

The 4C's from scientific point of view

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Scientific prolog

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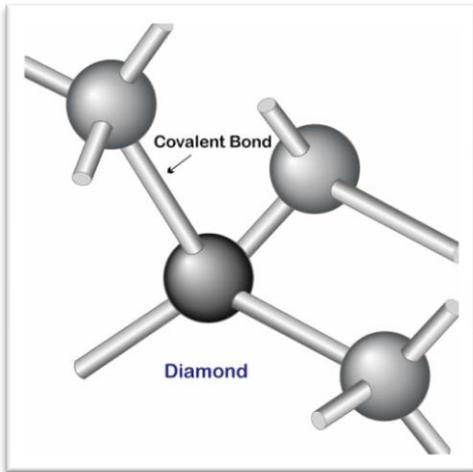
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Leveling Diamond color by HPHT

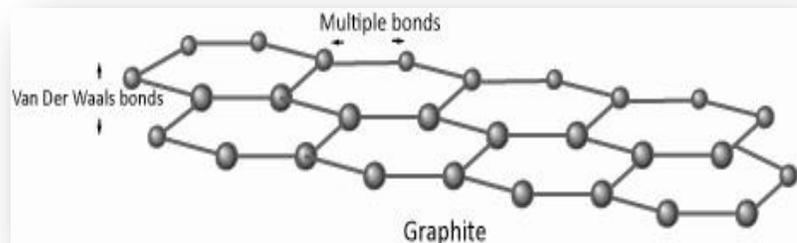
Scientific Prolog

The Carbon Atom [C] is the sixth element in the periodic table, Atomic No [6] which means it has 6 protons and 6 neutrons in its nucleus balanced by 6 electrons in its shell. The orbital configuration of the Carbon atom is $1s^2 2s^2 2p^2$ and he can attach 4 more Carbon atoms by a chemical Covalent bond in which every pair of atoms share 2 Electrons C : C. Carbon has Valence of 4 , since 4 electrons are needed to fill the 2p orbital. Carbon appears in nature in many molecular forms. An all world of organic molecules, that life on earth based on. As an allotropic forms Diamond, Graphite and in a contaminate form like Coal... The 2 allotropic molecular structures: diamond, where the carbon atoms are bonded together in a tetrahedral lattice arrangement, and Graphite, where the lattice carbon atoms appears in sheets of a hexagonal net. Each atom is surrounded by three other atoms so three valence electrons are tied up in covalent bonds. However, each carbon atoms has a free moving electron that can travel along the hexagonal layers. This is why graphite is a conductor. The 2 shapes differ only by their molecular structure in space. The diamond consists of pure Covalent bond. The Graphite plans consist of multiple bonds and the lattices are bonded in a Van der Waals bond. No single set of bond structure can be drawn that satisfies the octet rule (consistent with the behavior of the periodic table second row of elements an atom other then Hydrogen tends to form bonds until it's surrounded by 8 electrons). It's very important to understand that at normal temperatures and pressures, Graphite is only a few eV (enthalpy changed for the reaction = 432 cal/mole!) more stable than diamond and hence diamond is considered metastable and it can be stimulated to become unstable by HTHP treatment.

The 3D tetrahedral structure of the diamond consist of 8 carbon molecules arranged in an equidistant from each other , perform a cube shape like a network. The strong Covalent bond (separation energy = 170Kcal/mole) imparts the Diamond his hardness (10 in moss scale) and its high melting point ($>4000^\circ\text{C}$). In moss scale the Diamond serves as reference point.



Van Der Waals bond consist of small atom dipoles that attracts each other like small magnets. This bond is a lot weaker then Covalent bond.



Light refraction

Refractive index general equation (isotropic material)

$$n = \varepsilon\mu = \frac{1}{v^2} = \frac{c}{v}$$

$\varepsilon = \text{permittivity}$ $\mu = \text{magnetic permeability}$ $v = \text{Phase velocity}$

$c = \text{velocity of light in vacuume} = \sim 3 \times 10^8 \text{ m/s}$

Permittivity = how an external Electric Field affects and is affected by a Dielectric medium and to induce reoriented electric Dipole moments.

