IDENTIFICATION OF MELEE-SIZE SYNTHETIC YELLOW DIAMONDS IN JEWELRY

Hiroshi Kitawaki, Ahmadjan Abduriyim, and Makoto Okano

Melee-size yellow synthetic diamonds have recently been seen in the gem market, mostly as loose stones but sometimes set in jewelry. The material identified to date is synthesized by the high-pressure, high-temperature (HPHT) process. Randomly occurring pinpoint or flux-metal inclusions are diagnostic identification clues. However, some synthetic diamonds require advanced laboratory methods. This report describes the effectiveness of a technique new to gemology, infrared microspectroscopy, in combination with cathodoluminescence imaging for the rapid and accurate separation of melee-size natural and synthetic diamonds based on their characteristic mid-infrared absorption spectra and growth structures, respectively. With these techniques, the GAAJ-Zenhokyo Laboratory in Tokyo determined that approximately 10% of loose—and dozens of mounted—yellow melee diamonds submitted over a four-month period were synthetic.

Two techniques are known today for the laboratory growth of gem-quality diamonds: high pressure and high temperature (HPHT) and chemical vapor deposition (CVD). Synthetic diamonds grown by the HPHT process began to appear on the gem market in the 1990s. Most of the laboratory-grown diamonds seen commercially to date in the GAAJ-Zenhokyo Laboratory are yellow type Ib's in faceted sizes under 2 ct, but some are type Ia synthetics that have been HPHT treated to display a green-yellow color. We have also seen small quantities of type IIa colorless synthetic diamonds, as well as pink to red or purple synthetics that are produced by a multi-step process (HPHT plus irradiation and/or heat treatment; see, e.g., Shigley et al., 2004). Apollo Diamond Inc. recently announced the production and commercial sale of diamonds lab-created by the CVD method (Wang et al., 2003, 2007); however, such material has not been seen in the market in any quantity.

The HPHT synthesis of gem-quality diamonds has for the most part been based on the Russian BARS technique. In recent years, however, the U.S.–based Gemesis Corp. has been mass producing yellow synthetic diamonds by a process that represents an improvement on the original Russian technology (Shigley et al., 2002). Chatham Created Gems has also been selling synthetic diamonds in pink, blue, or yellow hues produced by a different HPHT method (Shigley et al., 2004; e.g., figure 1).

Given the current availability of synthetic diamonds and the fact that melee-size (especially those smaller than 0.05 ct) stones are more difficult to test, it should not come as a surprise that such small synthetic yellow diamonds have started to become mixed with natural diamonds, both in parcels of loose stones and in manufactured jewelry (figure 2). Although synthetic diamonds still represent only a very small part of the gem market, they receive a

Figure 1. The loose synthetic diamonds shown here, from Chatham Created Gems (San Francisco), illustrate some of the yellows produced by HPHT growth. Many are similar in hue and intensity to the natural yellow diamonds in this ring, which is courtesy of Alan Friedman, Beverly Hills, California. The loose synthetics weigh 0.05–0.30 ct; the Fancy Intense yellow center diamond in the ring is 1.09 ct, and the 93 yellow melee weigh a total of 1.12 ct. Photo by Robert Weldon.

great deal of attention within and outside the industry, so correct identification is of great importance. In the present article, we introduce a practical approach used routinely at the GAAJ-Zenhokyo Laboratory to identify melee-size HPHT-grown synthetic yellow diamonds (loose and in jewelry). Our laboratory also routinely checks for treatment in such stones, but that is beyond the scope of this study.

REVIEW OF IDENTIFICATION CRITERIA FOR HPHT-GROWN SYNTHETIC DIAMONDS

Today, most laboratories use a variety of indicators to identify HPHT-grown synthetic diamonds, as summarized below.

