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About the Cover: Responding to trade concerns about the possible commercial availability of cuttable-quality synthetic diamonds, De Beers researchers in Maidenhead, England, have developed two types of machines—the DiamondSure and the DiamondView—to separate natural from synthetic diamonds. These new instruments are the focus of the lead article in this issue, by Christopher Welbourn, Martin Cooper, and Paul Spear.

The natural-diamond rings shown here contain round brilliants weighing a total of 5.19 ct (top), 4.38 ct (left), 5.36 ct (bottom), and (right) 6.78 ct with a 3.49 ct yellow emerald-cut diamond. Courtesy of Hans D. Krieger, Idar-Obststein, Germany.

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Color separations for Gems & Gemology are by Effective Graphics, Compton, CA.

Printing is by Cadmus Journal Services, Baltimore, MD.

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Subscriptions in the U.S.A. are priced as follows: $59.95 for one year (4 issues), $149.95 for three years (12 issues).
Non-U.S. subscriptions are $70.00 for one year, $180.00 for three years.

Special annual subscription rates are available for all students actively involved in a GIA program: $49.95, U.S.A.; $60.00, elsewhere. Your student number must be listed at the time your subscription is entered.

Single issues may be purchased for $15.00 in the U.S.A. and $18.00 elsewhere. Discontinuance is given for bulk orders of 10 or more (minimum order: $100). All issues are available for purchase. Please address all inquiries regarding subscriptions to the Subscriptions Department.

To obtain a Japanese translation of Gems & Gemology, contact the Association of Japan Gem Trust, Okachimachi Cy Bldg., 5-15-14 Ueno, Taito-ku, Tokyo 110, Japan.

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Gems & Gemology is published quarterly by the Gemological Institute of America, a nonprofit educational organization for the jewelry industry, 1660 Stewart Street, Santa Monica, CA 90404.

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In this era of increasingly sophisticated synthetic materials and forms of enhancement, gemologists need every possible weapon in their testing arsenal. To this end, this issue of Gems & Gemology offers two articles relating to new methods of identification. One describes two instruments designed by De Beers researchers to help identify synthetic diamonds. These instruments successfully address a need and desire for quick, easy, and reliable diamond testing in a fast-paced, competitive world. Thus, they represent a growing trend in gemology toward “black box” methodology, which can be practiced with a minimum of skill and technical knowledge. Although such instruments provide a great service to our industry, there are some problems for which no “black box” may ever be available, that to date have been solved only by using expensive instrumentation in a well-staffed laboratory. Foremost among these are the separation of some natural and synthetic rubies, sapphires, and emeralds. The second article, by the Gubelin Laboratory’s Christopher Smith, involves an identification technique that may seem esoteric to some readers, but is actually closer to “grass roots” gemology. Internal growth-structure analysis (that is, analysis of features such as twinning and crystal planes within a cut natural or synthetic stone that reflect its distinctive growth history) requires relatively little new equipment. It does, however, call for the unique talents of the professional gemologist.

There are many levels of need among gemologists. Those operating in a well-financed testing laboratory may have the resources and space to consider X-ray, ultraviolet, infrared, and Raman spectroscopy—and/or other systems costing $100,000 or more. A well-equipped electron microprobe can cost over a million dollars. Most gemologists, however, have neither the finances nor the space to consider much more than a binocular microscope, refractometer, polariscope, desk-model spectroscope, and a few other relatively small and inexpensive items. As recently as the middle of this century, amateur gemologist Count E. C. R. Taaffe recognized that he had something hitherto unknown—the first taaffeite—using only a loupe and other rudimentary equipment. He lacked even a refractometer.

Although any gemologist with access to extensive testing instrumentation may benefit from internal growth-structure analysis, it is of particular value to the gemologist in the office, the jewelry store, the small laboratory. First introduced to gemology by Karl Schmetzer, its advantage is that it requires only a binocular microscope and a few accessories that can be purchased or even created by modifying equipment on hand. Such a method may require extensive and time-consuming “jury-rigging” (at relatively minor expense), but it can yield information as essential as that provided by the most sophisticated instrumentation. However, it depends heavily on the most important of all gemological tools: the gemologist’s own knowledge, skill, and meticulous practice in testing.

