AN UPDATED CHART ON THE CHARACTERISTICS OF HPHT-GROWN SYNTHETIC DIAMONDS

James E. Shigley, Christopher M. Breeding, and Andy Hsi-Tien Shen

A new chart, supplementing the one published in the Winter 1995 issue of *Gems & Gemology*, summarizes the features of both as-grown ("non-modified") and treated ("modified") synthetic diamonds currently in the gem market (that is, those grown by the high pressure/high temperature technique). It includes photographs of visual features, information about visible-range absorption spectra, and illustrations of growth-structure patterns as revealed by ultraviolet fluorescence imaging. The chart is designed to help gemologists recognize the greater variety of laboratory-created diamonds that might be encountered today.

Almost a decade ago, Shigley et al. (1995) published a comprehensive chart to illustrate the distinctive characteristics of yellow, colorless, and blue natural and synthetic diamonds. The accompanying article reviewed synthetic diamond production at the time, and discussed how the information presented on the chart was acquired and organized. It also included a box that provided a "practical guide for separating natural from synthetic diamonds." The chart was distributed to all *Gems & Gemology* subscribers, and a laminated version was subsequently made available for purchase.

Since that time, and especially within the past several years, the situation of synthetic diamonds in the jewelry marketplace has become more complicated. Lab-created colored diamonds are now being produced in several countries (including Russia, the Ukraine, Japan, the U.S., and perhaps China and elsewhere), although the quantities continue to be very limited. And today they are being sold specifically for jewelry applications (figure 1), with advertisements for synthetic diamonds seen occasionally in trade publications and other industry media. Recent inquiries to three distributors in the U.S.—Chatham Created Gems of San Francisco, California; Gemesis Corp. of Sarasota, Florida; and Lucent Diamonds Inc. of Lakewood, Colorado—indicate that their combined production of crystals is on the order of 1,000 carats per month (mainly yellow colors), a quantity that does not meet their customer demand.

The synthetic diamonds currently in the gem market are grown at high pressure and high temperature (HPHT) conditions by the temperature-gradient technique using several kinds of high-pressure equipment (belt, tetrahedral, cubic, and octahedral presses as well as BARS apparatuses), and one or more transition metals (such as Ni, Co, and Fe) as a flux solvent/catalyst. Typical growth temperatures are 1350–1600°C. Some lab-grown diamonds are being subjected to post-growth treatment processes (such as irradiation or annealing, or both) to change their colors (and, in some cases, other gemological properties such as UV fluorescence). Thus, the gemologist is now confronted with the need to rec-

See end of article for About the Authors and Acknowledgments.

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Figure 1. HPHT-grown Synthetic diamonds are now available in the gem and jewelry marketplace, as is evident from this attractive 1.00–1.25 ct synthetic yellow diamond jewelry provided by Gemesis Corp. (the colorless diamonds are natural) and the (each under 1 ct) loose synthetic diamonds from Lucent Diamonds and Chatham Created Gems. Composite photo: jewelry images courtesy of Gemesis Corp.; loose diamond photos by Harold & Erica Van Pelt.

ognize faceted synthetic diamonds with colors that are not only "as-grown" (yellow to yellow-brown to brown, blue, green, and colorless), but also result from post-growth treatment processes (yellow, yellow-brown, brown, pink, red, purple, green, or blue-green), as described in Shigley et al. (2004).

While the information presented in the 1995 chart remains valid, the contents of the updated chart reflect the wider variety of HPHT-grown synthetic diamonds now in the marketplace[1]. However, this new chart is not a comprehensive guide to the identification of as-grown and treated synthetic diamonds; rather, it provides an overview of the common characteristics of these materials, which can be helpful in separating them from their natural counterparts.

Recently, synthetic diamonds suitable for jewelry use have also been produced in small numbers at high temperatures but low pressures by the chemical vapor deposition (CVD) process. This material, which is not yet commercially available for jewelry purposes, has very different gemological properties from HPHT-grown samples and, therefore, is not included in this new chart. For further information on CVD-grown material, see Wang et al. (2003) and Martineau et al. (2004).

[1]Reports in the scientific literature indicate that as-grown synthetic diamonds with a green color can also be produced when growth occurs from a nickel solvent-catalyst along with a component in the flux that actively combines with nitrogen (i.e., a nitrogen "getter"; see Chrenko and Strong, 1975; Kanda, 1999). To the best of our knowledge, this kind of synthetic diamond is not presently available in the market.