Inclusions. Most natural diamonds form in the earth's upper mantle at depths of 150–200 km. Associated minerals that may be present as inclusions in natural diamonds include pyrope, chrome diopside, enstatite, and olivine (see, e.g., Kirkley et al., 1991). Oriented needle-like inclusions may also provide proof of natural origin (Crowningshield, 1994). In contrast, the HPHT process generally uses Fe, Ni, and Co as flux solvents because carbon readily dissolves in them. These metals may become trapped in the lab-grown crystals as inclusions, in which case they provide proof of synthetic origin.

Although some minerals with a metallic luster (such as chromite) may be present in natural diamonds, their crystal forms are different from those shown by the metallic inclusions in synthetic diamonds. The latter may also contain breadcrumb-like or pinpoint inclusions that are characteristic of laboratory growth.

Anomalous Double Refraction due to Lattice Distortion. Diamond belongs to the cubic crystal system and is optically isotropic. However, most natural diamonds show anomalous double refraction (ADR) when they are viewed between crossed polarizing filters, due to lattice distortion. This ADR can be divided into two types—one that is created during the growth process deep in the mantle, and one caused by post-growth plastic deformation of the diamond. A good example of plastic deformation–caused ADR seen in type II diamonds is the so-called tatami pattern, which can distinguish natural type II diamonds from HPHT-grown synthetic type I diamonds (Lang, 1967). Although plastic deformation–caused ADR may be seen in type I natural diamonds, the fact that ADR due to distortion occurs only along internal growth sectors in HPHT-grown synthetic diamonds distinguishes them from natural diamonds (Shigley, 1986).

Ultraviolet Luminescence. Natural diamonds emit various fluorescence colors when exposed to UV radiation. Common colors are blue-white (originating from the N3 center), orange (the NV center), and yellow and green (the S1 or S3 centers and H3 center; Fritsch and Waychunas, 1994). These fluorescence

Figure 2. The GAAJ-Zenhokyo laboratory has identified yellow HPHT-grown synthetic diamond melee set with natural yellow melee in fine jewelry. In this ring, 12 melee (0.46 ct total weight) surround a 3.89 ct ruby. Only one of the 12 was natural (see arrow); all the others were synthetic. Photo by Masaaki Kobayashi.

colors and their intensities correspond to some extent to specific types or colors of diamonds (Eaton-Magaña et al., 2007).

HPHT-grown synthetic diamonds also often show characteristic reactions to UV radiation. Type IIa colorless synthetic diamonds exhibit strong yellow-white phosphorescence after exposure to short-wave UV, which is not seen in their natural counterparts. Likewise, the orange fluorescence seen in pink synthetic stones is an indication of growth in a laboratory (Shigley et al., 2004). Another characteristic feature of HPHT-grown synthetic diamonds is the uneven fluorescence in a cross-like pattern that is associated with cubic growth sector zoning. HPHT-treated synthetic stones in highly saturated yellow colors may show a strong green-yellow luminescence to long- and short-wave UV that originates from a defect related to nickel (Shigley et al., 1993).

Color Zoning. Color zoning may occur in natural diamonds due to differences in the incorporation of impurity elements (mainly nitrogen) during crystal growth. This uneven color distribution is generally observed parallel to the octahedral crystal faces. Plastic deformation after crystal growth or a vacancy disk lying on {111} planes also can produce brown or pink color zoning parallel to octahedral faces and often in intersecting patterns (Bangert et al., 2006); these features, too, may help identify natural diamonds.

HPHT-grown synthetic diamonds have a crystal morphology different from that of natural diamond. Each internal growth sector (such as those sectors underlying cubic or octahedral faces) incorporates impurity elements in different concentrations; as a result, distinct color zoning corresponding to the arrangement of growth-sector zoning is generally observed with magnification. Hence, synthetic diamonds often display color zoning with a combination cross and square pattern, which is also an important identifying feature.

