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D, E, F, G or HPHT

Getting to the heart of high pressure, high temperature treatment

By Hemdeep Patel



Nearly 10 years ago, two American companies created the first commercially viable method of turning brown to dark brown diamonds into white. Prior to this, applying high pressure, high temperature (HPHT) treatment was primarily used to produce diamonds in fancy yellow, greenish yellow, and a range of yellowish brown. In the years since, numerous HPHT treating facilities have sprung up globally to supply an ever-increasing demand for high-colour and good-quality diamonds.

In short, this treatment takes cheaper, unsaleable brown diamonds and changes them into highly sought-after and expensive white diamonds in the D to H colour range. These stones are bought and sold in a highly competitive market, where any edge in price can be an advantage. The practice of selling HPHT-treated diamonds without disclosure is viewed as fraudulent and deceitful. Although the average consumer may be unaware of HPHT, part of a diamond's allure and romance is based on the fact it takes nature's millions of years to form. A natural diamond is unaltered and unchanged by human hands, except through cutting and polishing.

The HPHT process leaves behind key identifying characteristics, allowing gemmologists and jewellers to identify treated diamonds. Unfortunately, the methodology and technology needed to conclusively identify HPHT diamonds is beyond the scope of the standard equipment used by most appraisers and gemmologists.

In fact, to accurately identify HPHT-treated stones, specialized spectrometers that analyse diamonds at the atomic scale are needed. Unfortunately, the significant financial resources needed and expertise required to operate these devices make it prohibitive for most gemmologists to access. Another fact making HPHT identification problematic is that a vast number of diamonds are bought and sold on preliminary reports, where the diamond is only graded for the four Cs and no additional information is provided.

This lack of information is due to inadequate equipment for HPHT identification at the laboratory. Additionally, the cost of further treatment identification, which is time- and labour-intensive, is in addition to the preliminary report. Therefore, this has allowed a

number of HPHT diamond treaters and traders a unique opportunity to sell these colour-enhanced stones without any disclosure to the buyer.

As the jewellery industry struggles with the lack of treatment disclosure, identification, and education, HPHT-treated diamonds continue to make their way into the market. By following a few simple steps, jewellers and gemmologists can identify suspected HPHT-treated diamonds. The suspected stones can then be submitted to a gemmological laboratory, appropriately equipped for testing HPHT-treated diamonds for conclusive identification.

The treatment

The methodology of HPHT is quite simple. Diamonds were created about 100 million years ago some 250 to 350 km below the earth's crust in temperatures of about 1400 C and pressure in the 70 kbar range. It is during this period that a diamond's colour is imparted by the applied pressures, temperatures, and impurities in the environment. Recreating these conditions in a laboratory setting while applying these temperatures and pressures to a diamond's crystal lattice structure in a controlled manner results in colour change. Further, the type, amount, and manner in which impurities within a diamond's crystal structure are organized—also known as diamond types—predetermine the resulting colour.

The C and N of diamonds

Carbon (C) is the building block of organic matter, including diamonds. The way in which carbon atoms are organized determines its physical properties. When carbon creates an octahedral crystal lattice, its stable physical structure is diamond. When it forms a tabular crystal lattice, its stable physical structure is graphite (Figure 1).

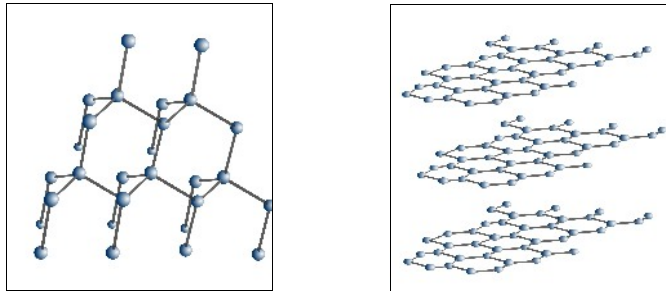


Figure 1 The arrangement of carbon atoms will dictate the strength of the carbon bonds and its final physical form. Carbon atoms within a diamond are arranged forming an octahedral crystal habit. It is this crystal habit which makes the diamond the hardest known mineral with a mohs hardness of 10. Carbon atoms within graphite are arranged forming a six sided tabular crystal habit. It is this crystal habit which makes graphite flakey in one direction with a mohs hardness of 1-2.

