De Beers Natural versus Synthetic Diamond Verification Instruments

By Christopher M. Welbourn, Martin Cooper, and Paul M. Spear

Two instruments have been developed at De Beers DTC Research Centre, Maidenhead, to distinguish synthetic diamonds from natural diamonds. The DiamondSure™ enables the rapid examination of large numbers of polished diamonds, both loose and set in jewelry. Automatically and with high sensitivity, this instrument detects the presence of the 415 nm optical absorption line, which is found in the vast majority of natural diamonds but not in synthetic diamonds. Those stones in which this line is detected are "passed" by the instrument, and those in which it is not detected are "referred for further tests." The DiamondView™ produces a fluorescence image of the surface of a polished diamond, from which the growth structure of the stone may be determined. On the basis of this fluorescence pattern—which is quite different for natural as compared to synthetic diamonds—the trained operator can positively identify whether a diamond is natural or synthetic.

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The subject of cuttable-quality synthetic diamonds has been receiving much attention in the gem trade recently. Yellow to yellow-brown synthetic diamonds grown in Russia have been offered for sale at a number of recent gem and jewelry trade shows (Shor and Weldon, 1996; Reinitz, 1996; Johnson and Koivula, 1996), and a small number of synthetic diamonds have been submitted to gem grading laboratories for identification reports (Fryer, 1987; Reinitz, 1996; Moses et al., 1993a and b; Emms, 1994; Kammerling et al., 1993, 1995; Kammerling and McClure, 1995). Particular concern was expressed following recent announcements of the production and planned marketing of near-colorless synthetic diamonds (Koivula et al., 1994; "Upfront," 1995).

Synthetic diamonds of cuttable size and quality, and the technology to produce them, are not new. In 1971, researchers at the General Electric Company published the results of their production of synthetic diamond crystals up to 6 mm average diameter by the high-pressure temperature-gradient technique using "belt"-type presses (Wentorf, 1971; Strong and Chrenko, 1971; Strong and Wentorf, 1971). These included not only yellow-brown synthetic diamonds but also reduced-nitrogen near-colorless crystals and boron-doped blue crystals (Crowningshield, 1971). In 1985, Sumitomo Electric Industries Ltd., in Japan, started marketing their Sumicrystal™ range of yellow-brown synthetic diamonds; in 1993, they produced high-purity (i.e., near-colorless) synthetic diamond crystals fabricated into diamond "windows." De Beers Industrial Diamond Division (Pty) Ltd. has marketed its Monocrystal range of yellow-brown synthetic diamonds since 1987. None of these three manufacturers has marketed synthetic diamonds for other than industrial or technical applications.

Figure 1. Like their predecessors in many other gem materials, cuttable-quality synthetic diamonds pose a potential threat in the diamond marketplace. To protect the integrity of natural diamonds should significant numbers of synthetic diamonds ever enter the trade, De Beers DTC Research Centre has designed and built two types of instruments—the DiamondSure and the DiamondView—that, together, can successfully identify all synthetic diamonds produced by current synthesis equipment. This figure shows, bottom center and to the right, six De Beers experimental synthetic diamonds: two yellow-brown samples weighing 1.04 and 1.56 ct and four near-colorless synthetics ranging from 0.41 to 0.91 ct. At the top center and to the left are six natural diamonds, ranging from 1.10 ct to 2.59 ct. It is substantially more difficult and costly to grow near-colorless synthetic diamonds than to grow the more usual yellow-brown crystals. De Beers cuttable-quality synthetic diamonds are not available commercially; they have been produced solely for research and education. Natural diamonds courtesy of Louis P. Cvelbar and Vincent Kong, Vincent's Jewelry, Los Angeles. Photo © GIA and Tino Hammid.

In 1990, researchers from Novosibirsk, Russia, published their work on the temperature-gradient growth of synthetic diamonds in relatively small-scale, two-stage multi-anvil presses known as "split-sphere" or "BARS" systems (Pal'yanov et al., 1990). Since then, there have been a number of reports of various groups within Russia intending to set up BARS presses for the purpose of synthesizing diamond. Usually, only one crystal is grown in a BARS press at any one time, whereas many stones can be grown simultaneously in the larger "belt" presses.

Gemologists from GIA and other gemological laboratories have extensively examined synthetic diamonds from each of the above-mentioned manufacturers. Gemological characteristics of these synthetics have been published in a series of articles in *Gems & Gemology* (Crowningshield, 1971; Koivula and Fryer, 1984; Shigley et al., 1986, 1987, 1992; Rooney et al., 1993; Shigley et al., 1993a and b) and elsewhere (Sunagawa, 1995). The conclusion drawn from these studies is that all of the synthetic diamonds examined to date can be positively identified by the use of standard gemological techniques. These results have been summarized in "A Chart for the Separation of Natural and Synthetic Diamonds," published by GIA (Shigley et al., 1995).

The problem facing the gem trade should synthetic diamonds become widespread is that, in general, most near-colorless diamonds are examined for grading purposes only, not for identification.

It is to be expected that, in the main, synthetic diamonds would be clearly identified and sold as such by an honest trade working within national laws and regulations. Nevertheless, a small number of synthetic diamonds have already entered the trade without being declared as synthetic. De Beers has long regarded this potential problem as a serious one. For the last 10 years, researchers at De Beers DTC Research Centre have been actively investigating the characteristic features of synthetic diamonds (see, e.g., Burns et al., 1990; Rooney, 1992). This work has been carried out in close collaboration with the De Beers Industrial Diamond Division's Diamond Research Laboratory in Johannesburg, South Africa, which has been developing high-pressure/high-temperature diamond synthesis techniques for industrial applications for over 40 years. One aspect of this work has been the production of experimental cuttable-quality synthetic diamonds (figure 1), with an extensive range of properties, both for research and for loan to the larger gemological laboratories throughout the world to give their staff members an opportunity to develop their own skills and identification techniques. (Note that these synthetic diamonds are not for sale by De Beers. The Monocrystal synthetic diamonds available commercially from De Beers Industrial Diamond Division (Pty) Ltd. are only sold in prepared forms and are not suitable for cutting as gems.) A second aspect has been the development of instruments that, should the need arise, could be made available to help rapidly identify synthetic diamonds. Such instrumentation would be important should near-colorless synthetic diamonds enter the market in significant numbers. If this were to happen, grading laboratories and others in the trade would need to screen substantial numbers of polished diamonds to eliminate the possibility of a synthetic diamond being sold as a natural stone and thus damage consumer confidence in gem diamonds.

