De Beers' Research Yields HPHT's Secrets

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With the announcement in March 1999 by General Electric and Lazare Kaplan International (LKI) at color-improved High Pressure-High Temperature (HPHT)-treated diamonds were hitting the market, the major gemological laboratories in Europe and the U.S. launched a surge of research initiatives. In fact, from the fall of 1999 to the winter of 2000, there were articles on HPHT-treated diamonds in each of six consecutive issues of the GIA journal *Gems & Gemology*.

In an effort to determine how best to identify HPHT-treated diamonds, the De Beers organization has also been very active in researching the HPHT diamond annealing process. This work is a collaboration between the Diamond Trading Company (DTC) Research Centre, Maidenhead, United Kingdom, and De Beers Industrial Diamonds' Laboratory in Johannesburg, South Africa. The Maidenhead laboratory's sophisticated spectroscopic equipment and expertise in characterization, combined with the Johannesburg laboratory's access to industrial diamond synthesis presses and expertise in high-pressure engineering, has led to substantial progress.

Detailed characterization research carried out at the DTC Research Centre and at some of the major gemological laboratories has led to the discovery of a number of features that can indicate HPHT treatment. Techniques include ultraviolet/visible and infrared absorption spectroscopy and, most importantly, laser-excited photoluminescence (PL) spectroscopy, which requires the sample to be cooled with liquid nitrogen to a temperature of –196°C.

The latter technique is particularly important for type II diamonds because it is such a highly sensitive technique. Type II diamonds are very pure and highly sensitive measurements are required to detect the very low concentrations of defects that they contain. Several of the major gem-testing laboratories have invested in this sophisticated PL equipment and are therefore in a position to identify the vast majority of HPHT-treated diamonds.

INVENTING THE TOOLS

One of the roles of the DTC Research Centre, in support of the De Beers Gem Defensive Programme (see page 46), is to assist gem-testing laboratories by developing practical instruments to deal with complex identification problems. To this end, a low-cost, easy-to-use instrument, currently in prototype form, is being developed for rapid screening of potentially HPHT-treated type II diamonds (see page 51). This instrument will not replace the sophisticated laboratory equipment but it will greatly reduce the number of stones that will need to be examined in such a time-consuming way.

Another role of the DTC Gem Defensive Programme is to anticipate future developments in treatment techniques (and also diamond synthesis techniques) that could challenge current identification methods, and to research new techniques that will continue to enable reliable identification to take place. Initially, research both by De Beers and the major gemological laboratories concentrated on areas of current commercial concern. In this area, detection criteria have been firmly established. The initial work has now developed into investigation of wider ranges of starting material, and treatment conditions in which identification may be more subtle. This forward-looking research is currently a very active area for De Beers scientists.

NO MAGIC BOX

So where do we stand at present in terms of the practicalities of detection? There certainly is no magic "black box" that can tell an HPHT-treated stone from an untreated stone with 100 percent accuracy. But this

would be an unrealistic expectation. Apart from the relatively trivial task of distinguishing diamonds from simulants, no gemological identification task can be achieved with a single, simple, fully automatic technique. The question is, can procedures and, where appropriate, instrumentation be developed that will allow sufficient numbers of stones to be examined cost effectively so as to address the concerns of the trade?

The sophisticated PL equipment that is now in use in the major gem laboratories can identify the vast majority of HPHT-treated diamonds but there will always be a small proportion for which the identification is uncertain. There will also be a small proportion of untreated stones that may be misidentified as treated. With a material as complex and varied as natural diamond such problems are inevitable.

Is the PL equipment so expensive and the measurement so time consuming as to be of little use in practice? It is certainly true that the need to make such measurements has greatly increased the workload of gem laboratories and has led to increased delays in having stones graded. But practical means of screening diamonds to reduce the number that need to be looked at in detail are being developed and implemented in grading laboratories. The fact that laboratories are willing to invest in such equipment demonstrates their determination to achieve the best results possible and so maintain the integrity of natural, untreated diamonds.

Will future developments in treatments invalidate the identification techniques? The honest answer, of course, is that one cannot be sure. But both De Beers and those gem laboratories that carry out research are committing considerable resources to address this issue. Three years ago, HPHT treatment was generally believed to be undetectable. The fact that this is certainly not the case today demonstrates what can be achieved in a relatively short time by dedicated research.

PRACTICAL ADVICE

What practical advice can be offered to the trade to help identify potentially HPHT-treated diamonds? Fancy yellow, greenish-yellow, pink and blue diamonds need to be referred to a suitably equipped gem-testing laboratory. For greenish-yellow diamonds, intense green fluorescence is a good indication that the stone may be HPHT treated but, ultimately, laboratory examination will be required.