All diamonds fall within four main type classifications, which refer to their crystallographic defects. (Figure 2) Type I diamonds are characterized by nitrogen (N) impurities in concentrations of 10 ppm (parts per million) and greater.

Diamond type	Impurity, arrangement & amounts parts per million (ppm)	Naturally occurring colours	Possible colours due to HPHT
Type Ia	Nitrogen (N) found in pairs or aggregates in amounts greater than 10 ppm	White, light yellow & brown	Yellow, intense yellow & brown Subset Type IaB – white, light yellow, light brown
Type Ib	N found as single atoms throughout in amount greater than 10ppm	Intense yellow & brown	Yellow, intense yellow & brown
Type IIa	N found throughout at amounts much less than 10ppm	White & brown	White, pink, light brown
Type IIb	Boron (B) found throughout at any amount	White, brown & blue	Blue

Figure 2 *Diamond type, corresponding naturally occurring colour range and resulting colour range resulting from HPHT.*

Type I diamonds are further classified by how nitrogen atoms are arranged within their crystal structure. The majority of the world’s gem-quality diamonds are type Ia, which means nitrogen atoms are found in pairs or groups and give rise to a colour range of white to light yellows and brown. Type Ib diamonds, where nitrogen is found isolated throughout the crystal structure, are very rare.

Type II diamonds are characterized by nitrogen impurities well below 10 ppm and are further classified by any additional impurities within their crystal structures. Type IIa make up a small amount of the world’s gem-quality production, but come in colours ranging from browns and yellows to pinks and reds.

Type IIb have no nitrogen impurities. Instead, these diamonds have boron impurities within their crystal structures, giving rise to a grey-blue to blue colour, as well as a semi-conductor quality. Type IIb make up a very small number of gem-quality diamonds, the Hope Diamond being perhaps the most famous of these.

Regardless of their classification, brown diamonds are the result of anomalies known as plastic deformation found within the crystal structure (Figure 3) In type IIa and IaB (a subset of type Ia), this feature is what makes these stones ideal candidates for HPHT treatment.

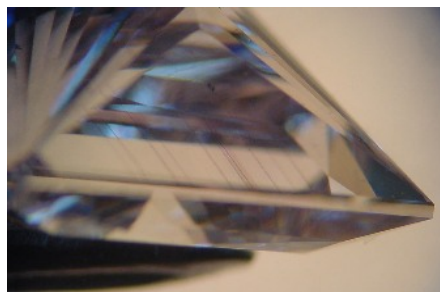


Figure 3 Defects, known as plastic deformation, within the diamond's crystal structure can give rise to the brown colour seen in diamond. In many instances, these defects and its resulting brown colour can be seen as brown bands which follow the diamonds growth plane

How does HPHT work

The HPHT process acts directly on the diamond crystal structure by altering or repairing the defects within the carbon crystal lattice. The resulting colour of the treatment depends on the diamond's type. With their higher concentration of N impurities, the majority of type I diamonds turn into a fancy yellow to a greenish yellow. Type IaB can be changed from a light or dark brown to a colour range of D through K. Type IIa diamonds, which are relatively free of nitrogen impurities, can be changed into pink or white. In the case of Type IIb, applying HPHT can impart a stronger blue colour.

The technology used to treat diamonds with HPHT is very sophisticated and extremely expensive. Derived from the oil and gas drilling industry, the cubic press was initially used to create polycrystalline diamond compacts (PDC) drilling bits, which are fused or sintered with metal catalysts and micron-sized synthetic diamonds. The conditions needed for effective diamond sintering are pressures of about 60 kbar and a temperature range of approximately 1400 C, making PDCs the most effective and abrasive drilling material to date.