CONTENTS OF THE NEW CHART

The new chart is organized differently from the one published in 1995 (figure 2). Given the greater amount of information and broader variety of material available, the present chart focuses entirely on synthetic diamonds and does not include entries for natural diamonds. (The reader is directed to the references cited in Shigley et al., 1995—and information provided on that chart—for information on natural as well as early synthetic diamonds. Also, during the past decade, several books and articles have described many of the features of both natural and lab-created diamonds; these are listed in the Additional Reading section at the end of this article.)

Regardless of their color, synthetic diamonds grown by the HPHT technique from a molten metal flux have some common characteristics as a result of their growth conditions. These include their cuboctahedral crystal shape (figure 3), growth features (such as surface markings, color zoning, and graining), and metallic inclusions. Representative photos and photomicrographs illustrating these three types of characteristics are grouped together across the upper portion of the new chart as a way to emphasize their common occurrence in HPHT-grown synthetic diamonds from all current manufacturers.

The lower portion of the chart is divided into two sections—one for information on synthetic diamonds with as-grown (or "non-modified") colors, and the other for those with treated (or "modified") colors (figure 4). For the latter, the entries are divid-

Figure 2. The chart published by Shigley et al. in 1995 included information on natural diamonds as well as on the kinds of HPHT-grown synthetic diamonds available at that time.

ed into the following four categories: (1) HPHT annealing, (2 and 3) irradiation plus annealing at two different temperatures, and (4) irradiation only. Organizing information in this way is not meant to imply that the distinction of untreated and treated colors in synthetic diamonds is important. Rather, it is designed to help the gemologist who must test an unknown sample with a color that might not at first be considered typical of synthetic diamonds. In addition, in some cases—such as yellow or green—the color may be either as-grown or treated.

In this lower portion of the chart, the entries are presented in a column format by color and diamond type (a grouping of diamonds into one of several categories based on their physical and spectral properties; see, e.g., Fritsch and Scarratt, 1992; Wilks and Wilks, 1994, pp. 62–82). Presenting information in this way provides a basis for better understanding the properties of the samples in each category. The visual features summarized in these two sections are supplemented by representative visible-range absorption spectra, as well as by ultraviolet fluorescence images of growth structure obtained with the Diamond Trading Company (DTC) DiamondView instrument (Welbourn et al., 1996). Such data are increasingly important to confirm the identity of some synthetic diamonds. Information obtained by nondestructive chemical analyses for transition metals (such as Ni and Fe), as well as by other spectroscopic (infrared and photoluminescence), cathodoluminescence, and analytical techniques available in the larger gemological laboratories, may also be useful for synthetic diamond recognition.

The information presented in the chart is based on data collected at GIA over the past 25 years on approximately 500 synthetic diamonds from all known sources of production. The photos and photomicrographs were selected to illustrate those visual features of lab-grown diamonds useful for identification purposes.

We do not indicate the manufacturer or distributor of the synthetic diamonds illustrated on the chart for two reasons. First, we know that once a synthetic diamond is sold in the trade, such information may no longer be available (unless a distinctive marking visible with magnification is placed on the girdle surface

Figure 3. HPHT-grown synthetic diamond crystals are usually cuboctahedral in shape, as illustrated by these colored synthetic diamonds from Chatham Created Gems, which weigh between 0.44 and 1.74 ct. Photo by Maha Tannous.

Figure 4. Synthetic diamonds currently sold for jewelry purposes display a range of as-grown and treated colors. The as-grown yellow crystal (2.43 ct) and three yellow faceted samples (0.28–0.84 ct) shown on the left represent the most common kind of synthetic diamond produced today. The colors of the green-to-blue faceted samples (0.20–0.40 ct) shown in the center also were produced during growth. In contrast, the pink-to-pinkish purple colors of the faceted samples (0.16–0.50 ct) shown on the right result from post-growth treatment processes. Photos by Maha Tannous.

by the manufacturer [see below]). Second, since each commercial source uses the same basic HPHT growth technique (although, possibly, with different equipment and procedures), all synthetic diamonds created with this method have many similar gemological properties that do not necessarily allow for a differentiation of the products of various manufacturers.