Spectroscopy. Typically, synthetic diamonds cannot be identified by standard gemological testing alone, so more sophisticated spectroscopic and imaging techniques must also be employed. One key way to distinguish natural from synthetic diamonds is spectral analysis in the UV-visible region, with the presence of absorption due to the N3 center (415 nm). The N3 center is a defect caused by the aggregation of three nitrogen atoms surrounding a vacancy; it is present in most natural diamonds, but it is not produced by normal diamond synthesis processes (Welbourn et al., 1996). The DiamondSure instrument developed by the Diamond Trading Company (DTC) to distinguish natural and synthetic diamonds operates on the basis of detecting the N3 center. Another defect related to a Ni-N center may also be detected in the UV-visible spectrum of some synthetic diamonds (Lawson and Kanda, 1993).

Fourier-transform infrared (FTIR) spectroscopy can reveal diamond type, as well as the existence of defects related to hydrogen or to the B' center that are characteristic of natural diamonds (Allen, 1981; Kiflawi and Lawson 1999).

X-ray Fluorescence Analysis. XRF analysis to detect the chemical composition of inclusions can be an effective way to recognize HPHT-grown synthetic diamonds. Since this method can analyze only the surface or near surface of an object, any inclusions need to be exposed on a polished facet of the diamond. In HPHT-grown synthetic diamonds, the flux-metal inclusions often reach the polished surfaces. In such cases, the presence of Fe, Ni, or Co can reveal the synthetic origin of the stone.

Cathodoluminescence (CL) Imaging. Sophisticated luminescence techniques can be very effective for the identification of natural or synthetic diamonds, because they clearly reveal the very different growth structures described above. One commonly used

instrument, the DTC DiamondView, employs strong deep-UV radiation to capture luminescence images of a diamond (Welbourn et al., 1996). Similarly, CL imaging employs an electron beam to create images of the gem's growth structure. While the DiamondView is easier to operate, luminescence images obtained by electron beam in the CL technique are sharper. Also, the lower limit for the DiamondView is about 0.05 ct (see, e.g., Welbourn et al., 1996), but—coupled with an optical microscope—the CL instrument can be used to test diamonds that are smaller than this.

Although CL images of natural diamonds ideally will show a rectangular growth structure that is caused by an octahedral {111} sector, variations in impurity concentrations and growth conditions typically lead to significant differences between stones in terms of observed growth pattern, interval of zoning, fluorescence, and intensity of CL. Some diamonds show very complicated images due to dissolution and plastic deformation processes. These images are unique for each diamond and possibly may be used to identify specific diamonds (Sunagawa et al., 1998; Miyata et al., 1999).

The CL images of synthetic diamonds are more predictable. They clearly show sector zoning, and an experienced operator will be able to visualize the morphology of the original crystal from this image.

Photoluminescence (PL) Analysis. This spectral technique can detect some point defects when excited by lasers in the UV or visible-light range. Since 2000, this has become an important method for detecting HPHT treatment at gemological laboratories (Chalain et al., 2000). In natural diamonds, PL analysis reveals spectral peaks that originate due to various point defects, nitrogen-containing defects, and defects for which the cause has not yet been identified. Conversely, in HPHT-grown synthetic diamonds, PL analysis can reveal peaks that are related to metal solvents such as nickel.

MATERIALS AND METHODS
The samples used in this study included 870 loose yellow melee (see, e.g., figure 3) and 500 yellow melee in 30 items of jewelry submitted to the GAAJ-Zenhokyo Laboratory by a number of clients for identification reports. These yellow melee were submitted over a four-month period from the end of 2006 into 2007. For the purposes of this study, we defined *melee* as diamonds smaller than 0.20 ct.
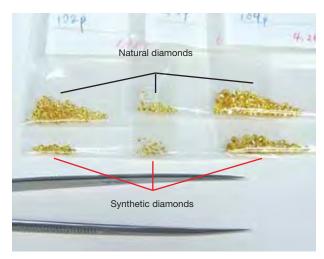

Figure 3. The study examined 870 loose yellow melee. All the samples shown here were originally submitted as a single parcel; testing revealed that a significant portion of them (the three bags in the lower row) were HPHT-grown synthetic diamonds. These samples ranged from 0.001 to 0.10 ct. Photo by H. Kitawaki.