Permeability = the degree of magnetization of a material in response to an external magnetic field.

Phase Velocity = $\frac{\lambda}{t}$ wave length divide by frequency cycle time.

The speed of light reduces velocity in a material due to influence of the electric field on the material atoms (electrons) which response in oscillating in the same frequency but phased delay inducing their own electric charges [q]. The sum of all microscopic charges yields a new wave with phase delay that slows the original wave.

The refractive index has different values for different wave length. Diamond's RI=2.417. Materials RI are measured at Fraunhofer's [Na] (D1-line 589.592 nm yellow on the visible spectrum). Recent studies have shown that the refractive index decreases with pressure in a slope of $0.85 \times 10^{-4}/\text{kbar}$ at temperature of 22°C. Under temperature gradient (0-925K) also was found that refractive index increase with respect to temperature.

In other words refraction index describes the ratio between speed of light in a vacuum and the speed of light in the material. The high refraction index of the diamond increases the internal reflection and due to the diamond's geometry more sparkles and "Fire".

Critical angle and total internal reflection

We can derive from Snell's law what happens to wave front when it passes from one medium to another.

$$n_1 \sin \theta_1 = n_2 \sin \theta_2$$

n_1, n_2 are refraction indices of the mediums respectively. θ_1, θ_2 are the incidence and refraction angles.

When the angle of incidence reaches Critical angle, the angle of refraction = 90° (the refraction ray points along the medium surface). The expression for the Critical Angle (θ_c) can be derived from Snell's law: $\theta_1 = \theta_c, \theta_2 = 90^\circ$

$$\sin \theta_c = \frac{n_2 \sin 90^\circ}{n_1} = \frac{n_2}{n_1} \quad (n_1 > n_2)$$

$$\theta_c = \sin^{-1} \left(\frac{n_2}{n_1} \right)$$

Example: calculation θ_c of Diamond immersed in 2 mediums Ethyl alcohol and air where the refraction indices are n_1 (Diamond) = 2.419, n_2 (Ethyl alcohol) = 1.362 and n_3 (air) = 1.

$$\sin^{-1} \left(\frac{1.362}{2.419} \right) = 34.2^\circ \quad \sin^{-1} \left(\frac{1}{2.419} \right) = 24.4^\circ$$

All rays that exceed the Critical angle will not reflect in to diamond and will not contribute to the diamond sparkle.

$\theta_c (\text{air}) < \theta_c (\text{Ethyl alcohol})$ Therefore the diamond will sparkle more in the air.

Dispersion and Spectroscopy

An ideal diamond is transparent to light from the visible to near UV range (700nm -400nm) due to his large band-gap 5.49eV. The waves of the visible light spectrum simply don't have enough energy to excite an electron and produce a photon, however impurities like nitrogen contributes free electrons that can be excited by the visible light spectrum producing varying hues of color.

The diamond is a very good insulator because of the 4 valence electrons are completely locked so no "free" electrons can donate to the conduction. The band-gap is described as the Energy gap between the Valence band and the Conduction band. The Valence band is the highest rang of electron energy levels and all the orbitals are fully occupied. The conduction band most unoccupied band where electrons can be exited to create electric current.

To explain the formation of a band-gap in solid crystal we will use some concepts of Quantum mechanics theory. In a single atom the electrons can "stay" in a discrete energy levels and form orbitals. The electrons orbitals can be described in quantum mechanics as a mathematical wave function that defines the region where electrons can occupy the space in discrete quantum states and the probability distribution that system will be in any of the possible energy states.