We can be thankful whenever a veritable “black box” is designed to meet one of our testing challenges. But when none is available, there is no substitute for creative gemology.
THE OBSERVATION OF MAGNETISM IN SYNTHETIC DIAMOND

"A Chart for the Separation of Natural and Synthetic Diamonds," by J. Slugley et al. [Winter 1995, pp. 256-264] is one of the jewelry trade's most useful reference aids. The article dealt with the range of indicator tests available to the jeweler/gemologist, one of which is magnetism. The article spawned two responses, which were published in the Spring 1996 "Letters" section (p. 63). Both Dr. Hanneman's original letter and the reply by GIA Gem Trade Laboratory Vice-President Tom Moses describe methods for observing magnetism in a gemstone, but both have their drawbacks.

Suspending a gemstone on a fine thread, and bringing a powerful magnet close, does enable observation of the slightest magnetic response, but great care is required, inasmuch as just the observer's breath can induce movement of the stone. Attaching the specimen to the thread with Blu-Tack or Stik-Tack is courting disaster, for both products induce a magnetic response!

In my 1992 Tucson lectures on the magnetism test, I suspended an elastic band from the thread, and the band acted as a cradle to support the stone. In 1995, I demonstrated two tests, using a ring set with a De Beers experimental synthetic diamond. In one test, I simply tied a fine thread through the ring shank and brought the magnet close; in the second test, I floated the ring on a polystyrene raft in a small basin of water. In both cases, the ring was drawn to the magnet. Australian gemologist Rod Brightman suspends the rare-earth magnet and brings the jewel close.

In my experience, these tests work for most of the known synthetic diamonds, including those from De Beers, General Electric, and Russia. However, I have not observed any such response in Sumitomo synthetic diamonds.

Dr. Jeff Harris, at Glasgow University, indicated that the iron sulphide minerals pyrrhotite and pentlandite would show a magnetic response in a natural diamond, and together we observed the slightest of responses from such mineral inclusions in natural diamond when the stone was floated on a tiny (5 x 5 mm) polystyrene raft. Fortunately for the gemologist, such inclusions are black, and they are almost always accompanied by stress fractures that are also filled with black mineral films. [Metallic inclusions in synthetic diamonds do not exhibit stress fractures.] Even when large, such natural inclusions produce only the slightest response. In contrast, the synthetic's inclusions induce a distinct response even when they are dust-like. When such inclusions are large, an unset synthetic diamond may jump 6 mm or more off a surface to reach the rare-earth magnet.

Readers might also like to know of a synthetic diamond demonstration last November at the Seattle (Washington) chapter of the GIA Alumni & Associates and the Canadian Gemmological Association Conference. A faceted blue synthetic diamond [courtesy of De Beers] phosphoresced for at least 12 hours after 30 minutes' exposure to short-wave ultraviolet radiation. Subsequent carefully controlled observations showed that the phosphorescence lasted 11 days!

ALAN HODGKINSON
Ayrshire, Scotland

Editor's note: We contacted Mr. Moses, who had mentioned Blu-Tack in his Spring 1996 issue reply to Dr. Hanneman's letter. He confirmed that he has seen a magnetic reaction in some Blu-Tack, but only when the Blu-Tack is noticeably dirty or discolored, in which case it should be replaced.
MORE ON THE HISTORY OF DIAMOND SOURCES

It was with great pleasure that I read the two-part article “A history of diamond sources in Africa” [Winter 1995 and Spring 1996, pp. 228-255 and 2-30, respectively]. Dr. Janse did an excellent job of condensing the most important historical events that have taken place during the more than 130 years since the first diamond finds were made in South Africa.

For those GoG readers with an avid interest in the early years of South Africa’s development following the initial diamond finds, I would like to add a reference to Dr. Janse’s extensive bibliography: South Africa’s City of Diamond: Mineworkers and Monopoly Capitalism in Kimberley, 1867-1895, by Dr. William H. Worger [Yale University Press, New Haven and London, 330 pp.]. I found it to be a fascinating factual account of the hardships experienced by those involved in the diamond industry during this formative period. It also looks at the impact of the diamond discoveries on South Africa, and Kimberley in particular, both culturally and economically.

I highly recommend this book to any gemologist, jeweler, or hobbyist who is intrigued by the history and development of the diamond industry.

CHRISTOPHER P. SMITH
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In Reply

I thank Christopher Smith for his kind words about my articles. I have a copy of Dr. Worger’s book in my library and, although the socio-economic background of early diamond mining was beyond the scope of my papers, I agree that this provides an excellent overview. For further information in this area, I would add Capital and Labour in the Kimberley Diamond Fields 1871-1890, by Dr. Robert V. Turrell [Cambridge University Press, New York and London, 1987, 287 pp.], and the more readable Kimberley, Turbulent City, by Brian Roberts [O. Phillip, Cape Town, in Association with the Historical Society of Kimberley and the Northern Cape, 1976, 413 pp.].

