GEMESIS LABORATORY-CREATED DIAMONDS

By James E. Shigley, Reza Abbaschian, and Carter Clarke

High-quality yellow, orange-yellow, and yellow-orange laboratory-created type Ib diamond crystals up to 3.5 ct are being produced commercially by the Gemesis Corp. of Sarasota, Florida. In some samples, color zoning (yellow and narrower colorless zones) and a weak UV fluorescence pattern (a small green cross-shaped zone combined with an overall weak orange luminescence) provide means of identification; when present, metallic inclusions also indicate laboratory growth. Some samples lack these diagnostic visual features, but all of these Gemesis synthetic diamonds can be identified by advanced instrumentation such as the De Beers DiamondView luminescence imaging system and EDXRF chemical analysis.

The possibility of high-quality synthetic diamonds being produced for jewelry purposes, and the potential for their misidentification, have concerned members of the jewelry trade since General Electric produced the first synthetic diamonds in 1954 (Strong and Wentorf, 1991). Such apprehensions have overshadowed the fact that only a very small number of gem-quality synthetic diamonds have entered the marketplace (approximately 1,000 carats annually). In spite of their limited availability, synthetic diamonds have been studied extensively, and the means to distinguish them from natural diamonds have been widely publicized (see, e.g., Crowningshield, 1971; Koivula and Fryer, 1984; Shigley et al., 1993a,b, 1995). To date, large-scale commercial production of synthetic diamonds for jewelry use has not been fully achieved.

This situation is beginning to change with the expansion of production capacity for high-quality yellow laboratory-grown diamonds by the Gemesis Corp. (figure 1). Using "BARS" diamond-growth equipment and expert Russian technicians, and with technical assistance from scientists and engineers at the University of Florida, this company redesigned the growth apparatus, commercialized the production process, and established a pilot plant in Gainesville, Florida. To accommodate expansion plans, the company has constructed a new 30,000 square foot (2,788 m^2) production facility in Sarasota, Florida. Over the next few years, this facility could be expanded to include more than 300 "BARS" units.

This article describes efforts by Gemesis to grow commercial quantities of 2.5–3.5 ct yellow synthetic diamond crystals with consistent color, quality, and yield (see also Attrino, 1999; Lerner, 2002). The company's goal is to achieve a production level at which 90% of their crystals are suitable for manufacturing as polished gems of 1 ct or larger. At present, there are no plans to produce crystals that would cut smaller or melee-size (<0.20 ct) material. Marketing efforts have initially focused on selected jewelry manufacturers and retailers, and the company advocates complete disclosure to jewelers and consumers.

Although development of equipment that will grow even larger sizes is under consideration, as are efforts to grow colorless and blue synthetic diamonds on a commercial scale, this article will focus on the yellow (including orange-yellow and yellow-orange) crystals now being grown by Gemesis, and

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See end of article for About the Authors and Acknowledgments.

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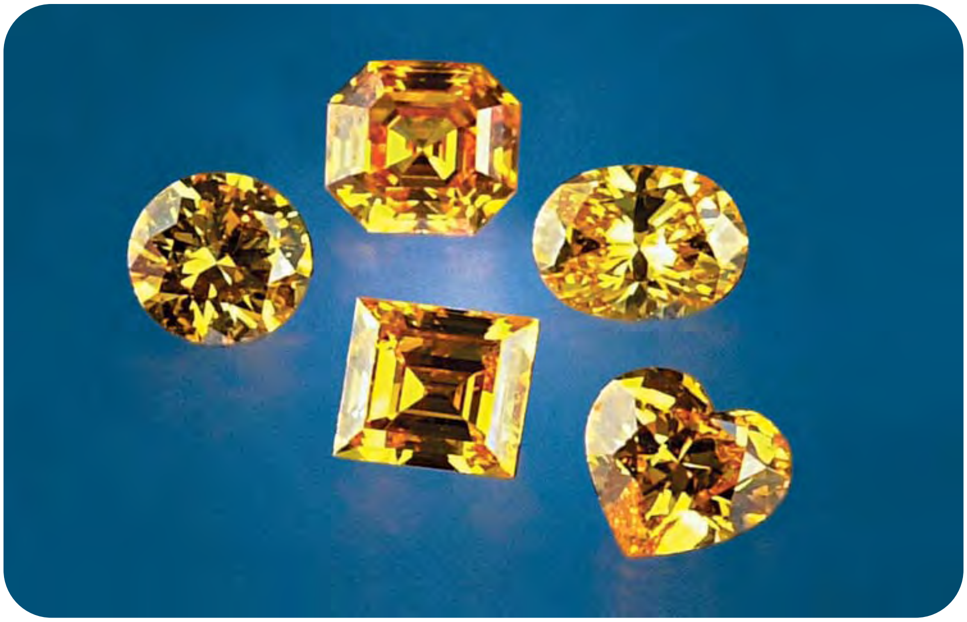


Figure 1. These five laboratory-created diamonds (0.80 to 1.25 ct) are representative of the material now being produced commercially by the Gemesis Corp. Photo by Elizabeth Schrader.

the means by which they can be identified with standard gemological techniques and, if necessary, advanced instrumentation.