Twenty of the 30 jewelry items were set with 3–12 yellow melee, and the remaining 10 items were set with 35 to as many as 66 stones. For the most part, the samples ranged between 0.01 and 0.15 ct, but the smallest weighed 0.001 ct (again, see figure 3). The jewelry items consisted of rings and pendants (figure 4).

We studied all the loose and jewelry-set samples with standard gemological instrumentation. We used an Olympus gemological microscope to examine inclusions, with magnification up to 60× and a narrow fiber-optic light source (diameter of 1–3 mm) for illumination. UV fluorescence was observed in a dark room using long- and short-wave UV lamps manufactured by Manaslu Co.

Infrared Spectroscopy. All the samples were tested by IR spectroscopy. The 870 loose stones were analyzed by a Shimadzu IR Prestige-21 instrument equipped with a KBr beam splitter and DLATGS detector. This instrument can analyze extremely small loose diamonds and requires less time than the micro-FTIR technique used for analyzing mounted stones. The analytical range was 4000–400 cm^{-1}, resolution 4 cm^{-1}, and total scans per spectrum 20. When the signal-to-noise ratio was not adequate, the measurement was made with 1.0 cm^{-1} resolution and 200–500 scans per spectrum. The monitor mode in 4 cm^{-1} resolution allows faster measurements. Although the data cannot be saved

Figure 4. The yellow melee in several items of jewelry were tested as part of this study. Although most of the stones were natural diamonds, HPHT-grown synthetic diamonds (indicated here by arrows) were identified in approximately half the jewelry items tested. Photos by H. Kitawaki.

to a computer in this mode, a measurement can be done in about 10 seconds (including positioning the sample on the microscope stage and data scanning).

The samples set in jewelry were measured by FTIR microspectroscopy (micro-FTIR). This kind of IR measurement under a microscope has emerged

relatively recently compared to the better-known Raman microspectroscopy (e.g., Macmillan and Hofmeister, 1988). It has been used in various fields; in geology, micro-FTIR has been applied to minerals in thin section to analyze hydrous microphases in areas as small as 10×10 μm, to understand the

Figure 5. A Shimadzu IR Prestige-21 FTIR spectrometer in conjunction with a Shimadzu AIM-8800 infrared microscope (left) was used to analyze each of the melee-size samples set in jewelry. By confirming the analytical site on the monitor, this configuration allows IR spectral analysis of a small area on a small sample (right). Photos by H. Kitawaki.

Figure 6. Many of the natural samples revealed the oriented needle-like (left) and highly reflective flake-like (right) inclusions often seen in yellow type Ib natural diamonds. Photomicrographs by H. Kitawaki; magnified 65×.

mechanism of water-rock interactions (Nakashima et al., 1989). However, we do not know of any prior report of its application to gemology. In the present study, we used the Shimadzu IR Prestige-21 spectrometer in conjunction with a Shimadzu AIM-8800 infrared microscope (figure 5).

Again, the measurement range was 4000–400 cm⁻¹, but a resolution between 0.5 and 16 cm⁻¹ can be chosen. For routine work, we set the resolution at 4 cm⁻¹ with a total of 20 scans per measurement. The IR microscope uses liquid nitrogen to cool the detector, but our sample measurement was done at room temperature. The instrument employs X15 Cassegrain objective/condenser mirrors, and its actual measurement range is 3–400 µm. We set the following positioning range for the sample stage, which can be adjusted automatically: X-axis—70 mm, Y-axis—30 mm, and Z-axis—40 mm. The operator sets the sample on the stage and then, viewing it on a monitor, designates the measurement location; the sample stage automatically moves to the desired spot for the analysis. With this arrangement, we were able to measure each sample set in jewelry individually.