There are many factors to consider when applying HPHT. As mentioned earlier, a diamond is formed in the earth's crust over hundreds of thousands of years at temperatures of 1400 C and pressures of 70 kbar. Therefore, any naturally occurring colour changes take equally as long.

To make HPHT treatment commercially viable, a much shorter time frame was needed. (Figure 6) outlines the stable physical structure of a diamond's carbon crystal lattice over a wide and extreme temperature and pressure range. Since modern cubic presses are effective at 60 to 70 kbars, the time it takes to change a diamond's colour can only be shortened by increasing the temperature to approximately 1900 to 2300 C. As seen in Figure 6, this combination of pressure and temperature is beyond the stable area of diamonds and into that of graphite. In other words, carbon atoms naturally organize themselves from a diamond octahedral crystal into a graphite tabular crystal lattice. HPHT treatment must be very short and monitored extremely closely to avoid graphitization, which is the transformation of a carbon structure into graphite.

The materials needed, how it's done, and what do I get

The quality of the initial diamond material submitted for HPHT treatment is the most important ingredient for successful application. Diamonds that are VS2 or better (*i.e.* where the inclusions are relatively small and contained within the stone) are good candidates as opposed to diamonds with surface-reaching feathers and fissures. With these stones, applying the treatment runs the risk of the inclusions extending further into the diamond or, in some cases, causing it to break apart.

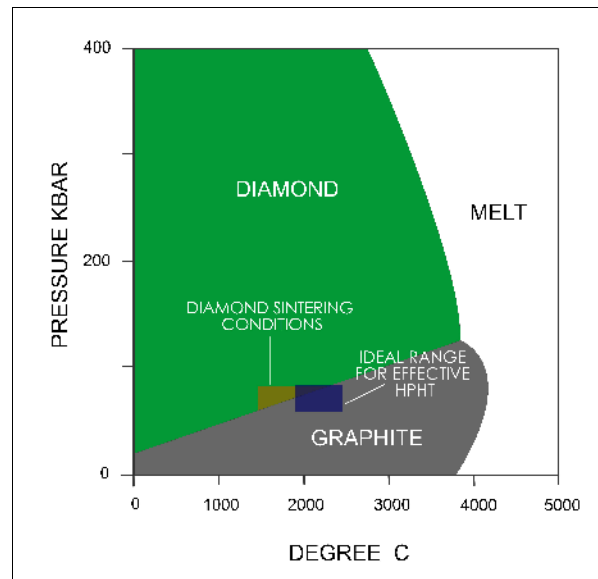


Figure 6 *Diamond and graphite equilibrium graph outlines the physical state of a stable carbon crystal habit through a wide range of extreme temperatures and pressures.*

Once a good candidate is found, the diamond's type is then determined through the use of a Fourier transform infrared spectrometer (FTIR). This provides some indication of what the final colour might be once the HPHT treatment is complete.

Once a diamond is selected for HPHT treatment, it is then prepared for the cubic press. First, the stone is placed in a heating sleeve, which is then inserted into an insulating medium before going into the cubic press. The device quickly increases the temperature within the heating sleeve, as well as the pressure exerted on the diamond. HPHT treatment only takes a few minutes to complete.

The treated diamond emerging from the cubic press exhibits a frosted skin, which is the result of graphitization that has occurred on its surface. The diamond is then re-polished, resulting in a weight loss of about two to four per cent.

Identifying HPHT-treated diamonds

Suspected HPHT-treated diamonds can be red flagged using standard gemmological equipment. The first step to identifying suspected HPHT-treated D to K diamonds is to distinguish diamonds that can be treated to produce colours in this range, which are generally type IIa and IaB. In the case of HPHT-treated diamonds, all type IIa and the rare type IaB diamonds are invisible to shortwave ultraviolet (UV) light and can be identified with the use of a diamond spotter (Figure 7). Once identified as a type IIa or IaB, the diamond can be further examined for any obvious graphitization inclusions under a microscope or with a loupe.