Figure 2. The DiamondSure is based on the presence or absence of the 415 nm line in the stone being tested. Here it is shown with its fiber-optic probe mounted vertically for testing loose stones. On completion of a test, which takes about 4 seconds, the liquid-crystal display on the front panel will give a message of "PASS" or "REFER FOR FURTHER TESTS" (or sometimes "INSUFFICIENT LIGHT" if the stone is very dark or very strongly colored yellow or yellow-brown). Photo by M. J. Crowder.

Although such a circumstance would potentially have a profound impact on the conduct of the gem diamond trade, it is important to put the problem into context. The high-pressure apparatus required to grow synthetic diamonds is expensive, as are the maintenance and running costs. In addition, it is substantially more difficult and costly to grow near-colorless synthetic diamonds than it is to grow yellow-brown crystals. To reduce the amount of nitrogen (which gives rise to the yellow-brown color) that is incorporated into the growing crystal, chemicals that preferentially bond to nitrogen are introduced to the synthesis capsule. These chemicals, known as nitrogen "getters," act as impurities which have an adverse effect on the crystal growth process. To the best of our knowledge, the only near-colorless synthetic diamonds to appear on the gem market thus far were 100 Russian-grown crystals displayed at the May 1996 JCK Show in Las Vegas. The largest of these weighed about 0.7 ct, but two-thirds of the crystals weighed 0.25 ct or less. Most of these were not suitable for polishing because of inclusions, internal flaws, and distorted shapes.

Nevertheless, De Beers has considered it prudent to invest substantial resources to address this potential problem and thus ensure that the trade is prepared for this eventuality. This article describes two instruments, developed at De Beers DTC Research Centre, that are capable of screening large numbers of diamonds and facilitating the rapid and unambiguous identification of synthetic diamonds.

Ideally, the trade would like to have a simple instrument that could positively identify a diamond as natural or synthetic with the same ease as a thermal pen distinguishes between diamonds and non-diamond simulants such as cubic zirconia. Unfortunately, our research has led us to conclude that it is not feasible at this time to produce such an ideal instrument, inasmuch as synthetic diamonds are still diamonds physically and chemically, and their distinguishing features are based on somewhat subtle characteristics involving the presence or absence of various forms of impurities and growth structures. The instruments developed at our Research Centre have been designed to be used in a two-stage procedure. The first instrument, called the *DiamondSure*™ allows the operator to screen large numbers of stones rapidly. This instrument will successfully detect all synthetic diamonds produced by current equipment (including experimental synthetics grown at the Diamond Research Laboratory at extremes of conditions and with non-standard solvent/catalysts). However, a small proportion of natural diamonds will also produce the same response from the instrument. A second stage of examination is therefore required. This could be by standard gemological examination. However, a second instrument has been developed, called the *DiamondView*™, which enables a positive identification to be made quickly and easily[1]. Certain aspects of the design of these instruments are proprietary and so cannot be described in this article. However, we have endeavored to give sufficient information on their operation to show clearly how they may be used to identify synthetic diamonds.

THE DiamondSure™ SCREENING INSTRUMENT

Description. The DiamondSure (figure 2) has been designed for the rapid examination of large numbers of polished diamonds, whether loose or set in jewelry. It is 268 mm long by 195 mm deep by 107 mm high (10.6 × 7.7 × 4.2 ins.), and it weighs 2.8 kg (6.2 lbs.). Measurements are made by placing the table of a polished diamond on the tip of a fiber-optic probe, the diameter of which is 4 mm. For loose stones, the fiber-optic probe is mounted in a vertical position, and a collar is placed around the end of the probe so that stones can easily be positioned over the probe tip (again, see figure 2). The instrument can be used on diamonds mounted in jewelry provided that the table is sufficiently accessible for the probe tip to lie flat against it (see figure 3). When the probe tip is in contact with the table of the diamond being examined, the operator presses the TEST button on the front panel of the instrument or, alternatively, presses the button mounted on the side of the probe. The time required for the instrument to complete a measurement is approximately 4 seconds. It is designed to work with diamonds in the 0.05–10 ct range. This size range is determined by the diameter of the fiber tip, because the instrument responds to the light that is retro-reflected by the cut diamond and re-enters the probe tip. Should the need arise, fibers with larger or smaller diameters could be manufactured to accommodate larger or smaller stones. The instrument is powered by a universal-input-voltage power supply, and so is suitable for use in any country.

The instrument automatically measures the intensity of retro-reflected light in a small region of the spectrum centered on 415 nm. Using proprietary software, it compares the intensity data, as a function of wavelength, to the 415 nm optical absorption line typically seen in natural diamond. The measurement is highly sensitive; values of 0.03 absorbance units at the peak of the 415 nm line, relative to a baseline through the absorption-line shoulders, are

Figure 3. The DiamondSure probe can be removed from its mounting to test a diamond in a ring or other setting. Photo by M. J. Crowder.

[1] *The* DiamondSure *and* DiamondView *instruments are covered worldwide by granted or pending patent applications.*

easily detected. We detected the 415 nm line in over 95% of all natural diamonds tested by the DiamondSure instrument (see Test Samples and Results section, below), but not in any of the synthetic diamonds. If this feature is detected in a stone, the instrument displays the message "PASS." If this feature is not detected, the message "REFER FOR FURTHER TESTS" is displayed. If very dark or very strongly colored yellow or yellow-brown stones are measured, the message "INSUFFICIENT LIGHT" may be displayed. With such stones, the optical absorption in the wavelength range used by the instrument is so strong that practically no light is being returned to the detector. However, in the tests reported in detail in the Test Samples and Results section below, all the yellow-brown synthetic diamonds used—including the largest (2.53 ct) sample—tested successfully. If the "INSUFFICIENT LIGHT" message is obtained with a particularly large stone, repositioning the stone on the probe will often produce a valid measurement. If the probe tip does not lie flat against the table, the light detected may be composed mostly of light reflected from the table without entering the diamond. In this case, the instrument would "fail safe" by "referring" the sample.