Cathodoluminescence Imaging. We used CL imaging to test a number of stones in 15 items of jewelry and 250 loose samples that either were suspected of being synthetic or did not display positive proof of natural
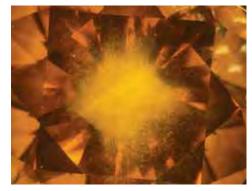
origin with standard gemological tests and micro-IR analysis. Specifically, we used a Premier American Technologies Corp. ELM-3R cathodo-luminescope, with a vacuum chamber 125 mm deep, 170 mm wide, and 60 mm high. This was large enough to easily hold the jewelry pieces in our sample base, although items with a height over 30 mm may block the electron beam. We were able to test nine loose melee (face up) at one time. The vacuum was 20–40 millitorr, and observations were made under the conditions of 4–6 kV and 0.5 mA at room temperature.

RESULTS

Gemological Observations. Microscopic examination revealed inclusions in all but a few of the samples. About 20% showed the oriented needle-like inclusions with a shiny black luster and the highly reflective flake-like inclusions that are characteristic of natural diamonds (Crowningshield, 1994; figure 6). We could not conclusively identify these inclusions, but we believe they were graphite or a sulfide mineral. The clouds of minute inclusions seen in more than half the samples examined also indicated natural origin. These clusters of white dots occasionally had a wispy appearance (figure 7).

However, we also saw features indicating HPHT synthetic origin. Approximately 85 loose samples

Figure 7. Natural diamonds commonly show minute inclusions in a cloud-like formation (left) that in some stones have a wispy appearance (right). Photomicrographs by H. Kitawaki; magnified 35×.
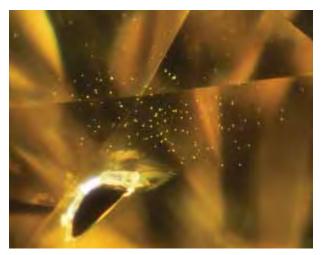
Figure 8. These scattered white pinpoint inclusions indicate that this melee sample is a synthetic diamond. Photomicrograph by H. Kitawaki; magnified 65×.

and several individual pieces of melee in 15 items of jewelry showed pinpoint-like inclusions. In contrast to the clouds of minute inclusions observed in the natural diamonds, these appeared as individual rounded shapes that were scattered irregularly throughout the stones (figure 8). Approximately 10–20% of the samples with pinpoint inclusions also contained metallic inclusions (figure 9), which are obvious indications of HPHT synthesis. A similar proportion of samples with pinpoint inclusions also contained breadcrumb-like inclusions (figure 10). Both the metallic and breadcrumb-like inclusions are typically a solid phase of the flux-metal solvent in which the diamond grew, which was trapped during crystal growth.

The vast majority (over 80%) of the samples showed weak fluorescence to long-wave UV radiation in slightly chalky yellow to orange colors, with a weaker reaction to short-wave UV. In general, these were the same stones that had inclusions of presumably natural origin. The remaining samples were almost or completely inert to both wavelengths. However, this reaction was seen in samples with needle-like inclusions that indicated natural origin as well as in those containing scattered pinpoint-like or metallic inclusions that indicated synthetic origin.

Infrared Spectroscopy. Most of the samples (~70%) were type Ia diamond, with a high nitrogen content; absorptions from the B center (a broad band with peaks at 1332, 1175, 1010, and 775 cm^{-1}) and platelets (1365 cm^{-1}) were detected. Many also showed hydrogen-related absorptions at 1405 cm^{-1} (Davies et al., 1984) or several H-related features in the 3500–3100 cm^{-1} region (figure 11). In addition, a peak at 1430 cm^{-1} is due to C-N absorption. The diamonds showing such spectroscopic features could be identified as natural in origin.

Eighty-five loose samples and about 50 samples in the 15 items of jewelry with synthetic-appearing inclusions showed absorptions only from the C center (i.e., single substitutional nitrogen, 1344 and 1130 cm^{-1}; again, see figure 11). Perfect type Ib stones showing only the C center are very rare among natural diamonds, so IR spectroscopy supported the identification of these samples as synthetic.