The exact position of the electron is unknown because **Heisenberg uncertainty principle**, any measurement the electron (particle) position makes the momentum uncertain and vice versa.

$$\Delta x \Delta p \geq \frac{\hbar}{2}$$

Measurement the position of a particle with accuracy Δx yields a momentum alteration of:

$$\Delta p \geq \frac{\hbar}{4\pi\Delta x}$$

For example if we want to have a good idea where the electron is, we wish to locate it with in 0.05A (angstrom) or 5×10^{-10} cm interval. The measuring the position yields uncertainty in momentum according to **Heisenberg uncertainty principle**.

The momentum is given by

$$\Delta p = \frac{\hbar}{4\pi\Delta x} = \frac{6 \times 10^{-27}}{6 \times 10^{-9}} \cong 1 \times 10^{-18} \text{ gm cm/sec}$$

Since the mass of an electron is 9×10^{-28} gm we can calculate the uncertainty in the electron velocity

$$\Delta v = \frac{\Delta p}{m} = \frac{1 \times 10^{-18}}{6 \times 10^{-28}} \cong 1 \times 10^{-18} \text{ cm/sec}$$

The uncertainty that we have calculated show us that the electron velocity is large as the speed of light and it's very far from what we might expect the velocity of the electron to be.

Erwin Schrödinger equation describes the quantum state of a physical system. It allows us to calculate the energies of the system and the expression $|\psi|^2$ (mathematically solved only for hydrogen atom) represent how the probability of finding a particle varies from place to place. Together with the wave function we can predict the materials behavior and understand much deeply how to deal with phenomenon like diamond intrinsic defects and light absorption.

When an electron makes a transition between 2 energy levels it emits a photon (light). The photon has both a wave and a particle characteristics and the famous duality of light duality theory explains it. The fact that electrons produce light when they transient between energy level give us the explanation why materials have spectrums of discrete wave length according to their specific molecular structure. Excitation of electrons needs an energy source such as laser beam or external IR radiation. Every atom or molecule has its own wave lines absorption like a footmark that is a consequence of its atomic structure. This fact gives us a tool (spectroscopy) to investigate and explore the diamond impurities and deformations.

When several atoms are brought together to form a molecule their orbitals split proportionally to the number of atoms consist the molecule. When a large group of atoms ($1\sim 10^{20}$) form a solid the orbitals number becomes exceedingly large and the energy differences becomes minor, resulting in a continuous Band-Gap of energy rather than discrete energy level in a single atom. Band-gap in diamond is huge electron barrier and exciting electrons from the valence band needs a lot of energy (5.49eV).

The band-gap is a very powerful aspect that influenced the optical and electrical properties of the material. Thermal energy and pressure (HPHT) can cause an excitation to the conductive band and its reason why conductivity depends on temperature. At 77°K Band-gap value is 5.48eV and 5.47eV at 300°K.

The dependence of refraction on the wavelength of light is called **dispersion**. In gemology the dispersion is defined by subtracting the RI at 2 Fraunhofer lines: B=686.719nm, G=430.790nm. The high dispersion in diamond=0.044 ensures high degree of spectral colors.

Natural Diamond and synthetic diamonds are made of carbon atoms but most of them have impurities such as nitrogen and boron, as well as inclusion and dislocations, which result in various colors and clarity levels. This impurities influence the market value of the diamond because they have direct impact on the diamond's optical and physical properties (the 4 C's). Spectroscopy is a very powerful tool to investigate the absorbance and distribution of impurities accurately. The absorbance line can show varies nitrogen impurities and they help to indentify the group type of a diamond (Ia, IIa...). FTIR, Photoluminescence, EDXRF, WDXRF... spectroscopy will be discussed briefly on next section.