"BARS" GROWTH OF SYNTHETIC DIAMONDS

Gemesis technicians use a redesigned and re-engineered split-sphere ("BARS") apparatus, as illustrated in figure 2 (for information on this equipment, see Palyanov et al., 1990, 1997, 1998; and Shigley et al., 1993b). With this type of apparatus, pressure is applied to the growth chamber (which measures approximately 2.5 cm on a side) by a series of anvils machined from carbide steel and composites. An inner set of six anvils, positioned perpendicular to the sides of a cube, surrounds the growth chamber. An outer set of eight anvils, positioned perpendicular to the sides of an octahedron, surrounds the inner set. This entire multi-anvil arrangement is housed within two hemispherical steel castings (which are hinged to allow access to the anvils and growth chamber; thus the name "split sphere"). Two large steel clamps keep these castings together. Pressure is applied hydraulically against the curved outer surfaces of the eight anvils. A graphite element is used to heat the chamber.

Typical diamond growth conditions with this equipment are pressures of 5.0 to 6.5 Gigapascals (GPa, equal to 50 to 65 kilobars) and temperatures of 1,350° to 1,800°C. Various transition metals (such as iron, nickel, and cobalt) provide a solvent and catalyst medium. Growth takes place on a tiny seed crystal, which can be either natural or synthetic diamond. The orientation of the seed determines the shape and geometry of the grown crystal. There is a small but important difference in temperature between the top of the growth chamber (the "hotter" end, where the carbon source material is located) and the bottom (the "cooler" end, where the seed crystal is located). This difference provides the driving force for diamond crystallization (hence, the growth method is called the "temperature gradient" technique; see Strong and Chrenko, 1971; Strong and Wentorf, 1971; Wentorf, 1971). Powdered graphite dissolves in the molten metal solvent in the hotter portion of the chamber; the carbon atoms move through the solvent under the influence of the temperature gradient and crystallize as a single diamond crystal on the seed in the "cooler" part of the chamber. Powdered diamond also can be used as a carbon source material.

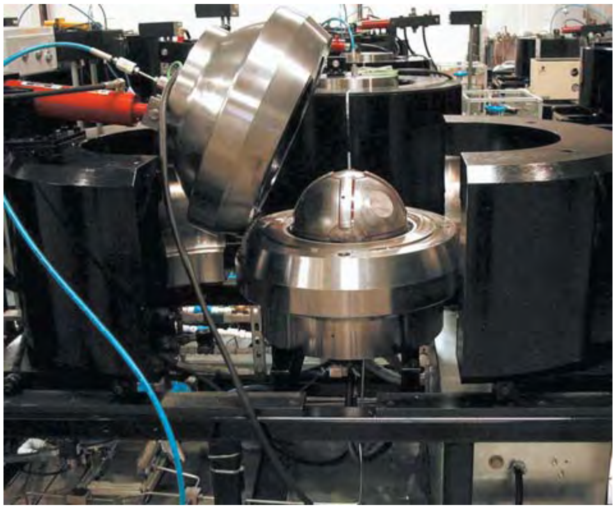
PRODUCTION AT GEMESIS

Currently, Gemesis has 23 "BARS" units in operation; an additional four units are dedicated for research purposes at the University of Florida. Although using the same fundamental design as the original Russian-made units, Gemesis scientists and engineers have modified the growth equipment in several important ways to comply with American engineering standards and improve its functioning. The redesigned equipment has greater longevity and is more easily manufactured, operated, and maintained than the original units. The equipment also is relatively safe to operate, with little possibility that catastrophic failure of the pressure vessel could cause danger to operating personnel. The redesign recognizes that one key to the commercial growth of gem-quality synthetic diamonds, in addition to the purity, consistency, and geometries of the starting materials, is careful computer monitoring of both temperature and pressure conditions to insure that steady growth conditions are maintained. Another innovation is a new mechanism to open and close the hemispherical castings, permitting easier sample loading and unloading. Eight patent disclosures have been filed with the University of Florida's Office of Technology Licensing, and have been transferred to Gemesis. Gemesis currently protects its intellectual property through trade secrets and selective disclosures, and it may seek patent protection on several proprietary aspects of its technology.