Among all the yellow melee tested, 170 loose stones and 100 samples in 15 items of jewelry showed a weak A center (a broad band with peaks at 1282, 1215, 1096, and 472 cm^{-1}) together with the C center. For the most part, these corresponded to stones containing oriented needle-like inclusions or cloud-like inclusions. Natural type Ib yellow diamonds showing the A center are occasionally seen. However, synthetic diamonds produced at extremely high temperatures, or HPHT-treated yellow
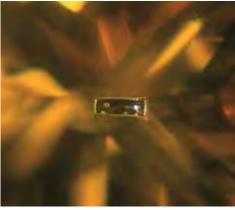

Figure 9. Also characteristic of HPHT synthetic diamonds are inclusions of the solidified metallic flux, which were irregular in some of the melee (left) and well formed in others (right). Photomicrographs by H. Kitawaki; magnified 65×.
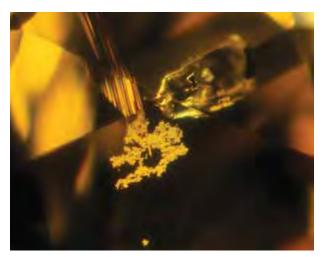
Figure 10. Breadcrumb-like inclusions of metallic flux are clear indicators that this sample is synthetic. Photomicrograph by H. Kitawaki; magnified 65×.

synthetic diamonds, may show similar infrared spectra. Therefore, the combined presence of a weak A center with a C center is not diagnostic for natural or synthetic origin.

Cathodoluminescence Observations. Among the diamonds in the jewelry items, those that were positively identified as natural by IR spectroscopy often showed slightly cloudy orange-yellow to yellow luminescence and a square zonal structure surround-

ed by octahedral {111} growth horizons, both of which are characteristic of natural diamonds (figure 12). However, some stones showed irregular patterns or an indistinct CL image. For example, a complicated structure may indicate a natural origin, but it is difficult to interpret the growth history (figure 13, left). An indistinct CL image will not help identify the natural or synthetic origin (figure 13, right).

For the most part, samples that showed a weak A center together with a C center in their FTIR spectra also showed linear CL patterns (slip bands; figure 14). This linear pattern with green-to-yellow luminescence originates from the H3 center (Welbourn et al., 1996); each of the fluorescing lines was parallel to octahedral faces and intersected one another. Such linear patterns in yellow diamonds are associated with plastic deformation and thus characterize the stone as natural.

The samples that showed only the C center in FTIR analysis displayed growth zoning characteristic of synthetic diamonds (figure 15). These images were composed of cubic {100} and octahedral growth sectors, among others. The most typical CL image for these samples was a combination of green-yellow cubic sectors, and inert octahedral sectors (again, see figure 15). Adding to this combination, minor sectors that glowed blue-white, such as {110} or {113}, were often recognized (figure 16).
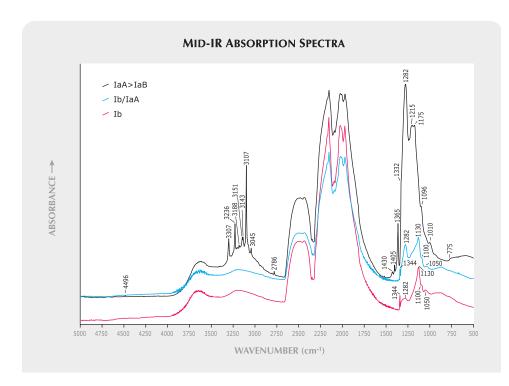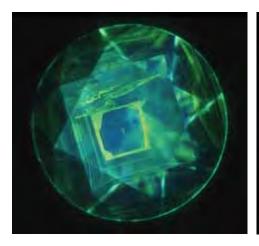