Spectroscopy

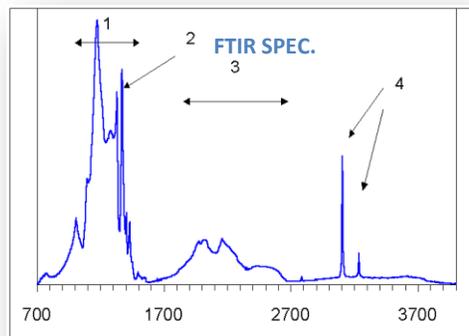
The equipment we use for spectrum analysis of the various diamond types differ from each other by their wavelength absorbance, sensitivity and the conditions the test is carried out. They consist of light source, detectors and computer system. Appropriate software needed for processing the raw data. There are quite a few instrument evolved in diamond diagnostic because the diamond happen to have different kind of defects and color variations and of course discriminating synthetic or treated diamonds from the natural ones.

The following table gives a short description of several methods used in diamond spectroscopy.

Method	Description
FTIR	Fourier transform infrared spectroscopy
Photoluminescence	Measures the Band-Gap energy.
EDXRF	Energy-dispersive X-ray fluorescence
WDXRF	Wave-dispersive X-ray fluorescence
UV-VIS-NIR	Uses light in the visible and adjacent near ultraviolet (UV) and near infrared (NIR) ranges.
Raman spectroscopy	Measures the influence of vibrational, rotational and other low-frequency modes in a system (Based on Raman scattering).
Fluorescence spec.	Analyzing Fluorescence radiation under UV excitation.

FTIR

FTIR works in a Spectrum range of 800-25k nm. Infrared spectroscopy exploits the fact that molecules have specific frequencies, at which they rotate or vibrate corresponding to discrete energy levels. This technique is used to identify chemical compounds based on how infrared radiation is absorbed. IR spectroscopy can differ between types Ia from IIa diamonds by showing nitrogen impurities as absorbance spectrum lines in regions 10000 nm to 7143 nm. The IR spectrum can show nitrogen impurities over 10 ppm that's why type Ia can be detected because it contains nitrogen impurities around 5000 ppm but Type IIa diamonds are less than 5 ppm, that makes it impossible to discriminate different type IIa diamonds by using IR absorption spectroscopy alone.



FTIR spectrum of IaB diamond type: **1.** Nitrogen impurities region **2.** B2 peak → A center. **3.** Diamond lattice self absorption **4.** Hydrogen peaks

Photoluminescence (PL)

We use Photoluminescence spectroscopy to see absorbance lines in diamonds that has around 5 ppm impurities and we can see the difference between IIa types of diamonds. The systems use a Laser beam (Light Amplification by Stimulated Emission Radiation) as excitation source usually Ar-Ion operating at 2 Wavelength 488nm and 514.5nm at 10 to 20mW. The diamond is cooled to $\sim 8^{\circ}\text{K}$ by He cryostat system. The cooling is needed in order to reduce “unwanted” emissions as a background noise and increases the system sensitivity and discrimination. For example the Following charts demonstrate the differences between FTIR and Photoluminescence. We compare 4 diamonds divide to type Ia and IIa with different C's.

Diamond types – grading by traditional methods

Sample	Type	Weight	Color	Clarity
A	IIa	1.02 ct	E	SI1
B	IIa	1.26 ct	E	SI2
C	IIa	0.117 ct	M Brown	VVS1
D	Ia	0.180 ct	Fancy Color	SI2