Colorless and near-colorless diamonds should first be checked to see if they are transparent to ultraviolet (UV) radiation, and are therefore likely to be type II. This is accomplished using a short-wave UV lamp and a UV-sensitive phosphor — simple equipment that is commercially available. Colorless and near-colorless diamonds that are UV transparent should be referred to a suitably equipped gem-testing laboratory.

What about diamonds that are too small to be cost effectively submitted to a gem-testing laboratory? Colorless and near-colorless stones can still be checked to see if they are UV transparent or opaque. Those that are UV opaque will be type I and therefore unlikely to have been treated. For the very small proportion of diamonds that are UV transparent and therefore likely to be type II, the only realistic option is to insist upon a written declaration from the supplier that the stones are natural and untreated. For fancy yellow, greenish-yellow, pink and blue diamonds, the same written declaration should be insisted upon.

THE RULES

In the U.S., the Federal Trade Commission Guides §23.22 state that "it is unfair or deceptive to fail to disclose that a gemstone has been treated if … the treatment has a significant effect on the stone's value. The seller should disclose that the gemstone has been treated. Note to §23.22: The disclosures outlined in this section are applicable to sellers at every level of the trade."

The rules of the International Jewellery Confederation (CIBJO) state in Article 6.1 of the CIBJO *Diamond Book* that "if the natural color of a diamond has been artificially altered, it has to be clearly declared as 'treated,' 'artificially colored, or 'irradiated.'"

In support of this position, the DTC requires all its sightholders to abide by its Best Practice Principles, which state, "We are committed to the highest industry ethics including the following: …full disclosure at all levels of the diamond distribution chain and, most importantly, to consumers, of all treatments to natural diamonds and compliance with rules, regulations and guidelines published from time to time by the diamond industry's governing bodies."

HOW HPHT WORKS

Brown diamonds, both type I and type II, as part of their geological history have been subjected to mechanical stresses at elevated temperatures while deep within the earth. This causes their crystal lattices to become deformed — a process known as plastic deformation. This process introduces linear defects into the crystal, which are known as dislocations. The brown color is associated with the presence of these dislocations.

When the diamond crystal is subjected to very high temperatures, the structure of the dislocations is modified, causing the brown coloration to be reduced. At these very high temperatures, diamonds will convert to graphite unless very high pressure is applied. This is done using the same kind of equipment as is used for diamond synthesis, e.g., the conventional "belt" presses developed by General Electric, the cubic or prismatic presses used by NovaTech or the BARS presses developed in Russia. Such equipment is complex and relatively expensive.

Typical HPHT conditions can be in excess of 2000ºC and 60,000 atmospheres. Use of such extreme conditions is not without its dangers. Diamonds with inclusions and fractures can be broken and surfaces can become frosted and pitted.

Type I diamonds contain nitrogen as a major impurity, typically present at levels of several hundred parts per million. Type II diamonds are extremely pure, with nitrogen concentrations of less than about one part per million. A very small proportion of type II diamonds are classified as type IIb, due to the presence of trace amounts of boron impurities, which give rise to a blue color. In type IIa diamonds (i.e., those type II diamonds that are not type IIb), if the brown coloration is reduced sufficiently by HPHT treatment, the result is a colorless, near-colorless or sometimes a pink diamond.

Less than 1 percent of natural diamonds are type II of adequate quality to be suitable for HPHT treatment. In brown or brownish-gray type IIb diamonds, which are of course extremely rare, a pale blue color can be produced by HPHT treatment. In brown type I diamonds, HPHT treatment produces particular nitrogen-containing defects which give rise to a fancy yellow to greenish-yellow color, often with strong green fluorescence.

DTC Research *Comes to* DIAMONDS' DEFENSE

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The aim of the Diamond Trading Company (DTC) Gem Defensive Programme is to maintain consumer confidence in the integrity and mystique of natural untreated diamonds. Consumer confidence can be adversely affected by any misrepresentation of diamonds, as for instance treated diamonds being sold as untreated, synthetic diamonds being sold as natural and simulated being sold as diamonds. It is, of course, perfectly legitimate to trade in treated diamonds, synthetic diamonds and simulated, provided they are declared as such. It is the responsibility of the diamond industry to ensure that reasonable steps are taken to safeguard the interests of the consumer, and the DTC is at the forefront of this endeavor.

The DiamondSure 2™ screening instrument (left) and the DiamondView™ fluorescent imager with its associated PC (right).