The visible absorption spectra included in the chart (collected at liquid nitrogen temperature) illustrate the general pattern of spectral features for each kind of synthetic diamond. These features correspond in some instances to those that might be seen using a desk-model spectroscope, and they give rise to the colors of the synthetic diamonds. Specific sharp absorption bands shown on the chart may or may not be present in the spectrum of a particular sample for several reasons (i.e., the type of sample, the method of growth, the flux metals used during growth, and the manufacturer, as well as the type of spectrometer and the data collection conditions). Conversely, other synthetic diamonds may exhibit additional spectral features not shown here. The interested reader is referred to the more complete descriptions of diamond spectra that have been published (see Wilks and Wilks, 1994; Collins, 2000, 2001; and Zaitsev, 2001).

The organization of information in the chart requires some clarification. First, in both natural and synthetic diamonds there are variations in nitrogen and boron contents, and in the degree of nitrogen atom aggregation. Both these factors define diamond type (i.e., type I, type II). Thus, the various type designations actually fall along a continuum, rather than being completely distinct categories as implied by the columnar organization of the lower portion of the chart. Also, with regard to "non-modified" (or as-grown) versus "modified" (or treated) colors, in reality, HPHT growth conditions and HPHT treatment conditions may in some instances (e.g., "yellow/brown," as-grown and as treated with HPHT annealing) be very similar. Consequently, one might see a corresponding similarity in the properties of some synthetic diamonds of the same color listed on the lower left and lower right portions of the chart. Last, the information given represents a consensus of observations on the synthetic diamonds that GIA has documented to date. In some cases (for example, irradiated green synthetic diamonds), only a limited number of samples were available to us for examination. As we study more samples in specific color groups, certain information may need to be expanded or modified.

It should be emphasized that we have not observed all the features shown on the chart in every synthetic diamond we have examined. Rather, all synthetic diamonds we have documented exhibited one or more of the distinctive properties listed here. This reinforces the importance of basing identification conclusions on as wide a variety of properties as possible rather than on just one or two features.

SYNTHETIC DIAMOND IDENTIFICATION

The ability to recognize a synthetic diamond first requires an understanding of the kinds of as-grown and treated materials that are now available. Overall production of gem-quality crystals remains very limited—to the best of our knowledge, perhaps 12,000 carats per year. Almost all are colored crystals up to about 2 ct (with faceted material up to about 1 ct). It is now possible to produce synthetic diamonds that contain little nitrogen and, as a result, might not be strongly colored. However, growth of type IIa colorless material continues to be difficult to achieve in the laboratory, and we do not believe it is available in significant quantities for jewelry purposes. GIA has documented only a few faceted colorless synthetic diamonds obtained from the gem trade during the past decade (see, e.g., Rockwell, 2004).

In recent years, improvements in growth technology and techniques have resulted in colored synthet-