I take this opportunity to thank the many readers for their complimentary remarks on these articles.

A. J. A. JANSE, PH.D.
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CORRECTION

In the article “Russian Demantoid, Czar of the Garnet Family,” by W. R. Phillips and A. S. Talantsev (Summer 1996), the identification of the yellow fibrous inclusions (often described as “horse-tails,” “tousled children’s hairs,” or “emanating from chromite grains”) was incorrectly attributed. These asbestiform inclusions—for many years believed to be byssolite—were identified as chrysotile, a variety of serpentine, by Dr. Edward J. Gübelin. He first reported the results of his analyses at the October 1992 International Gemmological Conference in Paris. The identification was made by means of SEM for the chemical composition and X-ray diffraction analysis for the crystal structure.
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 spectral 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 structures 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.

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 Kovula, 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 (Kovula et al., 1994; “Upfront,” 1995).

Synthetic diamonds of cuttable size and quality, and the technology to produce them, have not been 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 Chrenlo, 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 diamonds. 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; Kovola and Fryer, 1984, Shigley et al., 1986, 1987, 1992, Rooney et al., 1993, Shigley et al., 1995a 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 gen-
eral, 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., Bains 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.

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 quantities of polished diamonds for the presence of synthetic diamonds. Figure 2. The DiamondSue 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” for substances “INSUFFICIENT LIGHT” if the stone is very dark or very strongly colored yellow or yellow-brown. Photo by M. J. Crowder.
numbers of diamonds and facilitate 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 synthetic crystals 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. 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 DIAMOND SURE™ 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 x 7.7 x 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 at 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
De Beers Verification Instruments
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 (Brand 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 near-colorless 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 Organization. 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 4.5%. In a separate evaluation, we used a third DiamondSure to test 2D 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 "refereed 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 cubic-octahedra. The degree of development of cubic (100) or octahedral (111) faces depends on a number of parameters, but most notably on the growth temperature. At relatively low growth temperatures, cubic growth predominates; whereas at relatively high growth temperatures, the diamond morphology approaches that of an octahedron, although small cubic faces are still present (Sunagawa, 1984; see figure 4). For synthetic diamonds grown using pure nickel as the solvent/catalyst, pure cubic-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.)

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...
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 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. 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, {110} sectors tend to fluoresce green, {111} 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 sectors in such a crystal at this orientation). The slice is 5.01 mm across x 3.20 mm high x 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 cubic 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-
tens in minerals, including diamond (Woods and Lang, 1975; Hanley et al., 1977; Marshall, 1988; Ponahl, 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. x 10 in. x 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 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 fluorescence.

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-
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 800 Mb 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.

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. The stone was polished such that the table is close to a cube plane, and the striae visible in the image result from the cutting process.
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 blueness 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) centres. 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 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) centres. 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 Clarke 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 (111) 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 (111) 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.

Acknowledgments: The authors wish to thank all the members of the staff at De Beers DTC Research Centre, Maidenhead, who have been involved with the development of the instruments described in this article. Particular recognition is due to P. S. Rose and G. M. Brown, for the development of the DiamondSure™ instrument, and to T. M. Peyman and R. C. Burns, for the development of the DiamondView™ instrument. S. J. Quain was responsible for most of the instrument testing. The authors are also grateful to the synthesis team under Dr. R. C. Burns at the Diamond Research Laboratory, Johannesburg, for the supply of the De Beers experimental synthetic diamonds used to test these instruments.

REFERENCES


INTRODUCTION TO ANALYZING INTERNAL GROWTH STRUCTURES: Identification of the Negative d Plane in Natural Ruby

By Christopher P. Smith

Growth-structure analysis has become increasingly important as a gemological tool for locality classification and distinguishing between natural and synthetic gemstones. This article presents the methods and instruments needed to analyze the internal growth structures of corundum. As an application of this testing procedure, the negative rhombohedral d (0112) plane is documented as part of the crystal habit in a small number of natural rubies—both as a subordinate form and, for the first time in natural rubies, as a dominant crystal form. Until recently, this crystal face was primarily associated with flux-grown synthetic rubies and sapphires.

Over the past several years, the analysis of crystal habits and internal growth features observed in gemstones has gone beyond the realm of purely academic crystallography to applications in gem identification. The wealth of information learned through the determination of external habits and internal growth structures, which reflect the conditions in which the gem grew, can aid in (1) the separation of natural and synthetic gem materials, as well as (2) the identification and characterization of gemstones from different deposits around the world (figure 1).