The DTC's Gem Defensive Programme is a collaborative effort involving the DTC Research Centre in Maidenhead, United Kingdom, focused on gem diamond research; the Diamond Research Laboratory (DRL) in Johannesburg, South Africa, part of De Beers Industrial Diamonds; and the DTC sales and marketing department in London. In total, the annual budget for the Gem Defensive Programme is currently about $4 million.

The DTC works to ensure consumer confidence using the twin strategies of disclosure and detection.

Disclosure is the first line of defense. Because the diamond industry is primarily based on consumer sentiment and trust, the protection of these is fundamental. The Gem Defensive team at the DTC's sales and marketing department is charged with ensuring that full disclosure is standard industry practice, by working with the diamond industry to adopt best practices principles, by ensuring that the trade is given the proper education and advice and by providing the required marketing and communication support to make this happen.

The DTC best practices principles state that consumers expect to purchase natural, untreated diamonds and that therefore the danger of nondisclosure is contrary to the interests of the consumer. The DTC is committed to full disclosure throughout the diamond distribution chain. The DTC requires all its clients, the DTC sightholders, to commit to trading only in natural untreated diamonds unless full disclosure is made at the time of sale.

DTC staff have conducted a number of gemological

workshops providing hands-on experience of synthetic and treated diamonds to gemologists and the trade (see *Diamonds.net News*, October 8, 2001, "The Diamond Detectives," by Richard Williams). DTC gave technical and financial support to the production of wall charts published by the GIA on how to identify synthetic diamonds and simulated and a video on fracture-filled diamonds.

The industry's second line of defense is to ensure that disclosure is backed up by detection. The gem defensive team at the DTC Research Centre has a number of roles. It sets the overall strategy for the Gem Defensive Programme; it fully characterizes synthetic and treated diamonds, both those produced at the DRL and material acquired from other sources based on a detailed knowledge and understanding of their properties; it develops practical identification techniques and instruments; and it holds frequent discussions and technical briefings with staff from the major gemological laboratories worldwide.

A diamond being inserted into liquid nitrogen containing a sample chamber of the prototype instrument designed for screening HPHT-treated type II diamonds.

De Beers Industrial Diamonds is a major producer of synthetic diamonds for industrial and technical applications. The function of the gem defensive team at the DRL is to adapt high-pressure, high-temperature (HPHT) industrial diamond synthesis equipment to produce large, good quality synthetic diamonds suitable for cutting into gemstones and to carry out HPHT treatment experiments on natural diamonds. Synthetic diamonds produced at the DRL were first sent to the GIA for research into identifying them in 1986 and were the subject of an article in *Gems & Gemology* in 1987. Since then, De Beers' synthetic diamonds have been made available to numerous gem-testing laboratories and gemological education courses throughout the world. It should be emphasized that De Beers' synthetic diamonds cut as gemstones and HPHT-treated diamonds are produced only for research and educational purposes in order to protect consumer interests. They are made available only on loan, never sold.

The challenge facing grading laboratories nowadays is not only to keep abreast of a rapidly changing situation, but also to find the time required to carry out the tests necessary to identify whether a stone is synthetic or has been treated. The key to dealing with this problem is the ability to screen large numbers of diamonds rapidly and to select only a few potentially synthetic or treated diamonds for detailed examination.

To this end, we are engaged in a program to develop relatively inexpensive, quick and easy-to-use screening instruments suitable for grading laboratories with high output. The first, the DiamondSure™, was developed in 1996. This instrument was specifically designed to address the issue of synthetic diamonds. More recently, the functionality has been extended with the development of the DiamondSure 2™ which, besides distinguishing synthetic diamonds, also specifically identifies type II diamonds and moissanite.

A third instrument, the DiamondView™, is a PC-based instrument that conveniently produces a fluorescent image, which easily allows synthetic diamonds to be identified.

At present, these instruments have been manufactured in small numbers and loaned to some of the leading gem-testing laboratories for evaluation purposes. However, with the increased prevalence of synthetic and treated diamonds we now intend to make them available in larger numbers.

An instrument still under development, and currently being evaluated at a major grading laboratory, is one especially designed to screen for HPHT-treated type II diamonds. This uses the latest miniature solid-state laser technology and incorporates a small chamber filled with liquid nitrogen to cool a diamond down to –196ºC. A result is produced in less than 30 seconds, compared with about half an hour for standard spectroscopic equipment.

In a time of great technological innovation, a key role of the Gem Defensive Programme is to anticipate what might become a problem in the future. If unscrupulous individuals can get around existing identification techniques, we need to know this as soon as possible so that alternative techniques can be developed. The combination of skills at Maidenhead, London and Johannesburg means that the DTC gem defensive team is uniquely qualified to carry out such cutting-edge research and education for the benefit of consumers and the diamond industry as a whole. ✦