With this modified equipment, it takes approximately 80 hours to produce a single synthetic diamond crystal up to 3.5 ct. The depth of yellow color, as well as the shape, symmetry, and clarity of the crystals, can be controlled to some extent. Some multiple crystals have been grown experimentally in a single chamber during a 36-hour growth cycle, although the volume of the chamber means that these crystals must be smaller (about 0.6 ct each for four crystals in the chamber, and about 0.35 ct each for eight crystals).

Gemesis also has grown a limited number of blue and colorless synthetic diamond crystals, but it has not finalized plans for commercial production of this material. Also under consideration is the design of equipment with a larger growth chamber in order to produce crystals up to 15 ct. Since 2000, approximately 4,000 carats have been grown, with the present capacity being around 550 carats per month.

Figure 2. This modified "BARS" growth apparatus, shown here in a partly opened configuration, is of the type used by Gemesis to grow synthetic diamonds. Photo by Tom Moses.



MARKETING PLANS

In 2002, the company sold a limited quantity of both rough and polished yellow synthetic diamonds to a small number of manufacturers and retailers for use in jewelry. Future sales will be directed toward additional selected manufacturers and retailers. Prices vary depending on cutting style, color, and appearance. An external laser inscription and an internal mark are being considered as practical means of clearly disclosing the laboratory-grown identity of the material. In addition, each crystal will have a certificate of origin issued by an independent laboratory. At present, Gemesis has no plans to manufacture jewelry or use their growth equipment for the high-pressure/high-temperature treatment of their product or other diamonds to modify their color. Again, the company goal is to produce high-clarity laboratory-grown diamonds in several categories of yellow color in a consistent size and quantity that could be "made to order" for jewelry applications.

With commercial production now occurring at the expanded facility in Sarasota, and based on the success of initial sales, the company plans to install an additional 16 growth units per month beginning in mid-2003. This would result in approximately 300 units within two years in the present production facility, which has been designed to facilitate expansion to twice its current space if warranted. The company's close relationship with scientists at the University of Florida is an important part of its business plan. The current research emphasis is on optimizing growth conditions for blue diamonds and, eventually, colorless diamonds.

MATERIALS AND METHODS

We examined a total of 30 Gemesis laboratory-grown diamonds (six yellow, 11 orange-yellow, and 13 yellow-orange) that are representative of the product now being sold. These included five orange-yellow and yellow-orange crystals (1.81–2.47 ct) and 25 faceted samples (0.68–1.34 ct). Standard gemological properties were measured for these samples, and internal features were observed with a gemological microscope. Reactions to ultraviolet radiation were viewed in a darkened room with four-watt long-