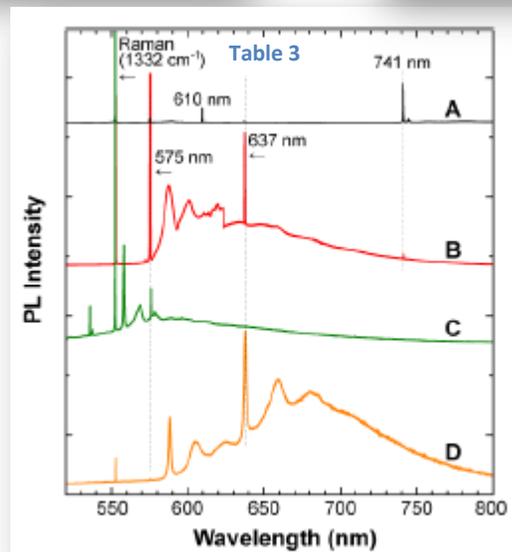
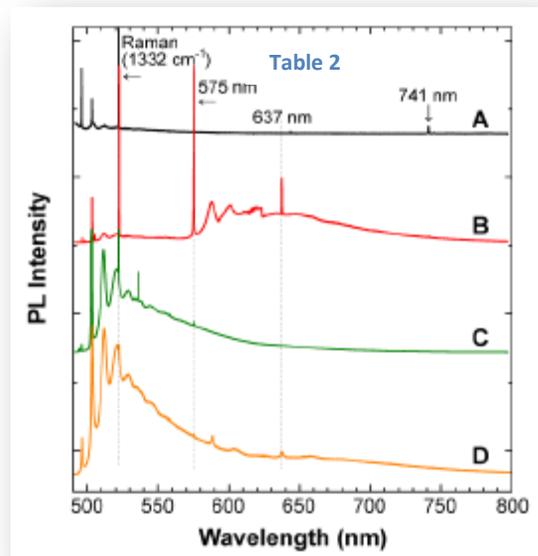
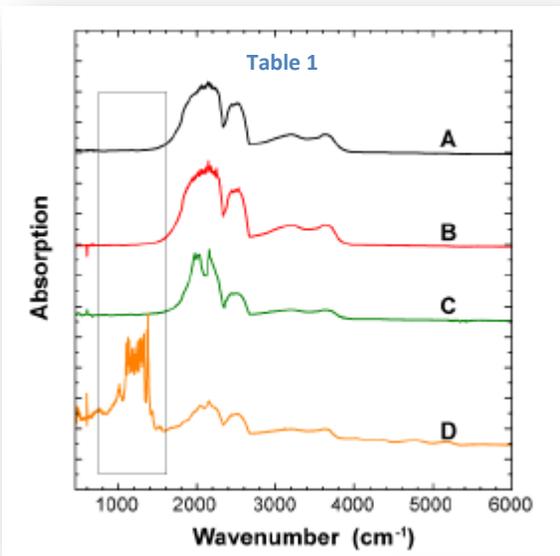


Table 1 shows IR spectrum of the diamonds. The signal from 1000 cm^{-1} to 1400 cm^{-1} is caused by Nitrogen impurities (marked by a rectangle). We can see it only on sample D which is type Ia. The 3 IIa types are very similar to each other. **Table 2, 3** shows Photoluminescence spectrum under 488 nm and 514.5 nm excitation. The lines at 575 nm and 637 nm are due to Nitrogen vacancies respectively. The lines at 522 nm and 552 nm are due to Raman scattering emissions (1333 cm^{-1}) this line is the fingerprint of a natural diamond.

Conclusion

The Photoluminescence show clearly the difference between the IIa diamonds. Sample **A** shows very little luminescence except for small peaks at 610 nm and 741 nm due to very little Nitrogen vacancies. Samples **B** show strong red luminescence besides the lines indicating the Nitrogen vacancies. Sample **C** has brown color but as we can see it doesn't have enough nitrogen so one can conclude that the brown color is due to structural dislocation like plastic deformation and this diamond is great candidate for HPHT treatment for color change. The Photoluminescence spectra give much more information on inspected diamond and therefore it improves the process of diamond commercial evaluation.

Raman Spectroscopy

The Raman spectroscopy is based on the Raman scattering effect. Scattering of photons due to incident light source causes a small amount of photons to inelastic scattered by an excitation. The scattered photons produce lower frequencies than the incident monochromatic light. This is Raman scattering. A Raman scattering of a laser light can be measured and the information on characteristic phonon modes can help to identify the material. The vibrational level is typically to the material inspected. One of the techniques being used is a Raman spectroscopy system that includes microscope which enables Raman analysis of samples on the micron scale.

Fluorescence

Differ from Raman scattering the incident light is completely absorbed and the system is transferred to an excited state. The major difference is that the Raman effect can take place for any frequency of the incident light. In contrast to the fluorescence effect where usually the absorbed photon is in the ultraviolet range, and the emitted light is in the visible range.