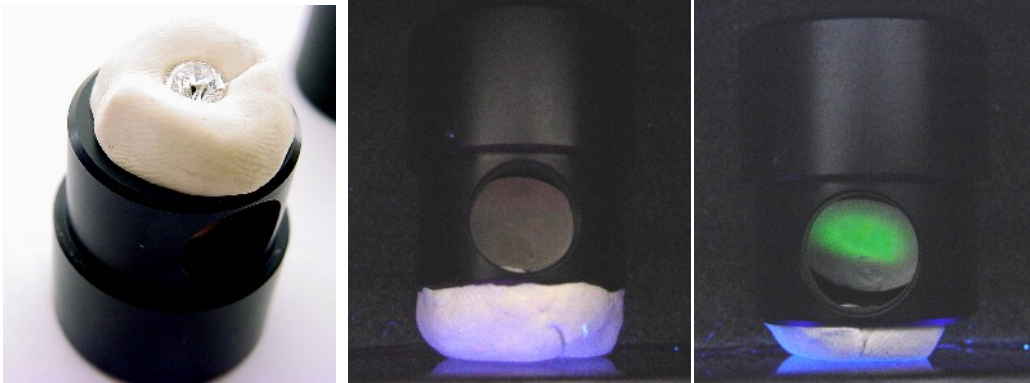


Figure 7 a) The SSEF spotter can be used to red flag possible HPHT treated D to K coloured diamonds. The spotter is comprised of a cylinder that has a diamond mounting window at one end of the cylinder. A larger viewing window on the cylinder and a fluorescent surface on the opposite end of the mounting area. b) The test diamond can be easily mounted on mounting area with the use of putty. c) The spotter is placed on an ultra violet light source with putty and the mounted diamond in between. Through the larger viewing window on the cylinder, the result of the spotter can be viewed. No reaction on the fluorescent surface indicates that the diamond has not been treated through HPHT d) When the fluorescent surface changes to a lime green, the diamond can now be red flagged for possible HPHT treatment and should be forwarded to a gemological laboratory equipped to conclusively identify if the stone has been HPHT treated.

For diamonds with a clarity grade of VS2 or lower, it may be possible to identify graphite inclusions that would indicate the presence of HPHT treatment. In these cases, graphite appears as a flat disc and is the result of the treatment pushing the diamond's crystal structure outside its equilibrium and into the stable state of graphite. (Figure 8). For diamonds with clarity grades better than VS1 and with no obvious inclusions, a microscopic examination may reveal nothing. Therefore, these diamonds can only be conclusively identified as HPHT-treated through spectrophotometer testing.

Since cubic presses act primarily on a diamond's carbon crystal lattice, they leave behind some evidence of treatment. With the use of cross-polarizing filters, a diamond exhibits a strain—also known as a Tatami strain—regardless of its colour. In the case of brown diamonds in the colour range of H and lower, the Tatami strain may be naturally occurring. However, with diamonds in the D to H range, and colours in the fancy yellows to intense pinks, Tatami strains may be the result of HPHT (Figure 9).

In addition to the previously mentioned gemmological techniques, colour may be used to identify possible HPHT treatment. For example, a green to greenish yellow colour may be the result of HPHT treatment. Also, since naturally occurring pink and red diamonds are extremely rare, these can be considered suspect until gemmological testing proves otherwise.