Figure 11. These mid-IR spectra of jewelry-set melee reveal three types of natural and synthetic diamond. The black spectrum is of a natural diamond with a dominant IaA component at 1282, 1215, and 1096 cm⁻¹; a minor IaB component with a sharp peak at 1365 cm⁻¹; and a peak at 1430 cm⁻¹ related to C-N absorption. Note also the H-related absorptions at 4496 cm⁻¹, in the 3307–2786 cm⁻¹ region, and at 1405 cm⁻¹. The blue spectrum shows a natural diamond that is a mixture of type Ib with IaA. The red spectrum is of a pure type Ib synthetic diamond, characterized by bands at 1344, 1130, and (weaker) ~1282, 1100, and 1050 cm⁻¹.
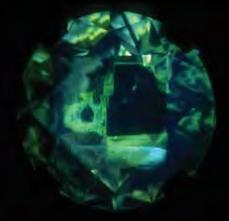
Figure 12. These CL images show the square zonal structure surrounded by octahedral {111} growth horizons characteristic of natural diamonds, as well as changes they undergo during growth. The mantle of the 0.15 ct diamond on the left has a rounded, irregular pattern, suggesting dissolution during crystallization. The ring-set 0.10 ct diamond on the right shows zigzag growth due to small stepped {111} faces. Photos by H. Kitawaki.

Also, though in small quantity, some stones showed orange-yellow luminescence in octahedral sectors and were inert in cubic sectors (figure 17).

The luminescence color seen with a CL image is usually the same as that observed with UV radiation, although it is typically more intense with CL. However, the CL image and fluorescence to UV radiation sometimes may differ due to the lower penetration depth of CL (1–2 µm; Ponahlo, 1992) in comparison with UV radiation (such as that from the DiamondView; Simon Lawson, pers. comm., 2008).

DISCUSSION

At the GAAJ-Zenhokyo Laboratory, yellow synthetic diamonds in melee sizes have been documented since 2005, as loose stones and set in jewelry. The identification of melee-size synthetic diamonds also was reported by Simic and Burnett (2007), but for the most part their methods could not be applied to mounted stones.

Today, it is very unusual for us to find synthetics among all the diamonds identified at our laboratory, and it is unlikely that synthetic diamonds have flooded the gem market. As described above, however, the results of this study suggest that almost 10% of 870 loose yellow melee diamonds that came in over this four-month period were synthetic, and 15 out of the 30 jewelry items studied each contained on average about 10% HPHT-grown diamonds. The diamonds confirmed as HPHT synthetic at our laboratory were reported individually to the clients. For the jewelry items, the clients elected to replace the synthetic stones with natural diamonds after learning our testing results.

Identification of melee-sized yellow diamonds at the GAAJ-Zenhokyo Laboratory is performed in three steps:
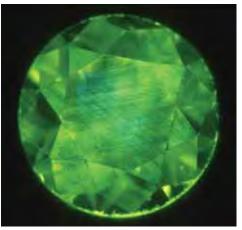
1. All diamonds are examined with a gemological microscope and their inclusions studied.

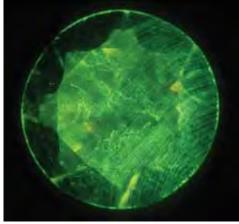2. All diamonds undergo micro-FTIR spectroscopic analysis.

Figure 13. Irregular patterns or unclear CL images were observed in some of the samples. Zoning parallel to an octahedral face at the center of the stone may indicate a natural origin (left). However, an indistinct CL image, such as that seen on the right, is not helpful in establishing a natural or synthetic origin. Photomicrographs by Hideaki Fukushima; magnified 20×.

Figure 14. The linear slip bands parallel to the {111} plane in these two CL images of yellow melee are associated with plastic deformation, which identifies the diamonds as natural. The yellowish green color of the growth bands is due to the H3 center. Photomicrographs by A. Abduriyim; magnified 20×.