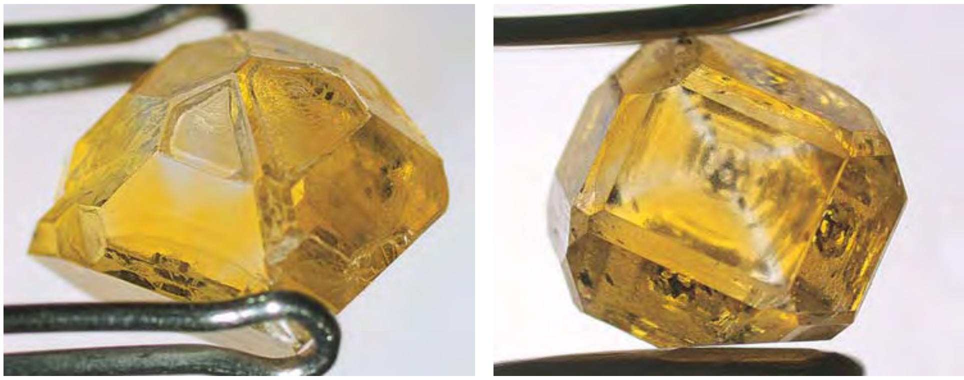


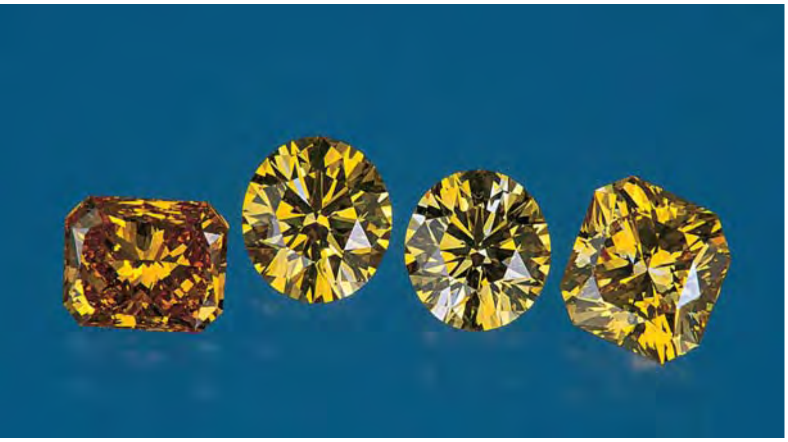
Figure 3. The 2.17 ct crystal on the left shows the cuboctahedral form typical of most synthetic diamonds grown by the temperature gradient technique. On the right, the four-fold arrangement of colorless growth sectors (and several dark metallic inclusions) can be seen in this 1.81 ct crystal. Photomicrographs by James Shigley; magnified 15× (left) and 25× (right).

wave (366 nm) and short-wave (254 nm) Ultraviolet Products lamps. Absorption spectra were observed with a desk-model Beck prism spectroscope.

Faceted samples of each of the three colors were selected for testing with advanced instrumentation. A Thermo Spectronic Unicam UV500 spectrophotometer was used to record absorption spectra of 16 samples held in a cryogenic cell cooled by liquid nitrogen over the range of 250–850 nm with a resolution of 0.1 nm. Absorption spectra in the mid-infrared range (6000–400 cm^{-1}) and the near-infrared range (10,500–4000 cm^{-1}) were recorded for 15 samples at room temperature with a Nexus 670 Fourier-transform infrared (FTIR) spectrometer. In the mid-infrared range, the resolution was set to 1.0 cm^{-1}. In the near-infrared, the resolution was 4.0 cm^{-1}.

Photoluminescence spectra were recorded for 13 samples with a Renishaw 1000 Raman microspectrometer over a range of 520–870 nm using a 514.5 nm Argon laser operating at an initial power of 20 mW. Five summed scans were accumulated for a better signal-to-noise ratio; the samples were held in a cryogenic cell cooled by liquid nitrogen.

Figure 4. The Gemesis synthetic diamonds are faceted to maximize color through the crown facets, minimize the visibility of any small metallic inclusions, and retain maximum weight. These four faceted samples (0.85 to 1.34 ct) represent the range of colors produced. Photo by Maha Tannous.



Qualitative chemical analyses of 19 rough and cut samples were obtained by energy-dispersive X-ray fluorescence (EDXRF) with either of two instruments. One was a Thermo-Noran Omicron system operating at accelerating voltages of 25 and 35 kV, with beam currents between 0.06 and 0.08 mA. The other was a Kevex Spectrace QuanX system operating at an accelerating voltage of 20 kV and a beam current of 0.30 mA. (The two sets of conditions used for the Omicron system were chosen to broaden the range of elements that could be detected.)

The cathodoluminescence reaction of one faceted yellow sample was observed with a Luminoscope ELM-3R cathodoluminescence (CL) unit operating at 10–15 kV and 0.5–1.0 mA. Similar reactions for 12 other faceted samples were observed with the De Beers DiamondView™ deep-UV luminescence imaging system (Welbourn et al., 1996).

RESULTS AND DISCUSSION

Visual Appearance. The five crystals exhibited the cuboctahedral forms typical of synthetic diamonds grown by the temperature gradient technique. These crystals were nearly equant in dimension (figure 3), or were slightly distorted in shape (i.e., unequally developed or missing crystal faces, irregular surfaces, etc.) as a result of minor changes in conditions during a growth run. The 25 faceted samples varied from yellow-orange through orange-yellow to yellow; they were high in saturation and moderate in tone (corresponding to "Fancy Vivid" in the terminology used for natural-color diamonds; see figures 1 and 4).

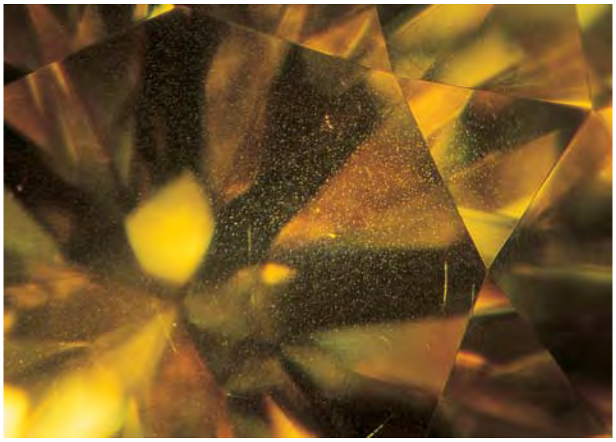
Clarity. In general, the faceted samples were of good