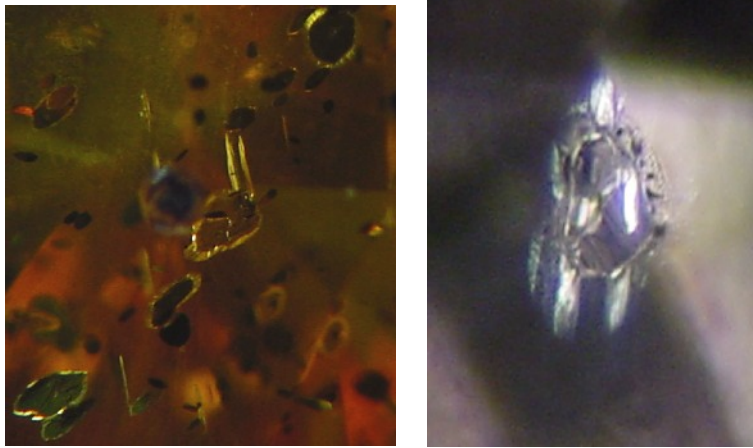


Figure 8 During the HPHT treatment, the diamond's crystal structure can become unstable and cause the carbon atoms to re-align themselves into the more stable graphite state. a) Graphite inclusion pressed into flat discs with a frosted outer edge. b) Graphite inclusions appear on the outer edge, along the top right quarter, of an included crystal. Both forms of graphite can only be used as a possible indication of HPHT treatment.

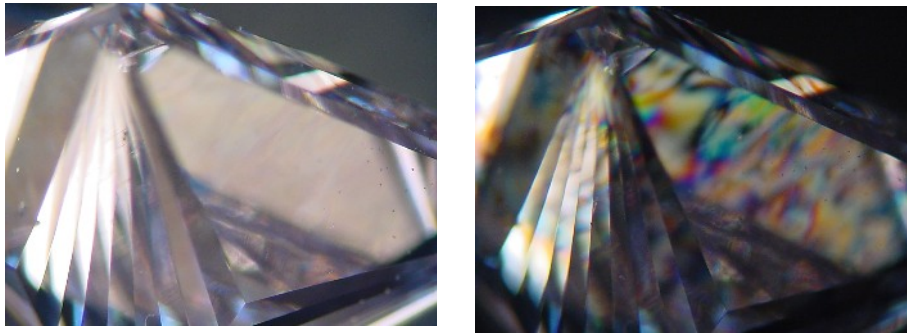


Figure 9 The pressures the diamond is put under either during its formation in the earth's crust or as the result of HPHT can cause a strain in the diamonds crystal structure, known as Tatami strain. The strain pattern becomes very obvious when the diamond is viewed under cross polarized light.

Diamonds exhibiting any of these characteristics must be submitted to an advanced gemmological laboratory, since conclusive identification can only be determined through an analysis of the diamond's absorption spectra. By using an ultraviolet, visible, near infrared to infrared (UV/Vis/NIR/IR) Raman spectrophotometer at -196 C with the use of liquid nitrogen, a very detailed analysis of the diamond's spectra can be conducted. By comparing a test diamond's absorption spectra to that of a natural untreated diamond, any key deviations can reveal the application of HPHT. (Figure 10). These deviations are the result of repairs and/or alterations in the defects within the diamond crystal structure and can be easily identified by any new, changed, or deleted key absorption lines.

