the diamond (Tab. 7).

CVD	Conditions	Applied fields
Hot filament	1.33 " 13.3 kPa 600 " 1000°C	
DC arc plasma	13.3 "101 kPa 700 "1200°C	
RF plasma (13.56 MHZ)	0.5 " 3.0 kPa 800 " 950°C	
(glow discharge)		
RF plasma (4 MHZ)	0.8 " 101 kPa 700 " 1200°C	Powder
(thermal)		
Microwave plasma (2.45GHZ)	1.33 " 13.3 kPa 700 " 1000°C	Semi conductor
		Fine cutting tools
ECR (2.45 GHZ)	< 6.6 kPa 650 " 950°C	Semi conductor

Table 6 : Chemical Vapour - Deposition techniques.

	D(*)	GaAs	Si
Resistivity(Ohm-cm)	1016	108	103
Heat conductivity (W/cm2DEG)	20	0.46	1.45
Electron velocity (in 100 km/sec)	2.7	1	1
Band gap (eV) (stability)	5.45	1.43	1.1

Table 7: Properties for electronic applications (from Peters *et al.*, 1988). (* Diamond).

It is already known that the diamond semiconductors will be of much higher quality than those used today. Moreover, the diamond-based chips will work above 100°C where the conventional semiconductors are already destroyed. The CVD diamond is the ideal material for the future high-power, high-frequency semiconducting devices.

REFERENCES

- BERMAN, R. (Ed.), 1965 Physical Properties of Diamond. Clarendon Press, Oxford.
- BOVENKERK, H.P., BUNDY, F.P., HALL, H.T., STRONG, H.M. & WENTORF,

R.H. Jr, 1959 - Preparation of diamond. *Nature*, 184 (Oct. 10, 1959): 1094-1098.

- BRADLEY, R.S., 1966 Advances in High Pressure Research. Volume 1. Academic Press, London, 396 p.
- BRADLEY, R.S., 1969 Advances in High Pressure Research. Volume 2. Academic Press, London, 280 p.
- BRADLEY, R.S., 1969 Advances in High Pressure Research. Volume 3. Academic Press, London, 391 p.
- BUNDY, F.P., BOVENKERK, H.P., STRONG, H.M. & WENTORF, R.H. Jr, 1961 - Diamond - Graphite equilibrium line from growth and graphitization of diamond. *Journ. Chemical Physics*, **35/2**: 383-391.
- BUNDY, F.P., 1963 Formation of new materials and structures by high pressure treatment. ASTM Spec. Techn. Public., 374: 52-67.
- BUNDY, F.P., 1964 Diamond synthesis and the behaviour of carbon at very high pressures and temperatures. *Ann. New York Acad. Sc.*, **105**: 951-982.

- DELWICHE, R., 1993 Diamond applications in the oil industry. *Bull. Soc. belge Géol.*, this volume.
- FIELD, J.E., 1979 The Properties of Diamond. Academic Press, London, 674 p.
- FUKUNAGA, O., 1988 Large volume high pressure apparatus for diamond synthesis. *New Diamond*, n°1, Ohmsha, Tokyo: 12-16.
- KAMO, M., 1988 Study on diamond synthesis from gas phase at National Institute for Research in Inorganic Materials. *New Diamond*, n° 1, Ohmsha, Tokyo: 24-29.
- MATTHEWS, A. & BACHMANN, P.K. (Eds), 1991 - Diamond and diamondlike carbon coatings : proceedings of the First European Conference on Diamond and Diamond-like Carbon Coatings, Crans-Montana, Switzerland, September 17-19 1990. Elsevier Sequoia, Lausanne, New York, 806 p.
- PECKNER, D., 1961 Ultra high pressures create new materials. *Materials in de*sign engineering, Sept. 1961: 111-117.

- SATO, Y., 1988 Characterization of Vapor-Deposited Diamond. New Diamond, n° 1, Ohmsha, Tokyo: 30-35.
- SVITS, C.G., 1964 Manmade diamonds -A progress report. *American Scientist*, 52: 395-407.
- VISART de BOCARME, P., 1988 Le diamant synthétique : complément ou concurrent du diamant industriel naturel. *ICHEC*, 126 p.
- WILKS, J. & WILKS, E. (Eds), 1991 -Properties and Applications of Diamond. Butterworth Heinemann, London, 544 p.

Further and future literature can be obtained from De Beers Industrial Diamond Division, Charters Sunninghill, Ascot Berkshire SL5 9PX, England or from GE Superabrasives, P.O. Box 568, Worthington, Ohio 43085, U.S.A.

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PLATE 1

Figures 1 to 4 : Different stages of diamond synthesis in a Ultra High Pressure Unit. The U.H.P. unit depicted is the patented experimental U.H.P. unit with semi-direct heating (Diamant Boart patent 706.964).

Figure 1: The Reaction Cell consists of graphite layers in a metallic container, usually an alloy of nickel, iron, cobalt and sometimes chromium and manganese. This Reaction Cell sits in a ceramic capsule which insulates the tooling from the heat and transmits the pressure from the pistons.

Figure 2 : After pressurizing the electrical resistances ensure a temperature of more than 1.200° C. In these conditions the metal alloy liquifies and dissolves part of the graphite.

Figure 3 : When this liquid becomes supersaturated, carbon will precipitate in its stable form : diamond.

Figure 4 : The transformation process continues until enough graphite has been transformed into diamond. After cooling and depressurizing the produced diamond crystals are recovered from the metallic alloy by acid leaching, cleaned and sorted.

Figure 5 : Acid leaching of metal and graphite.

Figure 6 : Diamant Boart's experimental Ultra High Pressure Unit capable of diamond synthesis in operation during the sixties.













PLATE 2

Figure 1: Synthetic gem-like diamonds, cut in different shapes, more expensive than natural gem diamonds.

Figure 2: Synthetic diamond, not cut, of cubo-octahedral morphology, suitable for natural stones sawing.

Figure 3: Synthetic diamonds, less regular but suitable for dry cutting in construction applications.

Figure 4 : Cubic boron nitride (CBN) manufactured in the same way as synthetic diamond.

Figure 5 : Polycrystalline diamonds (PCD) in various types, shapes and sizes.

Figure 6: Ultrasonic test to check for flaws in various PCD.











