LABORATORY-GROWN DIAMONDS

Dr. James Shigley
Gemological Institute of America (GIA)
Carlsbad, California, USA

INTRODUCTION

Efforts to grow "diamonds" extend back almost 200 years, but these attempts were unsuccessful or were never confirmed until the first documented production of small diamond crystals by the General Electric (GE) Company in mid-December 1954.[1] This discovery had actually been preceded by successful experiments carried out in early 1953 by the Swedish electrical company ASEA (Allmänna Svenska Elektriska Aktiebolaget), but this work was never publically reported until the 1980s.[2,3] The early GE success was followed by the company's announcement in 1970 of larger crystals which were transparent enough to be faceted as gemstones.[4] Since that time, continued research and industrial expansion have resulted in a commercial production of synthetic diamonds not only for gemological use but also for a number of important industrial and high-technology applications that utilize the material's unique physical and chemical properties.

While gem-quality crystals (mainly yellow in color) have been studied since 1970, it is only within the past five years that synthetic diamonds have become commercially available in the jewelry trade. This current presence in the market has several aspects: (1) greater quantity and larger size of the synthetic diamond crystals being grown; (2) production of small crystals used to create melee-size cut stones; (3) increased production of colorless or near-colorless crystals; and (4) improvements in the growth process to create higher-clarity crystals with few, if any, obvious inclusions.

SYNTHETIC DIAMONDS AND IMITATION MATERIALS

Synthetic diamonds have many of the same chemical and physical properties as natural diamonds, and they are visually unidentifiable based on face-up appearance when faceted as gems. Both should be distinguished from so-called "diamond imitations" such as cubic zirconia (CZ, or cubic zirconium oxide) and synthetic moissanite (silicon carbide), which have the appearance of polished diamonds but are very different in their chemical composition and physical properties. Other imitation materials, such as strontium titanate, have historically been used to simulate polished diamonds, but they are less often found today in the marketplace because of their inferior appearance. Cubic zirconia can easily be recognized from diamond using a thermal conductivity meter. In contrast, synthetic moissanite responds as "diamond" with this type of meter, but when it is examined with a microscope or loupe, the pavilion facet junctions appear to be doubled because of how light interacts with this non-optically isotropic material. Means of identification of diamond imitations have been widely discussed in the gemological literature.[5]

SYNTHETIC DIAMOND STUDIES

Gem-quality synthetic diamonds are becoming increasingly available in the jewelry marketplace, especially over the past few years. Studies published over the past 30 years have resulted in an understanding of both the production methods and means of identification of synthetic diamonds. More than twenty articles on this material have been published in GIA's professional journal, *Gems & Gemology* (see Further Reading list). Synthetic diamonds can sometimes be recognized by a trained gemologist, but in the absence of distinctive visual features, positive identification requires examination by an experienced gem-testing laboratory. This differentiation is based upon recognizing the evidences of ancient diamond growth and prolonged residence at high temperatures and pressures deep in the earth versus very recent and rapid growth of synthetic diamond in the laboratory or factory. The development of various testing devices for use by jewelers has helped with the challenge of synthetic diamond detection.

HIGH-PRESSURE, HIGH-TEMPERATURE (HPHT) DIAMOND GROWTH

The traditional method of creating diamonds involves high pressure (5–6 GPa) and temperature (1300–1600ºC/2372–2912ºF) flux growth from a molten metal alloy.[6] A small natural or synthetic diamond seed crystal, placed at the base of a growth capsule, is used to initiate growth. A slight temperature gradient or difference is created between the top and the base of the capsule. Powdered diamond (the source of carbon) dissolves in the molten metal at the upper, hotter end of the capsule, and the carbon atoms then migrate through the metal and attach themselves to the seed crystal at the cooler end of the capsule. The resulting cuboctahedral crystal shape is the outward expression of an internal growth-sector structure. During crystal growth, chemical impurities such as nitrogen or boron are preferentially concentrated in certain internal growth sectors and are almost absent in other sectors. This uneven impurity distribution gives rise to geometric (cross-shaped) patterns of color zoning and fluorescence, which are characteristic of synthetic diamonds grown by the HPHT method. The flux metal is usually an iron-nickel or iron-cobalt alloy—solidified remnants of the flux are sometimes trapped as dark metal inclusions in the synthetic diamond. Synthetic diamond crystals up to 100 carats in size have been produced by the HPHT method, although the majority of them weigh less than 3 carats. This method requires the use of large high-pressure equipment and the maintenance of stable temperature and pressure conditions over the growth period, which typically lasts several weeks. Fluctuations in these conditions create disruptions in growth and visible defects in the crystal. Several diamond crystals can be produced during one growth cycle.