Although the 415 nm defect is not present in as-grown synthetic diamonds, it can be formed in nitrogen-containing synthetics by very high-temperature heat treatment in a high-pressure press (Brozel et al., 1978). Temperatures in the region of 2350°C are required, together with a stabilizing pressure of about 85 kbars to prevent graphitization. At these extreme conditions, the lifetime of the expensive tungsten carbide press anvils becomes very short, the diamond surfaces are severely etched, and the likelihood that the diamond will fracture is significant. Given the present technology, it would not, therefore, be commercially practical to heat-treat synthetic diamonds to form sufficient 415 nm defects.

A small proportion of natural diamonds, less than 5% from our tests, do not exhibit the 415 nm feature strongly enough to be detected by the DiamondSure. These include D-color and possibly some E-color diamonds, as well as some brown diamonds. As for diamonds of "fancy" color, the 415 nm line is absent from natural-color blue (type IIb) diamonds, as well as from some fancy yellow and some pink diamonds. When these stones are tested, the DiamondSure displays the message "REFER FOR FURTHER TESTS." It is important to recognize that the fact that these stones were not "passed" by the instrument does not necessarily mean that they are synthetic or in any way less desirable than stones that have been passed. The message simply means that additional testing is required for an identification to be made.

Test Samples and Results. During the development of the DiamondSure, approximately 18,000 polished natural diamonds were tested. In the final phase of testing, which we report here, two instruments from an initial batch of 10 were each used to test a total of 1,808 randomly chosen known natural diamonds. Most of these 1,808 stones weighed between 0.25 and 1.00 ct, although we included some as small as 0.05 ct and some over 10 ct. The largest stone was 15.06 ct, and it tested successfully. Colors were in the D to R range, as well as some browns and some fancy yellows. In these particular tests, all except six stones were round brilliants; in an earlier experiment, though, more than a hundred fancy-shaped stones tested successfully.

The tests were carried out at the London offices of the De Beers Central Selling Organisation. The instruments were used by a number of operators. In general, a combination of daylight and fluorescent lighting was used, but no special care with respect to lighting conditions was necessary. The average figure for "referrals" for these 1,808 diamonds was 4.3%.

In a separate evaluation, we used a third DiamondSure to test 20 D-color stones, of various shapes, ranging from 0.52 to 11.59 ct. Eight of the stones were passed, and 12 were referred. This indicates that, because of its sensitivity, the instrument can detect a very weak 415 nm line even in some D-color stones.

The first two instruments were also tested on a range of De Beers experimental synthetic diamonds. A total of 98 samples were used: 23 in the yellow-brown range, 0.78–2.53 ct; 45 near-colorless, 0.20–1.04 ct; 15 pale-to-vivid yellow, 0.19–0.63 ct; and 15 medium-to-vivid blue, 0.24–0.72 ct. (See figure 1 for examples of the near-colorless and yellow De Beers synthetic diamonds tested.) All of these synthetic diamonds were round brilliants except for one fancy yellow sample, which was an emerald cut. Each was tested 10 times on each instrument. In addition, some yellow and near-colorless Russian BARS-grown synthetic diamonds were tested several times on one of the instruments. In all cases, the synthetic diamonds were "referred for further tests."

THE DIAMONDVIEW™ LUMINESCENCE IMAGING INSTRUMENT

Background: Growth Structure in Synthetic and Natural Diamonds. In the articles cited above on the gemological characteristics of synthetic diamonds, it was noted that the patterns of ultraviolet-excited fluorescence exhibited by synthetic diamonds are quite distinctive and so can be used to positively identify them. The DiamondView rapidly generates these fluorescence patterns—which are produced by differential impurity concentrations between growth sectors and growth bands—and provides clear images of them. With a little experience, it is relatively easy to recognize patterns that are characteristic of natural or synthetic diamonds. With practice, one can obtain and identify the fluorescence images of two or three diamonds per minute.

The reason that fluorescence patterns can be used to identify synthetic diamonds is that the basic growth structure of synthetic diamonds is quite distinct from that of all natural diamonds, and details of these growth structures can be inferred from the fluorescence pattern. Synthetic diamonds grow essentially as cubo-octahedra. The degree of development of cube {100} or octahedral {111} faces depends on a number of parameters, but most notably on the growth temperature. At relatively low growth temperatures, cube growth predominates; whereas at relatively high growth temperatures, the diamond morphology approaches that of an octahedron, although small cube faces are still present (Sunagawa, 1984; see figure 4). For synthetic diamonds grown using pure nickel as the solvent/catalyst, pure cubo-octahedra are produced. However, if other metals are used with or instead of nickel, then minor faces of dodecahedral {110} and trapezohedral {113} orientation also tend to be present (Kanda et al., 1989; see figure 5a). In certain circumstances (e.g., when cobalt is a constituent of the solvent/catalyst, and getters have been used to reduce the nitrogen content), additional trapezohedral {115} faces may be present (Rooney, 1992; Burns et al., 1996). For large synthetic diamonds grown by the temperature-gradient method, growth starts on a seed crystal of synthetic or natural diamond and develops outward and upward, as illustrated in figure 5b. If the crystal shown in figure 5a were to be sectioned along the planes A and B, the growth patterns exposed by these planes would be as shown in figures 5c and d, respectively. (For a comprehensive but easy-to-understand description of the numbers, or Miller indices, used to describe the orientation and position of faces on a crystal, see J. Sinkankas' *Mineralogy*, 1986, pp. 119–127.)

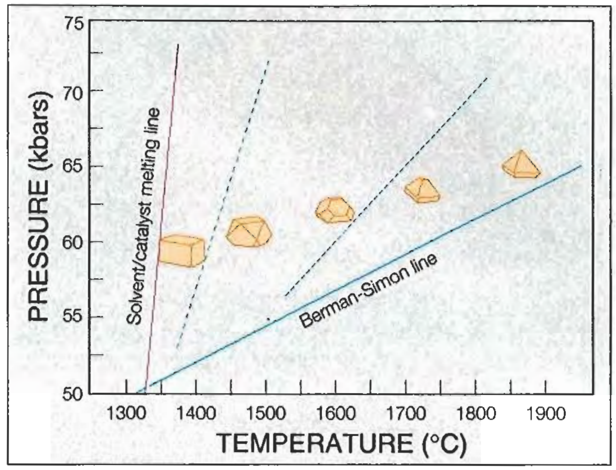


Figure 4. This schematic diagram shows the dependence of synthetic-diamond morphology on growth temperature (after Sunagawa, 1984). The Berman-Simon line separates the region in which diamond is the thermodynamically stable phase and graphite is metastable (above the line) from that where graphite is stable and diamond metastable (below the line). Diamond growth can only occur to the right of the solvent/catalyst melting line. The dashed lines approximately represent regions where similar morphologies are produced, indicating that pressure is also a factor in determining crystal shape.