Figure 5. Small metallic inclusions were observed in 10 of the 30 Gemesis laboratory-grown diamonds, three of which are illustrated here. Note in the center image that a small area also shows part of the original crystal surface. The metallic inclusion on the right, seen in direct view and by reflection in two facets, has an equant shape with flat surfaces. Photomicrographs by James Shigley; magnified 75×.

clarity and relatively free of inclusions in comparison to other synthetic diamonds that we have examined. Equivalent clarity grades of the test samples would be in the VS to SI range. Ten of the faceted samples did exhibit small, opaque metallic inclusions visible with 10× magnification (figure 5), and the majority of the samples exhibited a cloud-like arrangement of very tiny, dispersed pinpoint inclusions (figure 6). A few samples displayed remnants of the original crystal surface that was not removed during polishing (figure 5, center).

Other Features Seen with Magnification. Although it is not readily apparent in all samples, the most common feature seen with magnification was color zoning. When most of the polished samples were examined through the pavilion facets, larger yellow zones were seen to be separated by narrower colorless zones (figure 7). On occasion, this color zoning could even be seen through the crown facets. In two of the faceted samples, no colorless zones were evident. During manufacturing, efforts are made to reduce the visibility of these colorless zones through the crown facets by the choice of cut shape and facet arrangement. It is also possible to minimize this zoning by modifying the growth conditions.

Figure 6. Most of the faceted Gemesis synthetic diamonds displayed tiny pinpoint inclusions in a dispersed, cloud-like pattern that differs from the arrangement of pinpoint inclusions seen in natural type Ib yellow diamonds. Photomicrograph by Shane McClure; magnified 20×



No seed crystal was evident in any of the crystals; only a remnant of the imprint of the seed was visible on their bases. Typically the seed breaks away when the crystal is removed from the growth chamber.

Luminescence. In general, the samples displayed weaker long- or short-wave UV fluorescence than other synthetic diamonds GIA has examined; 10 were inert. In those that did fluoresce, the reaction was weak or very weak orange to both UV wavelengths. No difference in fluorescence intensity was noted between long- and short-wave UV in 11 of the samples that did fluoresce; in the remaining nine samples, the short-wave reaction was either slightly weaker or slightly stronger in intensity as compared to long-wave. In some samples (10 in long-wave and 15 in short-wave UV), a small green cross-shaped pattern was superimposed on the weak orange luminescence (figure 8). In two of the crystals, this cross-shaped pattern appeared near the center of the base where the seed had been located. Similar but more intense cross-shaped luminescence patterns could be seen in all of the samples examined by the Luminoscope CL unit

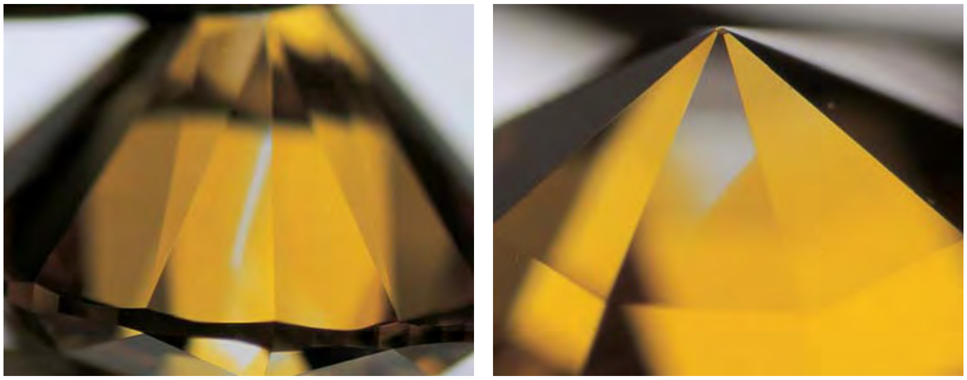
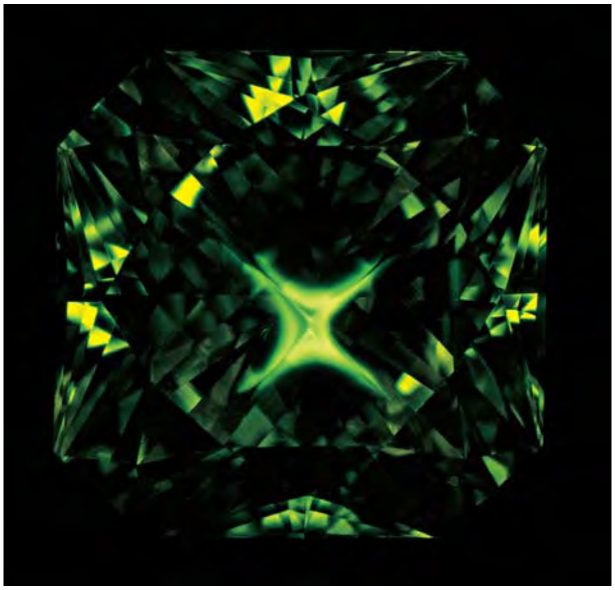


Figure 7. The narrow colorless zones in these two polished yellow Gemesis synthetic diamonds (0.80 and 0.88 ct) are diagnostic of their growth in a laboratory. Photomicrographs by James Shigley; magnified 25× (left) and 75× (right).