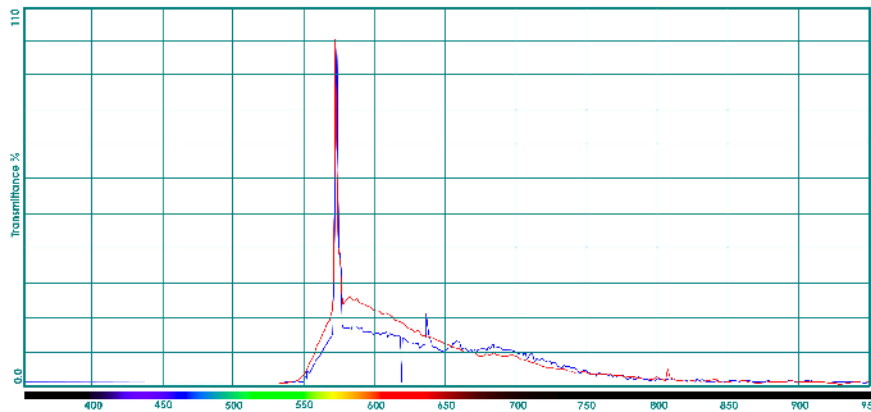


Figure 10 HPHT treatment leaves some tell tale signs of its effect at the atomic scale. a) These effects can only be seen through the spectra of the diamond that is created with the use of a Raman laser. b) There are apparent deviations in the spectra between a natural untreated diamond and a natural HPHT treated diamond. These deviations can vary depending on the diamond type, changes to the HPHT treatment recipe and if additional irradiation or annealing treatments have been applied to the diamond. Through the detail analysis of these deviations, it is possible to identify HPHT treated diamonds. Figure provided shows a comparison of a natural untreated white diamond (red) and natural HPHT treated white diamond (blue).

The prevailing myth of HPHT diamonds

As HPHT diamonds made their way into the marketplace, dealers and treaters worked hard to minimize the impact of the words used to describe them, shying away from terminology such as ‘treated’ and ‘colour enhanced.’ They have done so by introducing HPHT diamonds as the natural completion of a very long geological process, which yields a white D to K diamond. In other words, if a brown type IIa or IaB diamond was left within the earth’s crust, geological processes would have eventually turned them white.

However, what dealers and treaters fail to mention are two key facts. First, even though the diamond is subjected to pressures similar to HPHT when still in the earth’s crust, the environmental temperature for effective treatment is well outside the ‘natural’ range. While diamonds are exposed to temperatures of approximately 1400 C during the geological process, the temperature must be between 1900 to 2300 C for HPHT to be effective. Secondly, the time frame between geological processes and HPHT cannot be compared. The diamond still left in the earth will change on a geological time scale, measured in the hundred of thousands of years, whereas the changes occurring as a result of HPHT is measured in minutes.

JVC, disclosure, and where we go from here

Jewellers Vigilance Canada (JVC), the American Gem Trade Association (AGTA), and many other trade-governing bodies have taken a very strict view on the handling of HPHT-treated diamonds. Basically, full treatment disclosure throughout the supply chain from the initial trader and treater to the final consumer is mandatory. Further, ignorance is not a defence against non-disclosure in the event of any claims or legal actions.

With the growing number of treaters and traders worldwide, the supply of HPHT-treated diamonds has also increased and it is now possible to find treated stones in weights ranging from one to 10 carats. Also, a number of traders have streamlined their product line and carry only HPHT diamonds. This trend does not show any signs of slowing down since recently discovered diamond mines appear to produce primarily type II and low nitrogen type I diamonds.

Within the primary markets of India, Belgium, Russia, and Israel, HPHT diamonds are readily traded with full disclosure accompanied by full gemmological reports and laser inscription. Unfortunately, some of these diamonds make their way into secondary markets without disclosure reports and with the removal of laser inscription through polishing. Additionally, a minority of treaters have changed their treatment recipe to dampen any obvious HPHT absorption spikes. Unfortunately, they are unwilling to share their procedure with laboratories, since they claim it is part of their intellectual property.

What we are left with are some key identifying features that can allow us to red flag possible HPHT-treated diamonds. Jewellers, diamond traders, wholesalers, and gemmologists must be more vigilant when buying or grading fancy-coloured diamonds as well as high colour and clarity diamonds. Diamond buyers should ask for reports that go beyond the four Cs whenever making purchases of one-carat stones or bigger in VS2 H or better diamonds. A simple investment in a diamond spotter can be the first step in red flagging possible HPHT-treated diamonds.

With the unethical and illegal practice of non-disclosure, jewellers, diamond traders, wholesalers, and gemmologists need to educate themselves to stay on top of issues that can adversely impact the jewellery industry and specifically their businesses.

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[FIGURE] Figure 2

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