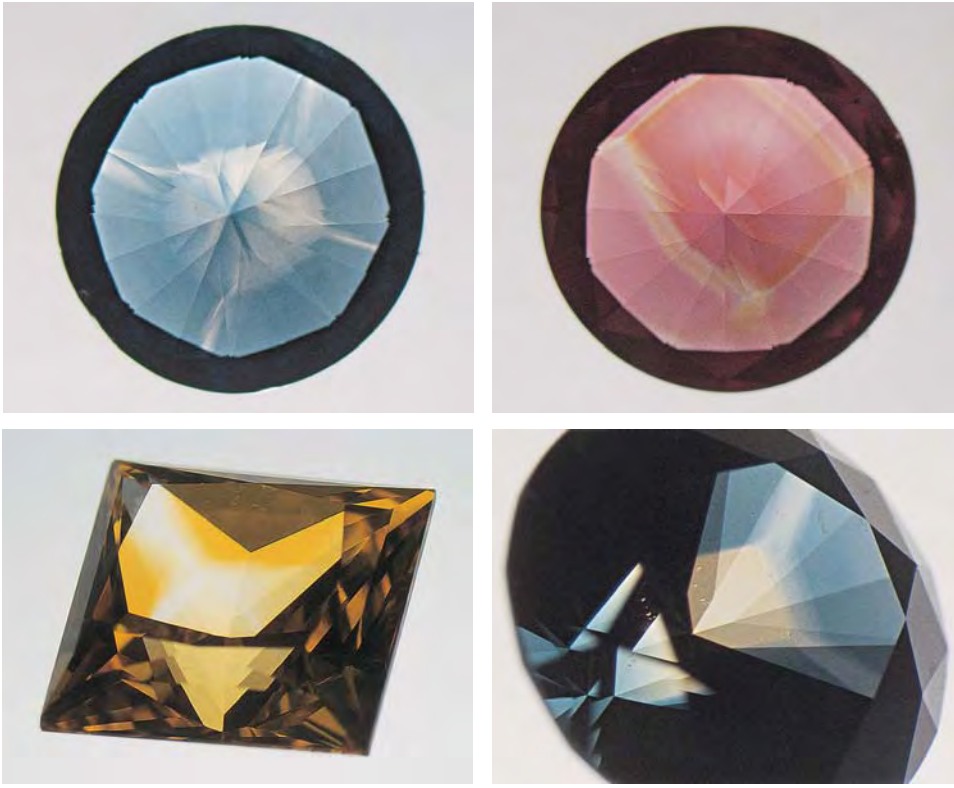


Figure 5. Colored synthetic diamonds often exhibit distinct color zoning due to differences in impurity contents between internal growth sectors. These four examples (0.17–0.68 ct) illustrate how these zoning patterns often appear as seen through the crown and pavilion facets. Immersing the material in a liquid (here, water) can aid in the observation of these patterns. Photos by J. E. Shigley; magnified 20×.

ic diamond crystals that are larger, have lower impurity contents, and are better quality. This finer quality is evident in the presence of few if any metallic inclusions and flaws, as well as less obvious color zoning in some cases. Nonetheless, lab-created diamonds can still be recognized by a variety of methods. Numerous articles (including the present one) and shorter reports describing these methods, which were published in *Gems & Gemology* over the past 30 years, have been collected together for a special volume that will be made available by GIA in early 2005 (Shigley, in preparation). By reviewing this information, as well as what is presented on this chart, the gemologist will be better prepared to recognize this material. The key identifying features of synthetic diamonds are summarized below.

Crystal Shape and Growth Structure. Natural diamond crystals typically exhibit an octahedral form, with many variations due to growth and/or dissolution (Orlov, 1977, pp. 59–106; Wilks and Wilks, 1994, pp. 108–126). In contrast, synthetic diamonds usually have a cuboctahedral form (again, see figure 3), which overlies a geometric arrangement of octahedral, cubic, and dodecahedral internal growth sectors. In a vertical orientation, these sectors radiate upwards and outwards from the seed location at the base of the crystal (see Welbourn et al., 1996, p. 162, figure 5). Diamond crystallization is accompanied by the incorporation of different amounts of impurities in these sectors—thus leading to a segregation of these impurities between sectors. Differential incorporation of impurities gives rise to the distinctive zoning of color, graining, and luminescence seen in many synthetic (as compared to natural) diamonds. When present, boundaries between adjacent color zones are usually sharp and planar (figure 5); they also may intersect to form angular patterns. Adjacent zones may be distinguished merely by lighter and darker appearances of the same color, or by zones of very different color. For example, certain lab-grown green samples now being sold by Chatham Created Gems exhibit both yellow and blue growth sectors when examined with a microscope (see Shigley et al., 2004). Post-growth color treatment processes do not obscure or remove these distinctive visual features, although it may be possible to lessen the visibility of the color zoning during growth (especially if one growth sector predominates within the crystal, while other sectors of differing color are smaller and thus less obvious).

Careful examination using a gemological microscope and different lighting techniques is the best way to see this growth sector–related color zoning in lab-grown diamonds. Immersion of the sample in a liquid (even water) for better observation is also