(figure 9) and the De Beers DiamondView (e.g., figure 10). None of the samples exhibited any phosphorescence to standard UV lamps, or any luminescence when they were illuminated with a strong light source from the desk-model spectroscope.

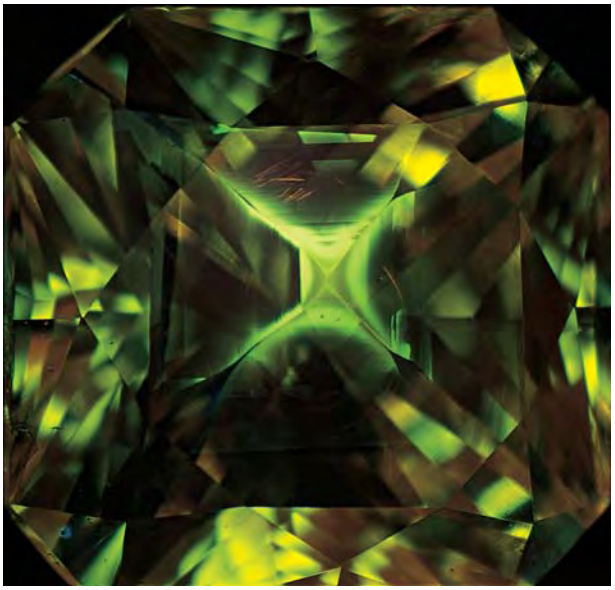
Visible Spectra. Using a prism spectroscope and transmitted light, we could not see any sharp absorption bands in the spectra of any of the 30 synthetic diamonds tested. There was, however, a gradual increase in absorption toward the blue end of the spectrum.

Figure 8. This 0.93 ct yellow Gemesis sample exhibits green luminescence in a cross-shaped pattern when exposed to long-wave UV radiation. Photography of this luminescence required an extended exposure time of several minutes due to its weak intensity. This fluorescence pattern is characteristic of many synthetic diamonds and has never been observed in natural stones. Photo by Shane Elen.



Information Obtained from Advanced Instrumentation. Based on their infrared spectra, all of the synthetic diamonds were type Ib (several exhibited a weak feature at 1284 cm^{-1} due to the aggregated "A" form of nitrogen [a N-N pair]; see Wilks and Wilks, 1994, pp. 68–70). There were no significant differences in the visible spectra of the samples tested that could be directly correlated to the small differences in their hues (i.e., yellow, orange-yellow, or yellow-orange). These spectra displayed increasing absorption toward the ultraviolet beginning at about 500 nm; no sharp absorption bands were noted. This kind of spectrum is typical of type Ib diamonds (natural or synthetic), which contain nitrogen, predominantly as dispersed single atoms (see Wilks and Wilks, 1994, pp. 70–73).

Figure 9. When exposed to a beam of electrons in a vacuum chamber, this 1.30 ct yellow Gemesis synthetic diamond displayed a green, cross-shaped cathodoluminescence pattern. Photo by Sam Muhlmeister.



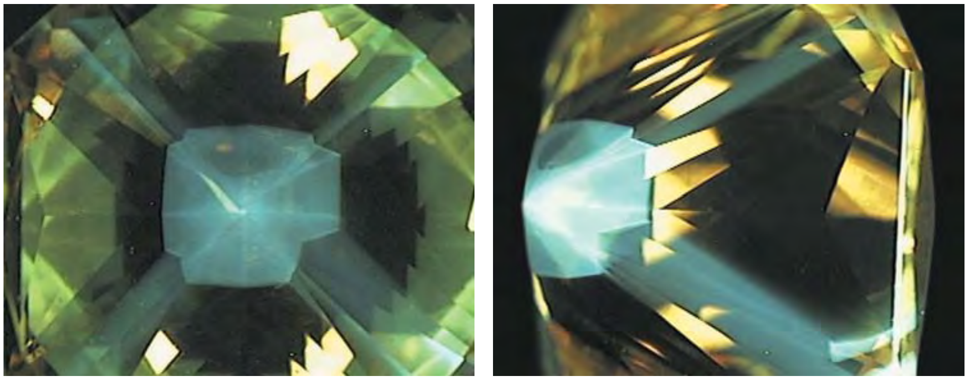


Figure 10. These two views in different orientations show the zoned luminescence pattern of a 1.30 ct Gemesis synthetic diamond as recorded with a De Beers DiamondView luminescence imaging system. Observation of this kind of pattern confirms that the sample is laboratory grown. Photos by Shane Elen.