3. Diamonds for which the origin is still in question are examined by CL.

In the first step, we look for characteristic inclusions, such as the oriented black needle-like inclusions and cloud-like clusters of minute inclusions that are typical of natural diamonds, or the irregularly distributed pinpoint-like inclusions that are seen in synthetics. The presence of metallic inclusions is proof of synthetic origin.
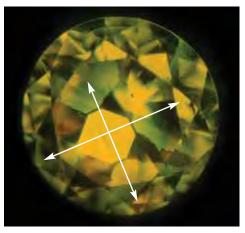
In the second step, the use of an infrared microscope makes it possible to analyze several loose melee-size diamonds at one time, or those set in jewelry that could not be measured by normal FTIR analysis. The samples are observed on a computer monitor, and each can be identified as to its diamond type individually in a matter of seconds. Those stones that show the B center or platelets, or that show absorption related to hydrogen, are identified as natural. Those that contain only the C center (i.e., single substitutional nitrogen) are likely to be synthetic.
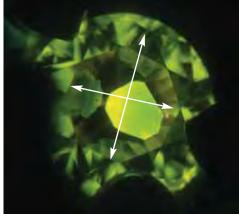
In the third step, the CL images of natural diamonds are differentiated by rectangular banding that is parallel to octahedral sectors, or slip bands parallel to octahedral sectors that intersect one another. The former pattern was observed in the type Ia stones, and the latter in type Ib. In contrast, HPHT-grown diamonds show sector zoning that is mainly composed of a combination of cubic and octahedral sectors.
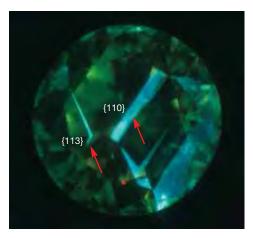
In many of the CL images of synthetic diamonds observed in this study, the center of the original crystal was located on or near the center of the table of the faceted sample. This means that these melee were not cut from remnants of larger pieces, but rather were cut from small as-grown crystals (that is, almost as small as the melee).

Using the procedures described, we can identify most natural and synthetic melee diamonds relatively quickly, a matter of a few minutes for loose samples. Examination of jewelry items with micro-FTIR may require more time to position and focus the individual stones, but nevertheless an item set with 10 melee can be analyzed in about 30 minutes.

Figure 15. CL imaging identified these yellow melee (left, loose; right, set in a pendant) as synthetic diamonds. Cross-shaped growth patterns were seen in most synthetics, from the presence of cubic (yellowish green) and octahedral (inert) sectors. These zonal structures are distinctly different from those seen in natural diamond. Photomicrographs by H. Kitawaki; magnified 20× (left) and 18× (right).
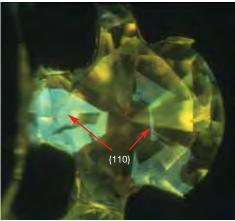
Figure 16. CL imaging revealed blue-white dodecahedral {110} and/or trapezohedral {113} sectors in some of the melee synthetic diamonds (left). The three-fold CL pattern displayed by the other synthetic diamond is related to its cubic growth sectors; the dodecahedral {110} sector luminesced a weak blue-white (right). Photomicrographs by H. Kitawaki; magnified 20× (left) and 18× (right).

Figure 17. A characteristic cross-shaped pattern is displayed in this CL image of a loose synthetic diamond. The orange-yellow and inert regions are related to octahedral and cubic growth sectors, respectively. Photomicrograph by H. Kitawaki; magnified 20×.

Additional time may be needed to determine if a natural diamond has been treated, but that is beyond the scope of this article.

CONCLUSION

Synthetic diamonds in various colors such as yellow, blue, and pink have been circulated in the gem market although in relatively small quantities. Those identified as synthetic to date in our laboratory were produced by the HPHT method, and they were identified by standard gem testing methods combined with more advanced spectroscopic and cathodoluminescence imaging techniques.

As more melee-size yellow synthetic diamonds are set in jewelry together with natural stones, their identification poses a new challenge for gemological labs. The three-step approach described in this study—examination with a microscope, micro-FTIR spectroscopy (to obtain the spectra of individual melee mounted in jewelry), and CL imaging—is effective in conclusively separating natural from synthetic diamonds.

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