EDXRF and WDXRF

Basically X-ray spectroscopy consist of an high energy source beam of charged particles such as electrons or protons, or a beam of x-rays, is focused into the material. It reacts as excitation source. The excited electrons "jump" energy states and the emitted energy is in X-ray form. An X-ray dispersive spectrometer detects the emitted radiation. This radiation is characteristic of the atomic structure of the material inspected. EDXRF can detect concentrations from few ppms to 100%. WDXRF measures a specific wave length by using a crystal as a diffractive device. WDXRF system has better energy resolution ($\sim 5\text{ eV}$ laboratory grade) but very strict collimation is needed and the optics is expensive. EDXRF has lower resolution ($\sim 150\text{ eV}$) and it's less expensive.

Extrinsic Defects

Most natural and synthetic diamonds have impurities due to their natural growth conditions in a harsh environment and/or deformations in the crystal structure. These defects impart the diamond a variety of new characteristic, start with color changes and end with unstable structure.

Nitrogen Defects

type	Defect description	remarks
Ia	Nitrogen presents up to levels of 4000-4500 ppm (0.3 % wt), incorporated in interstitial lattice or aggregates.	98% of natural diamonds Most of them mixture of IaA/IaB
IaA	The nitrogen impurity is in a form of N couple bonded as a molecule and replacing a Carbon atom in the lattice.	Doesn't effect color A center
Ib	500 ppm of nitrogen (0.06 % wt). Incorporated as substitution point defects. Most of synthetic diamonds are of this type. Isolated nitrogen atoms.	Colors: Clear, Deep yellow to brown . C center
IaB	Color caused due to amorphous carbon in the planes The N atoms aggregate in large numbers	Color yellow/brown B center
IIa	Effectively free of nitrogen impurity. Very rare in nature, these diamonds have enhanced optical and thermal properties.	colored pink, red, or brown
IIb	Extremely rare in nature; p-type semiconductor due to boron impurity up to 50 ppm.	Color: steely blue or grey

A center: Couple of Nitrogen atoms that replace a carbon atom in the diamond lattice. It's the most common defect in natural diamonds.

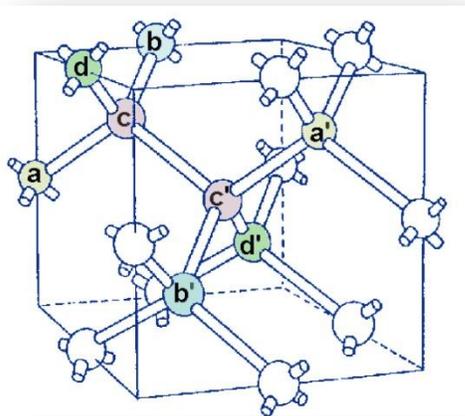
B center: Four Nitrogen atoms surrounding a vacancy.

N3 center: three Nitrogen atoms surrounding a vacancy in a flat formation (peak WL: 415nm).

N2 center: two nitrogen atoms of different configuration then A center (peak WL: 478nm).

C center: Isolated Nitrogen atoms

GR1: Lattice defect



Diamond lattice formation defects

Divacancy: $V \rightarrow c, c'$

[N-N] A center: $N \rightarrow c, c'$

B H4 center: $N \rightarrow a', b', d', c V \rightarrow c'$

[3N-V] N3 center: $N \rightarrow a', b', d' V \rightarrow c'$

[N-V-N] H2, H3 center: $N \rightarrow a', b' V \rightarrow c'$

[N-V] center: $N \rightarrow c, c'$

Leveling Diamond color by HPHT

The HPHT process is simply trying to mimic the natural geological conditions that occur at 450 km depth within mantle layer of mother earth. The conditions evolve very high pressure and temperatures (58,000 atmospheres, 2000°C) in order to make changes to the crystalline structure of natural diamond or to grow a synthetic one. A great deal of debate surrounds the aspects and influence of such a process on the Diamond industry.