Figure 1 *Colorless synthetic diamond crystals grown by the HPHT method in China. The crystals weigh about 2 carats each, and they display the combination of cube and octahedral crystal faces which is characteristic of HPHT-grown material. Photo Jian Xin Liao, ©GIA*

Faceted HPHT synthetic diamonds can exhibit visual features such as geometric color distribution, fluorescence zoning, and graining patterns (all related to their cross-shaped, growth-sector internal structure), as well as the presence of occasional metal flux inclusions. When they exhibit fluorescence (often in yellow, green, orange or occasionally red colors), the reaction is generally stronger to a short-wave as compared to a long-wave ultraviolet lamp. When the synthetic diamond contains boron, this blue or colorless material can exhibit persistent phosphorescence after the ultraviolet lamp has been turned off (i.e., the synthetic diamond continues to glow). In addition, HPHT-grown crystals normally almost exhibit no “strain” patterns when they are observed between crossed polarizing filters in the microscope or polariscope. All of these distinctive features have been illustrated in the articles published in *Gems & Gemology*.

Historically, most of this HPHT product has been yellow, orangy yellow or brownish yellow. Over the past decade, colorless crystals have become increasingly available, particularly in melee sizes (< 0.2 carat). Addition of boron in the growth system results in blue crystals; boron is often present in lower concentrations among colorless HPHT synthetics as well, thus resulting in telltale phosphorescence. Other colors (pink, green) can be produced either by post-growth treatment processes involving irradiation and heating or, in some cases, by the incorporation of nickel during the growth process.

***Figure 2** A parcel of approximately 10 carats of HPHT-grown colorless diamond melee produced in China. Each colorless crystal is attached to a yellow synthetic diamond seed on which the crystal is grown. When fashioned as gemstones, each crystal will produce a small (~0.01 carat) round-brilliant cut stone. Photo by Wuyi Wang, ©GIA*

LOW-PRESSURE, HIGH-TEMPERATURE (LPHT) OR CHEMICAL VAPOR DEPOSITION (CVD) DIAMOND GROWTH

The second method for creating gem synthetic diamonds involves growth at high temperatures (700–1300ºC/1292–2372ºF) and very low pressures in a vacuum chamber.[7,8,9] Hydrocarbon gases, such as methane (CH_4), are introduced into the chamber, and gas molecules are broken down by means of an energy source (such as microwave beam), which heats the gas to very high temperatures to release the carbon and hydrogen atoms. These atoms are attracted to the colder diamond substrate at the bottom of the chamber and precipitate out on the seed plates as thin layers of diamond. Multiple tabular diamond crystals can be created during a single growth run. When fashioning the tabular piece of synthetic diamond as a gemstone, the crystals are cut as round brilliants or as so-called "fancy" shapes, which better use the flatter crystal to create a larger cut stone.

Most CVD crystals are brownish or grayish (due to the presence of numerous dark nano-inclusions of carbon), but if a tiny amount of nitrogen, silicon, or boron as gases is introduced into the chamber, pink-orange, yellow or blue crystals can be created. Colorless crystals can be produced by this method, but they require a longer time to grow. Most of the CVD-grown colorless gem material being sold is believed to have been produced quickly as brown crystals that were then decolorized by high-pressure heating treatment. Other post-growth treatments can be used to create additional colors, typically pink or blue. CVD-grown diamond crystals are typically less than 10 carats in weight, and most are less than 2 carats as cut stones.

Figure 3 *A tabular synthetic diamond crystal grown by the CVD method. Diamond crystals grown by this technique usually exhibit a black outer edge of polycrystalline non-diamond carbon. This dark outer edge is first removed, and the cut gemstone is then fabricated from the central portion of the crystal. This crystal was grown by GIA researchers.*
Photo Jian Xin Liao, ©GIA

Polished CVD-grown synthetic diamonds exhibit different gemological properties than HPHT-grown material. They tend to display even coloration, banded "strain" patterns, with low-order interference colors (black, gray, white) often oriented perpendicular to the table facet, and are of high clarity with few, if any, tiny dark inclusions (thought to be non-diamond carbon particles). When tested with the DiamondView™ fluorescence viewing instrument, they occasionally display pink-to-orange or yellow-to-green fluorescence, whereas most natural diamonds exhibit blue (or sometimes yellow) fluorescence colors. This fluorescence often appears to have a banded or layered structure that is parallel or is oriented at an angle to the table facet.