Those regions of a crystal that have a common growth plane are referred to as growth sectors. As the crystal grows, different growth sectors tend to take up impurities in differing amounts. For instance, nitrogen, the impurity responsible for the yellow to yellow-brown color in synthetic diamonds, is generally incorporated at highest concentrations in {111} growth sectors, with the concentration in {100} sectors being about half that of {111} (Burns et al., 1990). (However, at low growth temperatures, the nitrogen concentration in {100} sectors exceeds that of {111} [Satoh et al., 1990].) Nitrogen levels are substantially lower in the {113} growth sectors and very much lower in the {110} sectors. The polished slice of synthetic diamond shown in figure 6 was cut parallel to the (110) plane, with the seed crystal at the bottom and the (001) face at the top. The variation in nitrogen concentration between growth sectors results in the zonation of the yellow color.

Nickel and cobalt impurities can also be taken up by the growing crystal to form optically active

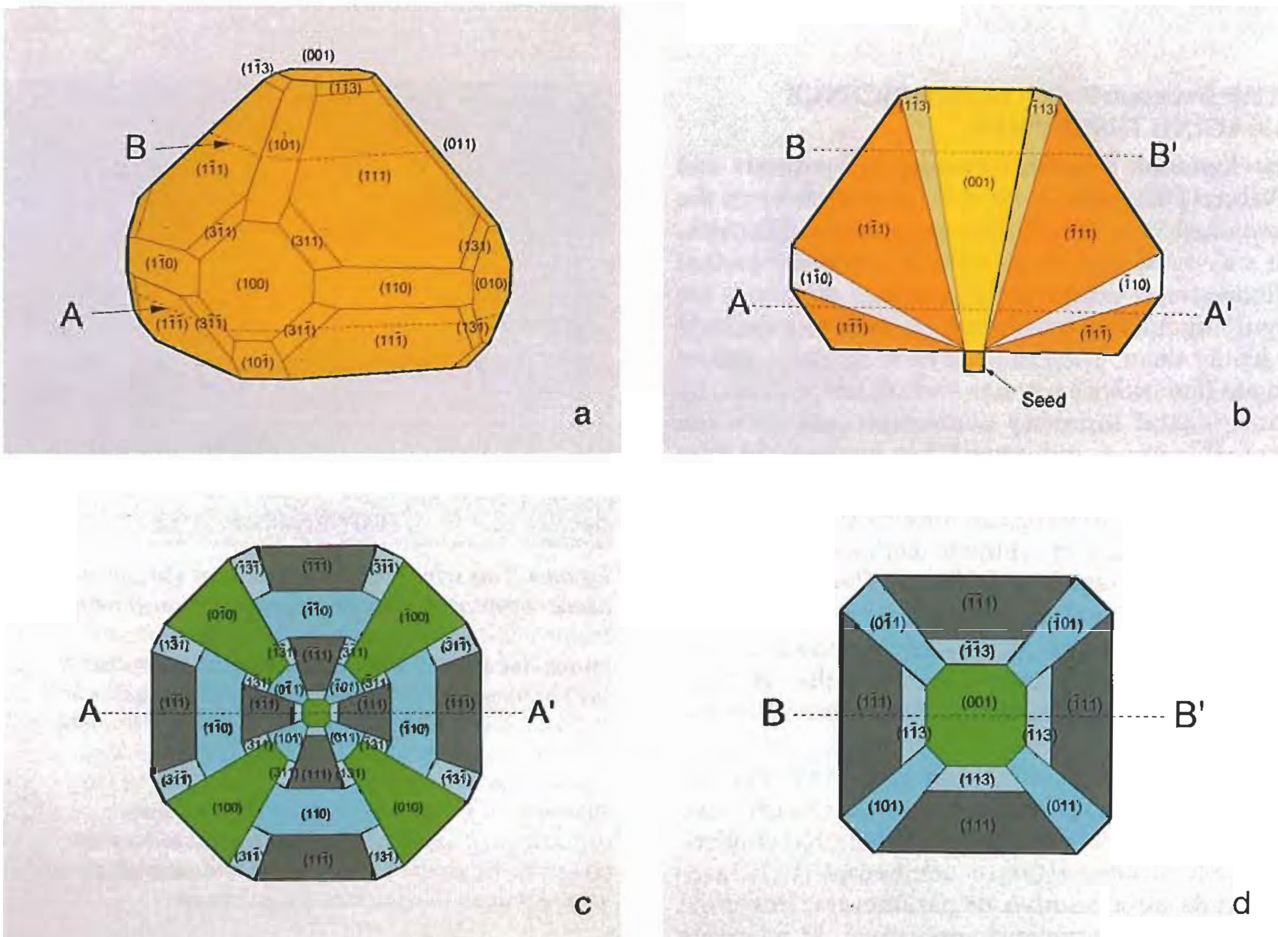


Figure 5. The idealized synthetic diamond crystal, seed-grown by the temperature-gradient method, exhibits major octahedral {111} and cube {100} growth faces, and minor dodecahedral {110} and trapezohedral {113} growth faces (a). A view of the central section parallel to the (110) dodecahedral plane of this same crystal shows the position of the seed at the base of the crystal, from which growth develops outward and upward (b). The variation in color saturation reflects the variation in nitrogen concentration between growth sectors in yellow-brown synthetic diamonds. The fluorescence pattern shown in (c) is that of a section from this synthetic diamond crystal, parallel to the (001) cube plane, indicated by the plane A in (a) and the line A-A' in (b). In yellow-brown synthetics, {100} sectors tend to fluoresce green, {110} and {113} tend to fluoresce blue. and {111} sectors are usually largely inert. The fluorescence pattern shown in (d) is that of a (001) section indicated by the plane B in (a) and the line B-B' in (b).

defects, but they are incorporated exclusively in {111} sectors (Collins et al., 1990; Lawson et al., 1996). In low-nitrogen synthetic diamonds, nickel gives rise to a green color; heat-treated cobalt-grown diamonds show a yellow fluorescence. Boron is another impurity that is readily taken up by a growing synthetic diamond. Blue, semi-conducting synthetic diamonds are produced by using chemical getters to reduce nitrogen levels and deliberately introducing boron into the synthesis capsule. Boron concentrations are highest for {111} sectors, next highest in {110} sectors, and substantially lower in other sectors. Even when these impurities are not present in concentrations high enough to influence the color of the crystal, they still can cause fluorescence behavior that varies between growth sectors.