For example, the type of twinning is an important factor in separating natural from synthetic amethyst (see, e.g., Crowningshield et al., 1986; Koivula and Fritsch, 1989; Kiefert and Schmetzer, 1991). In diamond, internal growth structures (commonly referred to as graining) can affect the clarity of the stone (see, e.g., Kane, 1980) and be responsible for certain color manifestations (see, e.g., Kane, 1985; Hofer, 1985; Kane, 1987). These and other growth structures also aid in the separation of natural and synthetic diamonds (see, e.g., Shigley et al., 1992, 1993; Sunaga, 1992, 1993; Rooney et al., 1993, and the Welbon et al., 1996, article elsewhere in this issue on the new De Beers DiamondView instrument). Beryl classification from various sources, in addition to the separation of natural from synthetic emeralds, has also benefited from the study of internal and external growth features (see, e.g., Lind et al., 1986; Kiefert and Schmetzer, 1991b; Schmetzer et al., 1991).

In corundum in particular, growth-structure analysis has become a standard procedure in many laboratories for both source determination and natural versus synthetic distinctions (see, e.g., Schmetzer, 1986a and b; Schmetzer, 1987; Kiefert, 1987; Kiefert and Schmetzer, 1987, 1988, 1991a; Hänni and Schmetzer, 1991; Peretti and Smith, 1993; Hänni
et al., 1994; Schmetzer et al., 1994; Smith and Surdez, 1994; Peretti et al., 1995; Smith et al., 1995).

To make this new tool more accessible to gemologists in all areas of the industry, this article describes the basic techniques and instrumentation that can be used to perform growth-structure analysis of gems. It also provides an example of how this procedure was recently applied to identify in natural rubies a rare crystallographic feature that had previously been considered an indicator of flux-grown synthetic rubies.

GROWTH-STRUCTURE ANALYSIS

Background. Understanding of the methods and applications that will be presented here requires, first, an explanation of some fundamentals of the analysis of internal growth structures. The external form of a crystal consists of a combination of individual crystal faces. The relative size and number of these faces dictate the overall external shape, or habit, of the crystal (figure 2). Which crystal faces form and their relative prominence are heavily influenced by the conditions of formation (e.g., pressure/temperature relationships, chemical fluctuations of the fluids from which the crystals precipitated, etc.). Consequently, analysis of these habits can provide important information concerning the way a particular gemstone grew. However, since the external habit of a stone is removed during the fashioning process, internal growth features are the only means to identify the original habit and crystal-growth characteristics in a faceted or polished gem.

The internal growth structures represent the succession of crystal “habits” that formed while the crystal was growing (figure 3). (That is, internal...
growth planes were at one point external crystal faces. For consistency, the word plane will be used to refer to internal crystal forms, and the word faces will be reserved for describing external crystal forms. Minerals form over a long period of time—sometimes continuously and sometimes discontinuously—until the conditions of the formation environment can no longer sustain that mineral's growth. This change in conditions can happen for a variety of reasons. For example, the crystal may be removed from the growth environment by geologic forces, or the growth environment no longer has the chemical/physical requirements (composition of fluids, temperature, pressure) to support the continued formation of that mineral, or there may be no more space for expansion, because of competition from other minerals.

The internal structures that indicate the growth of a crystal can be compared to the "rings" of a tree. Just as each "ring" represents one year's growth of tree bark, the internal growth structures illustrate various stages in the history of a crystal's formation. Internal growth features can be observed in transparent gem materials in a variety of ways: as color zoning (which can fluctuate between the different stages of growth); by inclusions that form and concentrate along crystal faces, or coat the crystal's entire surface at one growth stage and then are enclosed by later phases (the latter is how "phantoms" are formed); or by the presence of "lines" that represent the interface or contact plane between consecutive stages of growth. These lines generally are visible with a microscope only when the interior of the gemstone is viewed in a direction parallel, or nearly parallel, to the traces of a specific plane, particularly if the stone is immersed in a liquid of similar refractive index (to reduce the reflection of light).

The appearance of these lines can be visualized by imagining a common type of window in which two sheets of glass have been vacuum sealed together. When you look perpendicular to the glass plane (i.e., as you would normally look through a window), the objects on the other side are not obstructed. However, if you examine the glass parallel to its main surface (i.e., along the edge), a "line" is visible where the two sheets of glass meet.