COLORLESS SYNTHETIC DIAMOND DETECTION

Colorless synthetic diamonds are type IIa (they lack nitrogen), whereas almost all natural colorless or near-colorless diamonds are type Ia (they contain nitrogen). Colorless or near-colorless type I and type II diamonds can be distinguished by the latter's relative transparency to short-wave ultraviolet radiation, and both types can be quickly separated by infrared spectroscopy.

A number of devices are currently being sold for synthetic diamond detection. They are based on several techniques—transparency to ultraviolet light, ultraviolet-visible or infrared absorption spectroscopy, detection of persistent phosphorescence, and fluorescence or phosphorescence spectroscopy. Before investing in a testing device, one should have a clear understanding of how it operates, any limitations or restrictions on its use, and the frequency of the device giving false testing results. Some devices can distinguish natural from synthetic diamond but do not identify simulants. The Diamond Producers Association recently conducted an evaluation of a number of diamond verification instruments through its Assure Program. Results of this program can be found at the organization's website (https://diamondproducers.com/assure/).

All synthetic diamonds can be detected by the GIA Laboratory. Review articles with information on the synthetic diamonds examined during the past decade by the GIA Laboratory have appeared in the Fall 2016 and Fall 2017 issues of *Gems & Gemology*.[10,11]

Parcels of tiny, colored and colorless diamond melee that may contain synthetic material present a difficult detection problem for the jewelry trade because of their large quantity and small size. GIA has developed an automated instrument to rapidly sort round, colorless to light-yellow polished diamonds (0.005–0.2 carat). This equipment is the basis for a testing service provided by the GIA Laboratory to check parcels of loose diamond melee. GIA has also developed a portable testing device called the iD100 that provides jewelers a rapid and convenient way to check loose or mounted gemstones (0.9 mm [0.005 carat] or greater in diameter) to distinguish natural from synthetic diamonds or from imitations.

Figure 4 *The GIA iD100 diamond testing device allows for the rapid distinction of colorless to near-colorless natural from synthetic diamonds and imitations. The device works with rough or polished diamonds either as loose stones or mounted in jewelry. Photo by Emily Lane, © GIA*

CONCLUSION

Polished synthetic gem diamonds are increasingly encountered in the jewelry marketplace, where they have a place as long as their identity is fully disclosed by the jeweler and they are properly priced. The majority are colorless or near-colorless with good clarity and are less than two carats in size. They are type IIa diamonds, which is a rare type of diamond in nature. Because of their lack of color and high clarity, synthetic gem diamonds may not display any distinctive visual features that would help with identification. A number of commercial testing instruments are now being sold, which provide the most immediate way of detecting loose or mounted synthetic diamonds.

REFERENCES

1. F.P. Bundy et al., "Man-Made Diamonds," *Nature* 176 (9 July 1955): 51-55.
2. H. Liander, "Diamond Synthesis—The True Story," *Industrial Diamond Review* 40 (1980): 412-415.
3. K. Nassau, "Who Was First?" *Lapidary Journal* 45 (November 1991):87-96.
4. G.R. Crowningshield, "General Electric's Cuttable Synthetic Diamonds," *Gems & Gemology* 13 (Summer 1971): 302-314.
5. J. Hobbs, "A Simple Approach to Detecting Diamond Simulants," *Gems & Gemology* 17 (Spring 1981): 20-33.
6. R.C. Burns et al., "Growth of High-Purity Large Synthetic Diamond Crystals," *Diamond and Related Materials* 8 (August 1999): 1433-1437.
7. B.V. Derjaguin and D.V. Fedoseev, "Physico-Chemical Synthesis of Diamond in Metastable Range," *Carbon* 11 (1973): 299-308.
8. F.G. Celii and J.E. Butler, "Diamond Chemical Vapor Deposition," *Annual Review of Physical Chemistry* 42 (1991): 643-684.
9. B. Dischler and C. Wild, Eds., *Low-Pressure Synthetic Diamond: Manufacturing and Applications* (Springer Berlin, 2013): 384 pp.
10. S. Eaton-Magaña and J.E. Shigley, "Observations on CVD-Grown Synthetic Diamonds: A Review," *Gems & Gemology* 52 (Fall 2016): 222-245.
11. S. Eaton-Magaña et al., "Observations on HPHT-Grown Synthetic Diamonds: A Review," *Gems & Gemology* 53 (Fall 2017): 262-284.

Further Reading on Synthetic Diamonds from *Gems & Gemology*

The following journal articles are available for free viewing and download at the GIA website (https://www.gia.edu/gems-gemology). The articles are listed in chronological order so the interested reader can follow the development of ideas on synthetic diamond identification.