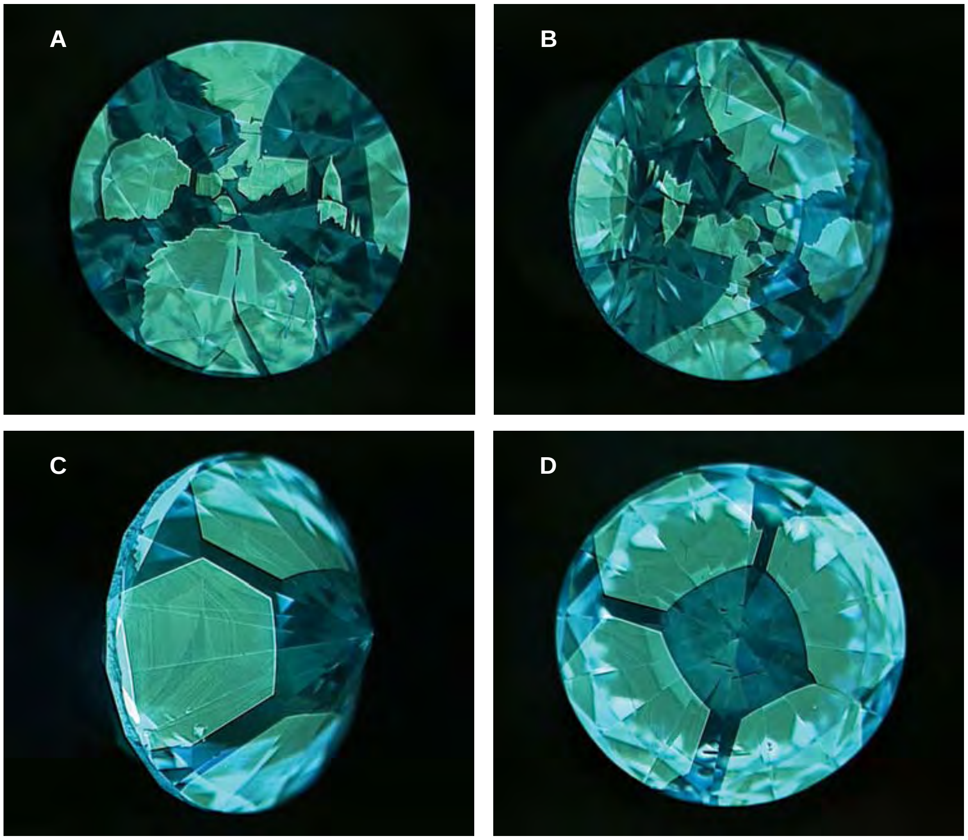
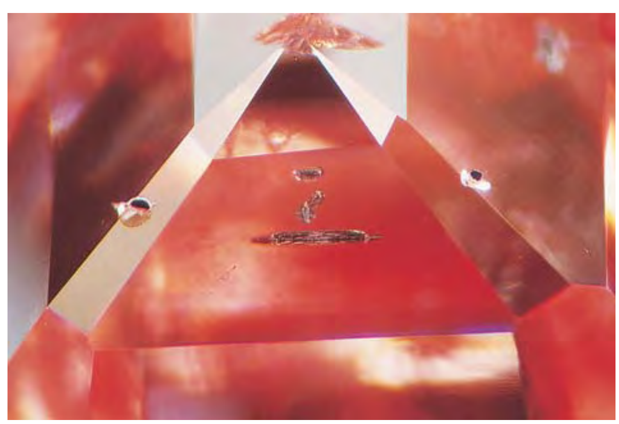


Figure 6. These four DTC DiamondView images of a 0.43 ct blue synthetic diamond illustrate the changing pattern of fluorescent and nonfluorescent growth sectors seen as the sample is rotated. The face-up view (A) shows the four-fold, cross-shaped fluorescence pattern typical of HPHT-grown synthetic diamonds, although in this orientation the sample displays a slightly complicated arrangement of growth sectors. As the sample is rotated toward the pavilion (B and C), the growth sector arrangement becomes more regular in structure. Fine growth striations (resulting from slight variations in impurity contents during growth) are visible within the yellowish fluorescing sectors in the pavilion views (C and D). Images by Andy Shen.

helpful. Such zoning should be evident as well when the sample is examined with a standard UV fluorescence unit or the DTC DiamondView. Depending on the viewing orientation, the zoning can display two-, three-, or four-fold patterns related to the diamond's cubic crystal symmetry. In most cases, the table facet of a polished sample is oriented approximately parallel to the cube face of the original crystal for maximum weight retention during faceting. Therefore, it is often best to look for any four-fold color or fluorescence zoning pattern by observing through the table or crown facets—or, alternatively, nearly parallel to the girdle facets—while rotating the sample. The key is to examine a sample in several orientations to look for changes in color or fluorescence separated by distinct planar boundaries (figure 6).