For purposes of this discussion, the additional spectral and chemical information is organized according to the three hue categories.

Yellow. The two yellow samples on which photoluminescence (PL) spectra were taken all exhibited a single band at 1332.5 cm^{-1}, which is the first-order and characteristic Raman peak for diamonds (Wilks and Wilks, 1994, pp. 66–67); sharp peaks at 637 and 747 nm were also seen in one sample. Three of the four analyzed by EDXRF displayed no X-ray emission peaks, while one exhibited peaks due to nickel and iron. Such elements are derived from the solvent material used for crystal growth by the temperature gradient technique, and small amounts are sometimes retained within the synthetic diamond as metallic inclusions or as tiny particles. Similar results have been obtained by this analytical method for other synthetic diamonds (particularly those that contain visible metal inclusions; see, e.g., Shigley et al., 1993b). Although iron has been found in natural diamonds (as an iron sulfide or oxide mineral), and as such may not be helpful for identification purposes, the presence of nickel in a diamond is considered proof of laboratory growth.

Orange-Yellow. The following sharp (s) or broad (b) emission peaks were noted in one or more of the six orange-yellow samples on which PL spectra were taken: 543 (s), 566 (s), 573 (b), 581 (s), 614 (b), 637 (s), 672 (b), 701 (s), and 747 (s) nm (see, e.g., figure 11). Many of these peaks are thought to be due to nickel-nitrogen, cobalt-nitrogen, or nitrogen-vacancy complexes in the diamond crystal structure (Collins and Stanley, 1985; Lawson and Kanda, 1993a,b; Lawson et al., 1996). Two of the seven samples analyzed by EDXRF displayed no X-ray emission peaks, while the remaining five samples exhibited peaks due to nickel, iron, and/or cobalt. Again, the presence of nickel or cobalt is considered proof that a diamond is synthetic.

Yellow-Orange. The following sharp or broad peaks were noted in one or more of the PL spectra for the five yellow-orange samples tested: 547 (s), 575 (s), 581 (s), 599 (s), 604 (s), 623 (s), 637 (s), 639 (s), 694 (s), 701 (s), 727 (s), 747 (s), 753 (s), 776 (s), 794 (s), and 805 (s) nm (again, see figure 11). Of the six samples analyzed by EDXRF, the spectra of three displayed no X-ray emission peaks, while the spectra of the other three exhibited peaks due to nickel, iron, or cobalt.

Figure 11. The photoluminescence spectra of all the Gemesis synthetic diamonds tested exhibit the sharp 1332.5 cm^{-1} Raman peak that is characteristic of diamond, as well as several PL peaks. The peak at 566 in the orange-yellow sample (top) is due to the presence of cobalt, and is evidence of its synthetic origin. The PL spectrum of the yellow-orange synthetic diamond (bottom) displays a different set of peaks, with the ones at 727, 747, and 753 nm (due to nickel) providing evidence of synthetic origin.

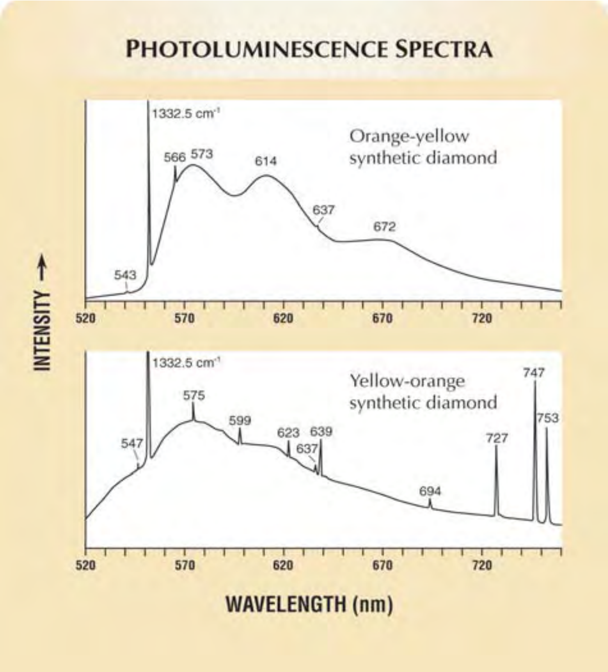


Figure 12. This 1.76 ct yellow Gemesis laboratory-grown diamond, set in a ring with colorless natural diamonds (1.10 ct total weight), is one example of the use of this material in fine jewelry. Photo by Elizabeth Schrader.