J.I. Koivula and C.W. Fryer, "Identifying Gem-Quality Synthetic Diamonds: An Update," *Gems & Gemology* 20 (Fall 1984): 146-158.

J.E. Shigley et al., "The Gemological Properties of the Sumitomo Gem-Quality Synthetic Yellow Diamonds," *Gems & Gemology* 22 (Winter 1986): 192-208.

J.E. Shigley et al., "The Gemological Properties of the De Beers Gem-Quality Synthetic Diamonds," *Gems & Gemology* 23 (Winter 1987): 187-206.

J.E. Shigley et al., "An Update on Sumitomo Gem-Quality Synthetic Diamonds," *Gems & Gemology* 28 (Summer 1992): 116-128.

T.M. Moses et al., "Two Treated-Color Synthetic Diamonds Seen in the Trade," *Gems & Gemology* 29 (Fall 1993): 182-190.

M–L.T. Rooney et al., "De Beers Near Colorless-to-Blue Experimental Gem-Quality Synthetic Diamonds," *Gems & Gemology* 29 (Spring 1993): 38-45.

J.E. Shigley et al., "Two Near-Colorless General Electric Type-IIa Synthetic Diamond Crystals," *Gems & Gemology* 29 (Fall 1993): 191-197.

J.E. Shigley et al., "The Gemological Properties of Russian Gem-Quality Synthetic Yellow Diamonds," *Gems & Gemology* 29 (Winter 1993): 228-248.

J.E. Shigley et al., "A Chart for the Separation of Natural and Synthetic Diamonds," *Gems & Gemology* 31 (Winter 1995): 256-264.

C.M. Welbourn et al., "De Beers Natural Versus Synthetic Diamond Verification Instruments," *Gems & Gemology* 32 (Fall 1996): 156-169.

J.E. Shigley et al., "Gemological Properties of Near-Colorless Synthetic Diamonds," *Gems & Gemology* 33 (Spring 1997): 42-53.

J.E. Shigley et al., "Gemesis Laboratory-Grown Diamonds," *Gems & Gemology* 38 (Winter 2002): 301-309.

W. Wang et al., "Gem-Quality Synthetic Diamonds Grown by a Chemical Vapor Deposition (CVD) Method," *Gems & Gemology* 39 (Winter 2003): 268-283.

J.E. Shigley et al., "Lab-Grown Colored Diamonds from Chatham Created Gems," *Gems & Gemology* 40 (Summer 2004): 128-145.

J.E. Shigley et al., "An Updated Chart on the Characteristics of HPHT-Grown Synthetic Diamonds," *Gems & Gemology* 40 (Winter 2004): 303-313.

W. Wang et al., "Experimental CVD Synthetic Diamonds from LIMHP-CNRS, France," *Gems & Gemology* 41 (Fall 2005): 234-244.

W. Wang et al., “Latest-Generation CVD-Grown Synthetic Diamonds from Apollo Diamond Inc.,” *Gems & Gemology* 43 (Winter 2007): 294-312.

W. Wang et al., “Strongly Colored Pink CVD Lab-Grown Diamond,” *Gems & Gemology* 46 (Spring 2010): 4-17.

S. Eaton-Magaña and U.F.S. D’Haenens-Johansson, “Recent Advances in CVD Synthetic Diamond Quality,” *Gems & Gemology* 48 (Summer 2012): 124-127.

W. Wang et al., “CVD Synthetic Diamonds from Gemesis Corp,” *Gems & Gemology* 48 (Summer 2012): 80-97.

U.F.S. D’Haenens-Johansson et al., “Near-Colorless HPHT Synthetic Diamonds from AOTC Group,” *Gems & Gemology* 50 (Spring 2014): 30-45.

U.F.S. D’Haenens-Johansson et al., “Large Colorless HPHT-Grown Synthetic Gem Diamonds from New Diamond Technology, Russia,” *Gems & Gemology* 51 (Fall 2015): 260-279.

L.C. Loudin, “Photoluminescence Mapping of Optical Defects in HPHT Synthetic Diamond,” *Gems & Gemology* 53 (Summer 2017): 180-188.

S. Eaton-Magaña and C.M. Breedin, “Charts—Features of Synthetic Diamonds,” *Gems & Gemology* 54 (Summer 2018): 202-204.

In addition to these articles, shorter entries on synthetic diamonds have regularly appeared in the Lab Notes and Gem News sections of the GIA journal. Publications on this topic have also appeared in other gemological journals.