In 1985, Dr. Karl Schmetzer published his first paper dealing with the methods for determining internal growth structures in some uniaxial gemstones, with specific focus on corundum, beryl, and quartz (see also Schmetzer, 1986a). For a more in-depth discussion on methods and applications of determining growth structures in some uniaxial gemstones, the reader is referred to these papers and others by Schmetzer (1985, 1986a and b), Kiefert and Schmetzer (1991a-c), and Peretti et al. (1995). Following is a brief, step-by-step description of how to use this technique; it has been written specifically for those gemologists, like the author, who do not have a formal background in crystallography.

The Technique. The manner in which specific crystal planes can be identified relates to: (1) the position, or the angle, that the planes form relative to the optic-axis direction of the uniaxial gemstone, and (2) the angle created by the joining of two planes. In the technique used by the author to iden-
tify crystal planes, the equipment includes: a stone holder, modified by the author, which allows for rotation about both the vertical and horizontal axes (Box A); a horizontal microscope; an immersion cell containing methylene iodide; and a light source located behind the gemstone so light is transmitted through it (figure 4). Note that growth structures, color zoning, and the like, can also be seen, but not measured, with a standard binocular microscope using darkfield and various other illumination techniques. However, immersing the gemstone in a liquid will reduce the distortion of light and other detrimental effects, thereby significantly improving the visibility (and measurability) of the growth structures. The recommended procedure is as follows:

First examine each specimen with magnification in the immersion liquid to locate any color banding or other structural features that might be present. Then position the stone in the stone holder so that the direction of the growth planes selected for identification is oriented vertically, that is, perpendicular to the platform base that holds the immersion cell, thus allowing for the most accurate measurement of the angle between the optic-axis direction and the growth plane to be identified. The author has added this first step to the methods described by Schmetzer (1985, 1986a and b) and Kiefert and Schmetzer (1991a-c), in order to guarantee the most accurate measurement for those just beginning to use this testing method. When the growth planes are not in a vertical direction, but rather are at an angle, the angles measured may be off by a couple of degrees, thereby decreasing the accuracy of the technique.

Next, center the optic axis (c-axis) of the gemstone both horizontally and vertically, parallel to the observer’s field of view. This is accomplished by using a pair of crossed polarizing filters and the dual-axis stone holder, which allows for a pivotal tilting both forward and backward, as well as a 360° rotation. By pivoting and rotating the stone holder (and the gemstone), look for the interference rings to converge toward the center of the stone (figure 5). At a certain point, you will notice the interference rings shift from converging to diverging. This indicates that the optic axis of the stone has just been “passed over.” Here, make smaller adjustments to center the stone in that small area between where the interference rings converge and diverge.

During the final centering of the optic axis, very subtle movements left to right and backward to forward will cause the gemstone to go from white to dark to white again (whereas more exaggerated movements will again display the interference rings). When the optic axis is centered, the gemstone should appear essentially black. It may take some practice to become familiar with this procedure. Nevertheless, the accurate centering of the optic axis is essential to the correct identification of the growth planes to be measured.

Figure 3. Internal growth structures reveal the history of a gem’s formation. Each internal growth plane was a face on the original crystal as it grew. Changes in the formation environment can change the presence and prominence of the various faces, providing clues to the origin of the stone. Often one can see the succession of crystal habits that formed over time. In this idealized example of a ruby from Mogh Hszi (Myanmar), the optic axis is located parallel to the length of the stone, and the small, horizontal, basal growth planes are positioned down the middle. An equal amount of growth-structure information is present to the left and right, revealing a habit of c, n, and w. Note that most gemstones do not reveal such a well-centered sequence of growth structures; typically, one side is either less complete than the other or absent. Immersion, magnified 10x.
BOX A: THE MODIFIED STONE HOLDER AND ITS BENEFITS

Under optimal measuring conditions, the "optic axis" method has an accuracy of approximately a 1° (Schmetzer, 1985, 1986a; Kiefert and Schmetzer, 1991a). These optimal conditions are present when the stone holder can be maintained in an essentially vertical position, so that the centered optic axis remains parallel to the observer’s field of view (along a horizontal rotation plane), even after a 180° turn of the stone and stone holder. However, because the ideal orientation of a faceted gemstone is usually random, the observer commonly must tilt the stone holder either forward or backward to center the optic axis. It has been proposed that the observer can tilt the stone holder up to approximately 40° and still get accurate readings (Kiefert and Schmetzer, 1991a). In the author’s experience, however, centering the optic axis frequently requires that the stone holder be tilted to this degree or even further. Yet, on a 180° rotation or even a portion thereof, the optic-axis direction does not remain parallel along the horizontal rotation plane (figure A-1a). This increases the error factor and makes the determination of individual crystal planes more difficult.