For natural diamonds, the basic form of growth is octahedral. Small natural cubo-octahedral diamonds have been found, but these are very rare (J. W. Harris, pers. comm., 1990). Dodecahedral and trapezohedral flat-faced growth has never been observed in natural diamonds. Rounded dodecahedral diamonds are very common, but these shapes are formed by the dissolution of octahedral diamonds (Moore and Lang, 1974). Figure 7a is a schematic diagram of a natural diamond in which

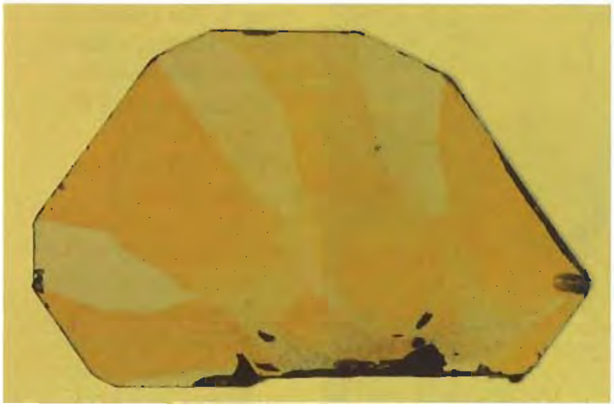


Figure 6. This optical micrograph of a slice cut parallel to the (110) dodecahedral plane from a De Beers yellow-brown synthetic diamond shows the greater concentration of nitrogen (and thus greater saturation of yellow) in the {111} growth sectors than in the {100}, {113}, or {110} growth sectors (again, refer to figure 5b for a diagram of the different growth structures in such a crystal at this orientation). The slice is 5.01 mm across × 3.20 mm high × 0.71 mm thick.

the octahedral faces have undergone partial dissolution so that rounded dodecahedral faces are beginning to form. A schematic diagram of a section through a central cube plane of this idealized crystal is shown in figure 7b.

Dodecahedral faces that appear flat may be found on "coated" diamonds, but here the growth is fibrous and quite distinct from flat-faced {110} growth (Machada et al., 1985).

A form of nonoctahedral growth that is relatively common in natural diamonds is so-called cuboid growth. The mean orientation of cuboid growth is approximately along cube planes, but the growth is hummocky and distinct from flat-faced cube growth. On the rare occasions that cuboid growth is well developed compared to octahedral growth, diamonds with quite spectacular shapes are produced, as is the case with the "cubes" found in the Jwaneng mine (Welbourn et al., 1989). It is not uncommon for otherwise octahedrally grown diamonds to have experienced a limited amount of cuboid growth, particularly on re-entrant octahedral faces. This is shown schematically in figure 7b.

For most natural diamonds, the conditions in which they grew fluctuated over time, so different types and levels of impurities were incorporated at different stages of growth. This resulted in differences in fluorescence behavior between growth bands within the crystal.

Uncut synthetic diamonds can be readily identified by visual inspection because of their crystal morphology and the remnants of the seed crystal present. However, these external features are lost when the stone is polished.

For many years, cathodoluminescence topography has been used to image growth-dependent pat-

Figure 7. In this schematic diagram of (a) the morphology of a typical natural diamond, the octahedral faces, decorated with trigon etch pits, have undergone partial dissolution so that rounded dodecahedral faces are beginning to form. The schematic diagram of the fluorescence pattern from a section through a central cube plane of this idealized crystal (b) shows concentric rectangular bands of octahedral growth and regions where re-entrant features have been overgrown by cuboid growth.

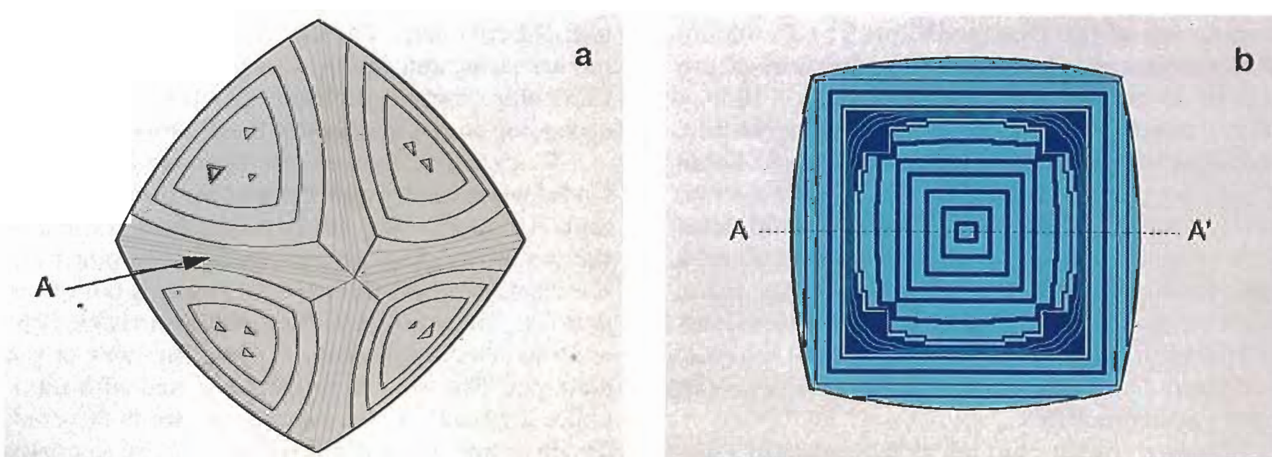


Figure 8. The DiamondView consists of a fluorescence imaging unit (left) in which the TV camera is located between two lamp housings (upper left), with special stone holders for loose (foreground and figure 9a) and ring-set (foreground and figure 9b) diamonds, and a specially configured computer. Photo by M. J. Crowder.

terns in minerals, including diamond (Woods and Lang, 1975; Hanley et al., 1977; Marshall, 1988; Ponahlo, 1992). In cathodoluminescence (CL), an electron beam, rather than ultraviolet radiation, is used to excite luminescence. Commercial CL instruments use a cold cathode discharge tube operating in a relatively low vacuum to produce the electron beam. Although CL is invaluable in the study of minerals, the fact that it requires a vacuum can be a disadvantage when large numbers of stones must be surveyed rapidly, as it may take several minutes to pump down to the required pressure. Also, the surfaces of samples may become contaminated by deposits of products from the pump oil. It was to avoid these practical problems associated with CL that our Research Centre developed an ultraviolet-excited fluorescence imaging technique.