Inclusions, Graining, and "Strain" Patterns. Unless they are prevented from forming during growth, or are physically removed during faceting, metallic inclusions are a common feature in many polished synthetic diamonds. They may be rounded, elongate, or irregular in shape, and will appear opaque in transmitted light and dark gray-to-black (sometimes with a metallic luster) in reflected light. They may occur singly or in groups, and can vary in size. In some cases, their large size makes them virtually eye-visible (figure 7); whereas in other instances, they are so tiny as to be described as "pinpoint" inclusions, which are often seen in diffuse, cloud-like arrangements (figure 8). (Note that although some of these pinpoint inclusions may be metallic, others may represent different phases formed during synthesis.) Some of these inclusions may even be

Figure 7. Metallic inclusions, such as those shown in this 0.26 ct pink sample, are a distinctive visual feature of many HPHT-grown synthetic diamonds. Photomicrograph by Shane McClure; magnified 25×.



invisible with the magnification of a standard gemological microscope. Because the flux inclusions often contain iron, they can result in the synthetic diamond being attracted to a magnet.

Natural diamonds may display linear, cross-hatched, or irregular ("mosaic") internal graining patterns (Kane, 1980). In synthetic diamonds, internal graining in linear or intersecting geometric patterns appears to be the result of slight differences in refractive index between adjacent growth sectors, or between successive parallel "layers" of material beneath the crystal faces. It is best seen along the boundaries between sectors, or in planes that parallel the outer shape of the original crystal. Since the cuboctahedral crystals are often faceted in square or rectangular shapes for weight retention, one good place to check for graining in faceted samples is near the corners of the table facet (and adjacent crown facets) with magnification (a fiber-optic illuminator can be quite helpful).

Most natural diamonds exhibit anomalous double refraction (ADR) in banded, cross-hatched, or mottled patterns with bright interference colors (when observed through crossed polarizing filters; see Orlov, 1977, pp. 109–116). In comparison, our experience is that synthetic diamonds display much weaker, cross-like "strain" patterns with subdued interference colors (black or gray).

Luminescence. Given the wide variety of synthetic diamonds now available, their reactions to long- and short-wave UV radiation can differ greatly in terms of fluorescence intensity, color, distribution pattern, and phosphorescence. While it has been widely reported that most lab-grown samples display stronger fluorescence to short-wave UV than to long-wave, the opposite reaction has also been observed (as well as the same intensity reaction to both UV lamps), and some samples are inert to both UV excitations. To check for weak UV fluorescence reactions, it is best to observe the sample while in a darkened room, after the eyes have had time to adjust to low light levels. In more recent years, we have noticed an increasing number of synthetic diamonds that display only weak UV fluorescence, or no fluorescence reaction at all.

As mentioned, fluorescence colors can also vary, but typically they range from green to blue to yellow to orange or orange-red. More importantly, however, this fluorescence is often unevenly distributed, so that some portions of the sample fluoresce whereas others do not (or they fluoresce with different colors; see figure 9). This uneven distribution is again a reflection of the arrangement of internal growth sectors with their differing impurity contents, so there is a direct spatial relationship between color, graining, and UV fluorescence patterns. In the most obvious cases, this uneven fluorescence is seen as a square and/or cross-shaped geometric pattern. Again, the orientation of the faceted shape with respect to the original crystal will influence how color, graining, and fluorescence patterns appear, so it is important to examine a sample in several orientations.

Figure 8. In some cases, synthetic diamonds display "clouds" of pinpoint inclusions of uncertain identity. Since some natural diamonds also exhibit similar cloud-like arrangements, these pinpoint inclusions do not provide a reliable means of separation. Photomicrograph by Shane McClure; magnified 30×.

Similar fluorescence patterns in synthetic diamonds can be observed using the cathodoluminescence technique (where the sample is exposed to a beam of electrons while being held in a vacuum chamber). The DTC DiamondView, where fluorescence reactions are excited by exposure of the sample to UV radiation with wavelengths shorter than 230 nm, also provides an excellent tool for viewing surface-related fluorescence and phosphorescence patterns in a sample at different orientations (see Welbourn et al., 1996, and figure 6).