MEANS OF IDENTIFICATION

Shigley et al. (1995) summarized the gemological methods of distinguishing natural and synthetic diamonds. On the basis of their examination of yellow synthetic diamonds from Russia, Shigley et al. (1993b), Scarratt et al. (1994), and Sosso (1995) identified their diagnostic features as a cuboctahedral crystal shape and striated or dendritic crystal surface features; crystallographically oriented color zoning; planar or angular graining (along growth-sector boundaries); patterns of yellow or greenish yellow luminescence of varying intensity that correspond to the patterns of color zoning; and opaque metallic, small black triangular, or tiny pinpoint inclusions. Additional distinguishing features can be obtained by visible, infrared, and PL spectroscopy; by fluorescence imaging techniques such as cathodoluminescence and the De Beers DiamondView; and by chemical analysis to detect nickel or cobalt.

In comparison to synthetic diamonds that GIA researchers or others examined previously, the Gemesis material exhibits fewer distinctive visual gemological features. Their yellow/colorless zoning, metallic inclusions, and weak cross-shaped fluorescence patterns are all diagnostic of synthetic diamonds. In addition, their color is more saturated than that of most natural type Ia yellow diamonds (although it can resemble that of natural and other synthetic type Ib diamonds). The cathodoluminescence and De Beers DiamondView luminescence images of the Gemesis samples are unique to synthetic diamonds. Although the visible and infrared spectra were not useful for identification purposes, photoluminescence spectra revealed weak spectral emission peaks due to nickel or cobalt that are indicative of synthetic origin. The laboratory-grown character of this material could be detected by the presence of nickel or cobalt in the EDXRF spectra. However, the absence of these trace elements does not necessarily mean that a particular sample is natural, since two yellow, two orange-yellow, and three yellow-orange specimens lacked these indicators.

EXPERIMENTAL SYNTHETIC DIAMONDS

Gemesis scientists have also grown other kinds of synthetic diamonds on an experimental basis, although the company has not finalized plans to produce them commercially at this time. In addition to the group of 30 commercial samples we examined, we also documented the following experimental specimens:

1. A colorless type IIa 0.20 ct round brilliant, which did not exhibit any X-ray emission peaks in its EDXRF spectrum, and had only a weak fluorescence to short-wave UV radiation, but did display persistent greenish blue phosphorescence (lasting more than 1 minute) after exposure to short-wave UV. This phosphorescence is a key identifying feature of colorless synthetic diamonds.
2. A bluish green irradiated type IIa 0.42 ct round brilliant, which exhibited an X-ray emission peak due to iron and a GR1 radiation band at 741 nm in its absorption spectrum.
3. A blue type IIb 0.55 ct crystal, which exhibited X-ray emission peaks due to iron and cobalt.
4. A green irradiated type IaA 0.65 ct rectangular cut, and a yellow-green type IaA 0.42 ct square cut, both of which displayed facet-related color zoning typical of treated colored diamonds that have been irradiated with electrons or other charged particles. The 0.65 ct sample also exhibited an X-ray emission peak due to iron.

CONCLUSIONS

High-quality yellow laboratory-grown diamonds produced by the Gemesis Corp. represent one of the first commercially available sources of this material specifically for use in the gem and jewelry trade. The company is focusing its marketing approach on the sale of rough and polished stones to jewelry manufacturers (figure 12) and retailers. There are no current plans for the company to produce finished jewelry.

Gemesis is making every effort to prevent the misrepresentation of their laboratory-grown diamonds, and is cooperating with the jewelry industry to provide appropriate identification markings, laboratory certification reports, and factual promotional material. These steps are important since there is no inexpensive testing meter available to quickly distinguish all natural from synthetic yellow diamonds. The best gemological clues to detecting Gemesis laboratory-created yellow diamonds are color zoning and/or metallic inclusions (as seen with a gemological microscope), and their patterns of UV fluorescence. Since these visual indicators may be absent, confirmation of the identity of a Gemesis synthetic diamond may require testing at a gemological laboratory with the appropriate advanced instrumentation.

ABOUT THE AUTHORS

Dr. Shigley (jshigley@gia.edu) is director of GIA Research in Carlsbad, California. Dr. Abbaschian (rabba@mse.ufl.edu) is professor of Materials Science and Engineering at the University of Florida in Gainesville, and Mr. Clarke (cclarke@gemesis.com) is co-chairman of the Gemesis Corporation in Sarasota, Florida.

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