Description of the DiamondView. The DiamondView consists of a fluorescence imaging unit (60 cm high by 25 cm wide by 25 cm deep (24 in. × 10 in. × 10 in.), which weighs approximately 20 kg (44 lbs.), and a specially configured computer (figure 8). Loose stones are mounted between the jaws of a stone holder that allows the stone being examined (from 0.05 to approximately 10 ct) to be rotated about a horizontal axis while it is being viewed (see figure 9a). Ring-mounted stones can also be examined, provided that the total height of the ring is not too great (see figure 9b). Other simple jewelry mounts can also be accommodated.

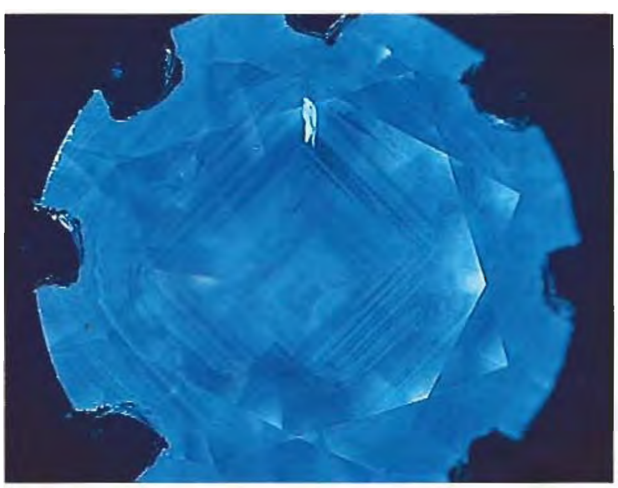
The instrument illuminates the surface of a diamond with intense ultraviolet light, specially filtered such that almost all of the light reaching the sample is of wavelengths shorter than 230 nm. The energy of this ultraviolet light is equal to or greater than the intrinsic energy band-gap of diamonds. This has two important consequences. First, radiation of this energy will excite fluorescence in practically all types of diamond irrespective of whether they fluoresce to the standard long- and short-wave UV radiation (365 and 254 nm, respectively) routinely used by gemologists. Second, at wavelengths shorter than 230 nm, all types of diamond absorb light very strongly. This means that fluorescence is generated very close to the surface of the diamond, so that a clear two-dimensional pattern can be observed. The fluorescence emitted is viewed by a solid-state CCD (charge-coupled device) video camera that has been fitted with a variable-magnification objective lens. The camera has a built-in video picture store, and images can be integrated on the CCD chip from 40 milliseconds up to 10 seconds, depending on the intensity of the fluoresecence.

To examine a stone, the operator inserts the loaded stone holder into the port at the front of the unit. An interlocking safety mechanism eliminates the possibility of any ultraviolet light escaping from the instrument when the stone holder is out of the port. The stone is first illuminated with visible light and the camera is focused on, say, the table of the diamond. The stone is then illuminated with ultraviolet light and the fluorescence image is recorded. The instrument is controlled by an IBM PC-compati-

Figure 9. The loose-stone holder is inserted into the measurement port of the DiamondView (left). The gear mechanism allows the stone to be rotated about a horizontal axis while located within the instrument, for alignment and observation of surface fluorescence patterns characteristic of its internal growth structures. The ring holder (right) can accommodate a ring-set stone that has a total height no greater than 30 mm (1.2 in). Rings mounted in this holder can be rotated about the axis of the holder and moved forward and backward along this axis. Photo by M. J. Crowder.

ble computer running Microsoft® Windows™ 3.1–compatible proprietary software. The computer has a 120 MHz Pentium processor, 32 Mb of RAM (random access memory), and PCI (peripheral component interconnect) video input and graphics display cards. The fluorescence image is displayed on a high-resolution, 1024 × 768 pixel, computer monitor. If additional views of the stone are required, the stone holder can be rotated, without removing it from the chamber, to bring other parts of the stone's surface into view. Fluorescence images that are required for future reference can be stored on the PC's hard drive. The number of images that can be stored is limited only by the size of the hard drive. In this model, the 800Mb drive could hold over 500 images. Images can be archived using, for instance, a tape drive or writable compact disk. The display screen produced by the DiamondView software can be seen in figure 8. The mouse-operated buttons that control the instrument are located beneath the main window, in which the current image is displayed. This image may be compared with up to 16 previously recorded images. These can be recalled on four pages, each of which has four "thumb-nail" windows, displayed on the right of the main window. Tutorial files consisting of 16 "thumb-nail" images, complete with text notes, are provided in the software to help the operator identify fluorescence patterns. The user can also produce "customized" tutorial files.

Figure 10. This fluorescence image of a 0.3 ct near-colorless natural diamond shows concentric bands of octahedral growth with a re-entrant feature below the center of the image and several regions of hummocky cuboid growth. The blue color is typical of most natural diamonds and results from so-called band A emission together with some fluorescence from the 415 nm system.