Colorless synthetic diamonds, and any colored samples that contain boron as an impurity, frequently display persistent greenish or yellowish phosphorescence (for up to 60 seconds or longer) when the UV lamp is turned off (see Shigley et al., 1997). Since phosphorescence is a phenomenon that decreases in intensity over time, it is again important to check for this kind of luminescence by viewing the samples in a darkened room. A good technique is to close one's eyes, and then open them at the same time the UV lamp is turned off. Blue (and some near-colorless) synthetic samples containing boron will exhibit electrical conductivity and, interestingly,

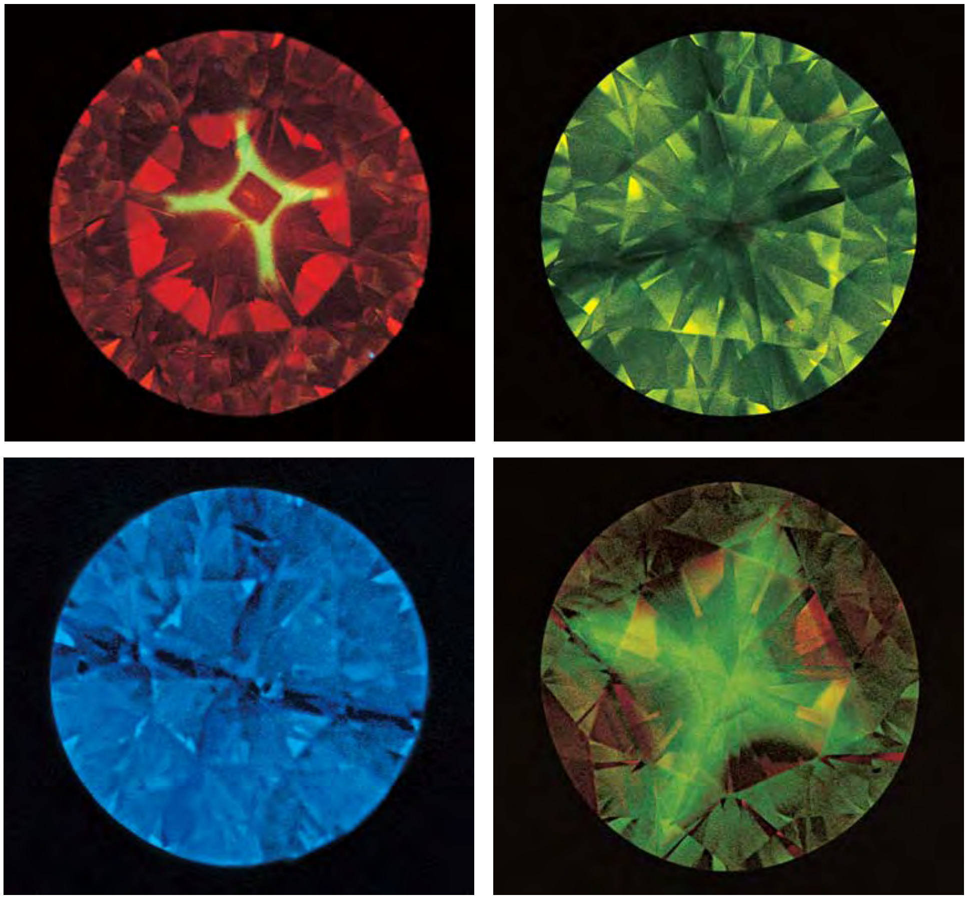


Figure 9. These four synthetic diamonds exhibit typical zoned fluorescence reactions when exposed to long-wave (top left, treated) and short-wave (remaining three images, as grown) ultraviolet radiation. In our experience, the fluorescence is usually stronger to short-wave UV than long-wave for as-grown synthetic diamonds, whereas it can be of equal intensity or stronger to long-wave UV for treated synthetic diamonds. In each instance shown here, certain growth sectors are fluorescing while others are not, resulting in a four-fold cross-shaped pattern. It is important to examine the sample both face-up and face-down, since the fluorescence may be emitted from just a localized area. Photos by Shane Elen and John I. Koivula.

will often display visible electroluminescence in the form of momentary tiny flashes of white to bluish white light when the samples are touched by the conductometer probe.