Companies such as Gemesis located in Florida, Apollo etc. use different methods to manufacture artificial diamonds. Gemesis uses high pressure, high temperature chamber with a metal solvent as catalyst and Apollo uses a CVD (Chemical Vapor Deposition) which is more promising in the future to come cause, it can produce extremely pure crystal that will make them almost undetected by current analysis equipment.

Known facts

Most natural Type Ia diamonds have a brown color. A brown color may result from a mixture of many other colors. For example, the brownish color may result from a mixture of yellow color (such as from isolated nitrogen atoms (C Centers) or N₃ centers) with some black coloring (such as from submicroscopic inclusions of graphite). The mixture of yellow and black colorings will produce a brown color. Further, a brown coloring in a diamond can be formed from a mixture of color centers that produce a green and red coloring in a diamond. An infinite number of color combinations that produce a brown color are possible. Therefore, it is generally impossible to determine the color centers causing the color of a diamond by its color alone. One must analyze the spectral absorption of natural type Ia diamonds in the IR and VIS spectral region to know what the centers responsible for the diamond color are.

Type II diamonds are divided into Type IIa and Type IIb. Type IIa diamonds have impurities of nitrogen at less than a ~1 PPM level. Type IIb diamonds contain boron of few ppm's and the boron concentration always exceed any nitrogen concentration in the crystal. Type IIb diamonds are blue in color and are extremely rare, and thus have a high value per carat as jewelry items.

A phenomenon known as plastic deformation a slip or/and rotated plains can also cause the appearance of the brown and pink colors.

The HPHT process can alter the Nitrogen centers of the diamond due to the healing of plastic deformation and increasing the mobility of Nitrogen atoms. On going to high temperatures, the mobility of nitrogen increases orders of magnitude. Also, the higher temperatures can break up stable B centers and A centers. Because nitrogen atoms tend to assemble to the aggregate locations with lower energy, Diamonds with C form aggregates can be converted to the A form. An A form can be converted to B1 Nitrogen center with platelets b2 centers where most of the nitrogen is in B1 form. During the "heal" vacancies (V) are released and are trapped by the

nitrogen pairs (N-N) to form N-V-N centers. Single N atoms also form N-V centers. The HPHT process creates new centers [N-V-N] +/- and N-V.

HPHT effect on Diamond types

Type	Original color	After HPHT	Cause	Spectra
Ila	Brown	No color, pinkish, light yellow	Plastic deformations healed, rupture bonds repaired, some N-V centers created	637nm [NV ⁻] 575nm [NV ⁰] 3760 <i>cm</i> ⁻¹
Ilb	Brown	Blue	Healed deformations “leave” the diamond with the Boron as main impurity	From UV to 250nm
IaB	Brown	light yellow-grey	New A and new C Centers and fewer B Centers. (sometimes N3 centers appear giving light yellow-grey color)	
IaA	Colorless Light yellow		new C and new B centers and less A centers	
Ib	yellow	Becomes Ia Greenish-yellow	new A and new B Centers and less C Centers	*
IaA/B	yellowish	Strong yellow, brownish-yellow, olive, or green	New C center less A and B centers	503 nm 986 nm
Ia	Transparent colorless IaA			
	Light yellow IaA			
	Yellow IaB			
	Brown IaB			