Sample Images. The DiamondView was tested with the same synthetic diamonds described above for the DiamondSure tests, together with about 150 randomly chosen natural diamonds. Following are some examples of the images obtained. Figure 10 shows the fluorescence image of a near-colorless natural 0.3 ct diamond mounted in an eight-claw ring setting. The fluorescence in this sample ranges from bright blue to dark blue; it is typical for natural diamonds and results from so-called blue band A emission together with some fluorescence from the 415 nm system (see, e.g., Clark et al., 1992). The stone was polished such that the table is close to a cube plane, and the striae visible in the image result

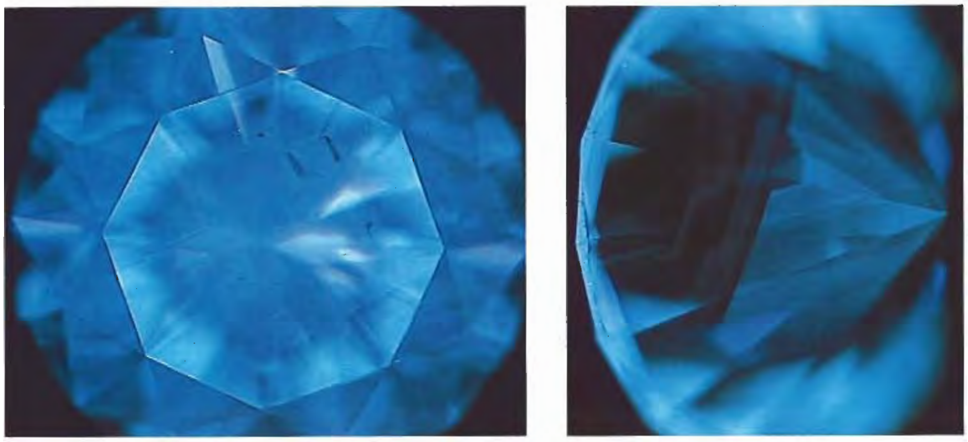
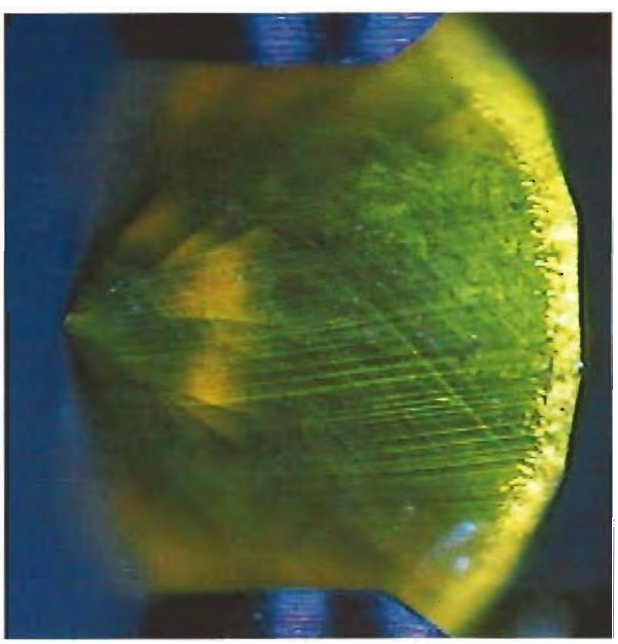


Figure 11. The fluorescence image of the table (left) of this 1.5 ct natural diamond shows concentric bands of octahedral growth and a number of re-entrant features. The pavilion of this stone (right) shows some narrow bright blue octahedral bands, with some re-entrant features, in an otherwise weakly fluorescing region.

from bands of octahedral growth intersecting the table. Re-entrant features are evident in the lower part of the image, and cuboid growth horizons can be seen in various places, particularly toward the left in the image. The concentric rectangular bands, the re-entrant feature below the center of the image, and the hummocky cuboid growth bands are all similar to those shown in idealized form in figure 7b.

Figure 12. In this yellow-brown plastically deformed natural diamond, approximately 0.1 ct, the fluorescence image shows green H3 (503 nm) emission from two sets of parallel slip bands. This type of plastic deformation, covering the entire stone, is not uncommon in natural diamonds, but it has not been found in synthetic diamonds.



The fluorescence image of a 1.5 ct near-colorless natural diamond is shown in figure 11 (left). The banding is less pronounced in this stone than in the one shown in figure 10, but it is still apparent. However, the image of part of the pavilion of this stone shows greater contrast, as is evident in figure 11 (right).

The fluorescence image of an approximately 0.1 ct yellow-brown natural diamond is shown in figure 12. This diamond is plastically deformed, and the green lines are produced by slip bands (planes along which part of the crystal has undergone a shearing displacement) decorated by nitrogen-related H3 (503 nm) defects. Two sets of parallel slip bands may be seen. This type of plastic deformation, which covers the entire stone, is not uncommon in natural diamonds but has not been found in synthetic diamonds.

The DiamondView image of a 2.19 ct yellow-brown De Beers experimental synthetic diamond is shown in figure 13. From the symmetry of the pattern, it is clear that the table has been cut close to a cube plane. This image may be compared with the schematic diagram shown in figure 5c. The central (001) sector is surrounded by four other cube sectors, which fluoresce yellowish green, and by four inert octahedral sectors. The yellowish green color is due to the H3 (503 nm) system together with some green band A emission (again, see Clark et al., 1992). Narrow, blue-emitting dodecahedral sectors lie between pairs of cube and pairs of octahedral sectors.

The fluorescence image of a 0.33 ct near-colorless De Beers experimental synthetic diamond is shown in figure 14 (left). Although the fluorescence is blue, it is a less saturated, more grayish blue than is typical of natural diamonds (again, see Shigley et al., 1995). A brief examination of this image reveals