Chemical and Spectroscopic Analysis. Nondestructive methods of chemical analysis provide another rapid means of identifying synthetic diamonds by detecting flux metals (Ni, Co, and Fe) that are used in diamond growth. Particularly useful in recognizing lab-created diamonds, especially those that lack distinctive visual features, are several spectroscopy techniques that are found today in many gemological laboratories. Because diamond is relatively transparent from the infrared through the visible and ultraviolet regions of the electromagnetic spectrum, numerous absorption and emission features can be detected by these techniques (Zaitsev, 2001, lists the spectral features individually along with a brief description of what is known about them). Specific bands caused by the presence of transition metals are valuable for detecting either as-grown or treated synthetic diamonds by visible spectroscopy (for example, those at 494, 658, and 732 nm, as well as several others, which are all due to nickel; see again Zaitsev, 2001). Caution must be exercised, however, as we now know that some natural diamonds contain small amounts of nickel (see, e.g., Chalain, 2003; Lang et al., 2004; Hainschwang and Notari, 2004). Photoluminescence (PL) spectroscopy is increasingly important for gem laboratories, since many of the optical centers in diamond have associated sharp PL bands that are useful for identification purposes. The interested reader is referred to articles cited in the reference list for examples of the application of these and other spectroscopy methods to diamond characterization (see, e.g., Lawson et al., 1996; Collins, 2000, 2001; Zaitsev, 2000, 2001; Yelisseyev et al., 2002). Additional analytical techniques for detecting synthetic diamonds may become useful in the future.

Other features. As an aid to identification and disclosure, some manufacturers are inscribing a distinctive mark or other information on the girdle facets of their polished synthetic diamonds; such a mark is easily visible with 10× magnification. In addition, certain gem-testing laboratories have agreed to issue grading reports on synthetic diamonds along with a clearly worded statement that they are laboratory grown.

CONCLUSION

The chart accompanying this article presents characteristics of both as-grown and treated synthetic diamonds produced under HPHT conditions using a metal or metal-alloy flux. While visual features such as color zoning and metallic inclusions remain valuable identification criteria, efforts to produce better-quality synthetic diamonds have resulted in such features becoming less evident (or even absent) in some recently grown material. Therefore, the recording of visible-range absorption and other spectra, and the observation of UV fluorescence patterns, have become increasingly important for synthetic diamond identification.

Particularly problematic for jewelers and gemologists are small stones. It is easier, faster, and cheaper to grow synthetic diamonds in the form of melee, but the small size means that the visual identifying features usually are more difficult to see with the microscope, and the large numbers make individual testing of whole parcels impractical. The best solution for parcels of melee is to submit representative samples to a gem-testing laboratory where the material can be fully characterized.

As one looks toward the future, continued research on diamond growth—and the possibility that more and larger synthetic diamonds will be produced—could pose further challenges for the jewelry industry. Although not yet a commercial process, the chemical vapor deposition (CVD) technique could yield larger synthetic diamonds that might lack, for example, growth sector–related color and UV fluorescence zoning patterns (in addition, they would not contain metallic inclusions). The absence of these features would make identification in a standard gemological laboratory more difficult, especially in colorless material, although the evidence from samples examined to date indicates that such material is clearly identifiable with advanced techniques such as the DTC DiamondView.

Scientific efforts are also underway to grow diamonds using flux materials other than transition metals (such as carbonate or silicate compounds; see, e.g., Arima et al., 2002: Litvin et al., 2002; Okada et al., 2002; Pal'yanov et al., 2002; Yamaoka et al., 2002). So far, only microscopic diamond crystals have been produced in this way. However, crystallization from these fluxes under HPHT conditions may eventually yield larger synthetic diamonds that lack some of the diagnostic features (such as metallic inclusions) seen in the HPHT material currently being marketed. Again, there is no evidence to date that such a growth process has been devised that can yield a synthetic diamond crystal of sufficient size and quality to make it suitable for faceting.

Improvements in diamond growth capabilities are an inevitable result of the ongoing scientific interest in diamond as a desirable high-technology material. As new kinds of synthetic diamonds are produced, gemological research must continue to develop practical means of identifying them using both standard and more sophisticated techniques. The goal is to create detection methods that can be applied to all synthetic diamonds—even those of melee size, where the rapid screening of large numbers of diamonds of unknown origin will be important.

ABOUT THE AUTHORS

Dr. Shigley is director of research, and Dr. Breeding and Dr. Shen are research scientists, in the GIA Gem Laboratory, Carlsbad, California.

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