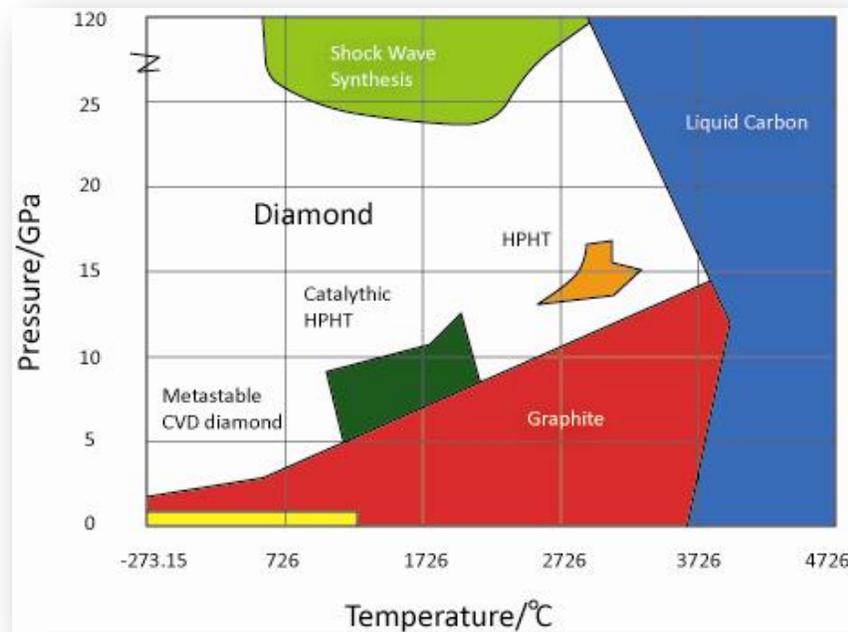
*FTIR, 1130 *cm*⁻¹ band in type Ib crystals is normally used to characterize the Ib crystal. For purposes of accuracy, the 1345 *cm*⁻¹ band which is correlated to type Ib nitrogen is used. There are two forms of type Ia diamond, an A band form and a B band form, and these forms are differentiated by their infrared spectra wherein the A band form has its absorption band at 1280 *cm*⁻¹ and the B band form has absorption band at 1175 *cm*⁻¹.

Correlation between centers and colors

Center	Description	Color
H3	2 Nitrogen atoms + Lattice defect (Vacancy)	Yellow
H4	4 Nitrogen atoms + Lattice defect (Vacancy)	Yellow
N-V	1 Nitrogen atom + Lattice defect (Vacancy)	Purple
GR1	Lattice defect (Vacancy)	Bluish green
N3	3 Nitrogen atoms	Light Yellow

The process of HPHT is putting the diamond under extreme conditions. In order to achieve the strongest colors and hue's its necessary to reach the highest temperatures and pressure and the diamond gets very near to graphitization. In nature the growth of the diamond takes around $\sim 10^9$ years but in the artificial course of action we use metals as catalyzers in order to achieve faster processing times (a few min. to hundred of Hours) and a lower pressures and temp as we can see in the Carbon phase diagram. Trace of these metals can be found in the synthesized diamond. Today most HPHT diamond are growth with Ni-Fe (Nickel, Ferrum) alloy as a catalyst but Cu (Copper), Pd (Palladium), Cr (Chromium), Ru (Ruthenium), Rd (Radium), Os (Osmium), Ir (Iridium), Ta (Tantalum) , Pt (Platinum) and alloys of them are used also. The difference in diamond graphite solubility in metal is the base for the diamond crystallization.

Phase Diagram of Carbon



Catalytic HPHT occurs in a lower temperature and pressure then non catalytic HPHT.

Conclusion

Natural diamonds, as we can see contain multiple N centers and annealing them will produce different types of color centers. It's very difficult to find a rough stone with exactly the centers that one need in order to get a specific color. For example to produce a purple color we will need a rough diamond that consist only N-V centers, that will be very hard to find, so instead we can use synthetic diamond which is very easy to make that matches exactly the need. A lot of patents have been registered regarding diamond color leveling and the influence on the various markets is not clear yet. The magic and the mystery surrounds the natural diamond, especially the rare ones is still on top and will remain so for very long time.

References and special thanks

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Benzion Menashe has spent many years learning science (Tel Aviv, Jerusalem universities) Physics, Electronics and Chemistry. In the high-tech region he worked in the R&D department and gains a lot of experience regarding international sales as a sales manager and FAE.