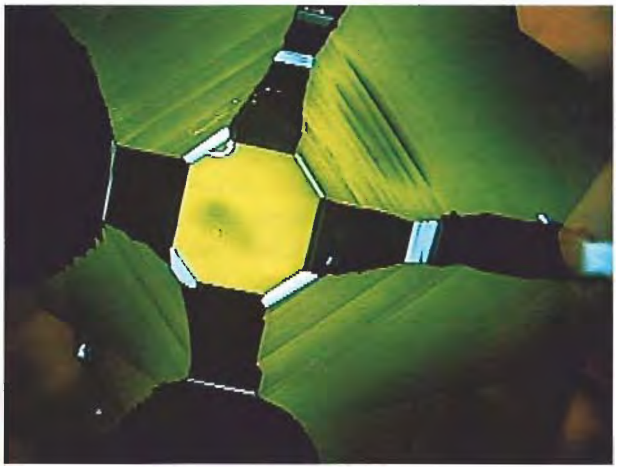


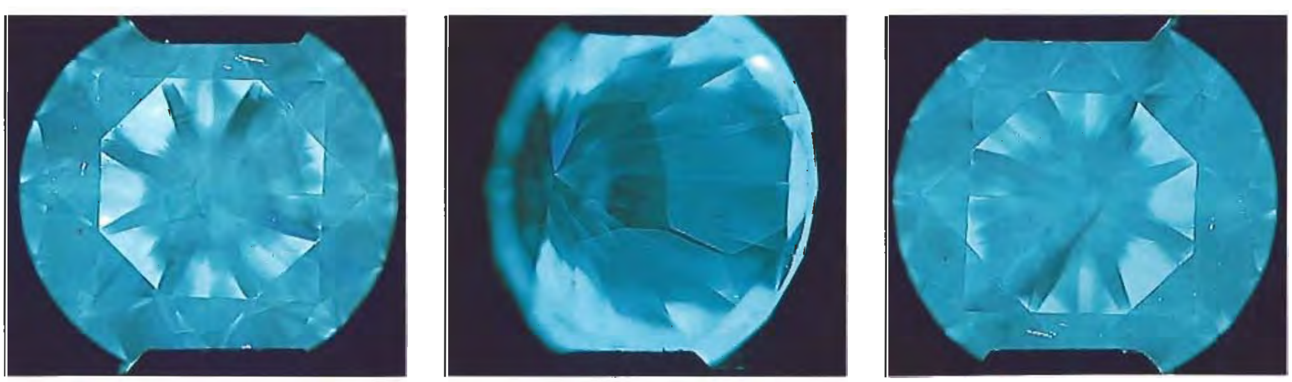
Figure 13. The fluorescence image of the table and some of the surrounding crown facets of this 2.19 ct yellow-brown De Beers experimental synthetic diamond shows yellowish green emission from the central (001) sector and four other cube sectors. The color is due to the nitrogen-related H3 (503 nm) system together with some green band A emission. The inert regions between the yellowish green cube sectors are octahedral sectors. Narrow, blue-emitting dodecahedral sectors lie between pairs of cube sectors and pairs of octahedral sectors. Trapezohedral {113} sectors had not developed significantly in this sample.

a central (001) sector surrounded by four somewhat brighter octahedral sectors. Pairs of octahedral sectors are separated by narrow, less intensely emitting dodecahedral sectors. The view of the pavilion of this stone (figure 14, center) shows the growth-sector pattern even more clearly. A weakly emitting (001) sector may be seen in the region of the culet. This is surrounded by pale blue {111} sectors lying between narrow, less strongly emitting {110} sectors.

The DiamondView has also been used to examine a complete range of synthetic diamonds, including both yellow and near-colorless Russian BARS stones. In all cases, the stones could be positively identified as synthetic from their fluorescence patterns.

Another feature of near-colorless and blue synthetic diamonds is that they tend to exhibit long-lived phosphorescence after excitation by ultraviolet light. Many natural diamonds do phosphoresce, but phosphorescence is relatively uncommon in near-colorless stones and is generally much weaker and for a shorter period than in near-colorless and blue synthetic diamonds. The DiamondView instrument has been designed to exploit this phenomenon in order to assist further in the identification process. Phosphorescence images can be captured at times from 0.1 to 10 seconds after the ultraviolet excitation has been switched off. An example of a phosphorescence image from the 0.33 ct near-colorless synthetic diamond is shown in figure 14 (right). The exposure time was 0.4 second, commencing after a delay of 0.1 second. Phosphorescence is strongest from octahedral growth sectors.

Figure 14. The fluorescence image of the crown (left) of this 0.33 ct near-colorless De Beers experimental synthetic diamond shows a near-central (001) sector surrounded by four somewhat brighter octahedral sectors, which are separated by narrow dodecahedral sectors. The blue color is less saturated than is typical of natural diamonds. The view of the pavilion (center) shows a weakly emitting (001) sector in the region of the culet surrounded by pale blue octahedral sectors lying between narrow, less strongly emitting dodecahedral sectors. A phosphorescence image (right), recorded with an exposure time of 0.4 second and a delay of 0.1 second after the ultraviolet excitation had been switched off, shows strongest phosphorescence from octahedral sectors. Strong, long-lived phosphorescence is a characteristic feature of near-colorless and blue synthetic diamonds.



We have loaned the GIA Gem Trade Laboratory DiamondView and DiamondSure instruments, which they are evaluating for use as part of GIA GTL's standard diamond testing procedures. In this evaluation, the DiamondSure is the first test for all diamonds that the laboratory takes in (T. Moses, pers. comm., 1996). Using the DiamondView, GIA Research in Carlsbad, California, recorded fluorescence patterns on eight Russian and three Sumitomo Electric synthetic diamonds (all yellow). From these patterns, all of these diamonds were quickly and easily recognized as synthetic (J. E. Shigley, pers. comm., 1996).

CONCLUSION

The DiamondSure is a relatively inexpensive instrument capable of screening 10 to 15 stones per minute and automatically producing a "PASS" or "REFER FOR FURTHER TESTS" result. It is based on the presence or absence of the 415 nm line, which was found in more than 95% of natural diamonds tested but has not been found in any synthetic diamonds. Because a small proportion of natural diamonds would be referred by this instrument, additional testing may be required. The DiamondView is a more complex and significantly more expensive instrument. It enables the operator to determine whether a diamond is natural or synthetic on the basis of a far-ultraviolet-excited fluorescence image. Synthetic diamonds are identified by their distinctive growth-sector structure, whereas natural diamonds show either purely octahedral growth or a combination of octahedral and hummocky "cuboid" growth. Because only two or three stones can be examined per minute, and an operator must interpret the fluorescence image, it would not be practical to use the DiamondView alone for screening large numbers of stones. It would therefore be appropriate for both instruments to be used together or for operators of a DiamondSure to have ready access to a laboratory with a DiamondView.

At present, DiamondSure and DiamondView instruments are being loaned to a number of major gem testing laboratories throughout the world. Both instruments have been designed so that they can be manufactured in volume should near-colorless cuttable synthetic diamonds enter the gem market in significant numbers. Although it has yet to be shown that this will be the case, these instruments could be made commercially available quickly, should a real need arise. The price of the instruments will depend very much on the numbers to be produced, but it is estimated that a DiamondSure instrument might cost in the region of a few thousand dollars, whereas the more complex DiamondView might be 10 times as much.

The development of these instruments ensures that synthetic diamonds of cuttable quality can be easily identified. With such tools available to members of the gem trade, the existence of such synthetics should not be a cause of major concern.

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