The author’s modification to the basic stone holder consists of two independently rotating, slightly curved, grooved channels (figure A-3). These channels allow for an additional rotation axis about the vertical plane. Therefore, the observer does not have to tilt the stone holder to large degrees, or reposition the gemstone in the stone holder, to center the optic axis of certain gemstones. Instead, he or she can rotate the gemstone itself by means of the channels, so that the c-axis maintains a parallel rotation about the horizontal plane and the field of view (figure A-1b). With this new rotation capability, the operator can correct major deviations, leaving the “fine tuning” adjustments of the tilting movement. It should not be necessary for the tilting movement to deviate more than approximately 5° either forward or backward from the vertical position, thereby always maintaining optimal measurement conditions. In addition, after locating the optic axis, the operator can now turn the gemstone 90° along the vertical rotation axis, to view the growth structures perpendicular to the c-axis (again refer to figure A-3) without removing the gemstone and repositioning it in the stone holder.

Figure A-2. The author’s modification to the basic stone holder consists of two independently rotating, slightly curved, grooved channels, which allow for an additional rotation capability so that the c-axis remains in a vertical position. Photo by Shane P. McClure.
A simple method to check whether the optic axis is centered involves holding a jeweler’s loupe between the microscope and the immersion cell. If the gemstone is centered properly, the loupe will act as a conoscopic lens (similar to the glass sphere used with a polariscope), revealing the uniaxial optic figure.

Once the optic axis is centered, remove the polarizing filters and use the set screw to position the pointer of the stone holder at the 0° mark on the indicator dial (figure 6). Then rotate the stone and stone holder (left or right) until a vertical series of growth features is sharply delineated, often by color zoning (figure 7). The position of the pointer on the dial will show how many degrees these growth features are located from the optic-axis direction (figure 8). This number defines which crystal plane is represented by the growth features being examined (see table 1) for the crystal planes seen in corundum and their corresponding angles from the c-axis. The prism planes a (1120) are seen at 0° (because they are parallel to the optic axis), whereas the basal pinacoid c (0001), which is perpendicular to the optic axis, has the largest potential reading, 90° (again, see table 1).

All other crystal planes are located at angles between these two. Typically, a gemstone contains more than one series of growth planes. When complex growth structures are encountered, it may be helpful to center the optic axis first, and then rotate the stone holder as previously described to identify which growth planes are present.

Connecting growth planes can also be identified using a specially designed eyepiece that attaches to one of the microscope oculars. This eyepiece acts as a mini-goniometer, an instrument used by crystallographers to measure angles between crystal faces, enabling measurement of the angles created by connecting crystal planes within a stone (see, e.g., Kiefert and Schmetzer, 1991a). Contained within the eyepiece are two independently rotating disks, one with two lines intersecting at 90° (creating a cross) and the other consisting of small numbered marks around the periphery, indicating 360° in a complete circle. By lining up the longer “cross hair” parallel to one series of crystal planes and then rotating it so that it is parallel to the other series of crystal planes, one can use the scale around the periphery to determine the number of degrees in the angle created by the meeting of two or more crystal planes. (Author’s note: The eyepiece described here is no longer commercially available from the Leica Corp. The reader is therefore referred to Kiefert and Schmetzer, 1991a, for a description of commercially available microscope oculars with “cross hairs” and an additional scale to measure the angles, which is useful in gemological practice.)
Figure 6. Once the optic axis has been successfully centered, before the stone is rotated to observe the growth features, the pointer must be positioned at the 0’ mark on the indicator dial. Photo by Shane F. McClure.

Figure 7. One of the most obvious ways to identify sharpened growth structures is through color zones, or bands, the thickness of which may fluctuate between consecutive periods of crystal growth. Shown here are broad blue color bands alternating with narrow colorless bands, parallel to dipyramidal (4483) planes, in a sapphire from Kashmir. The vertical growth planes are at 15.4° from the optic axis direction, and the angle ceased at the junction of the v-v planes is 123°. Immersion, magnified 15x.

Figure 8. By rotating the stone holder until the growth features are sharply defined, the pointer will show the number of degrees, on the indicator dial, that the growth features are located from the optic-axis direction of the gemstone. The measurement of 20 shown here identifies a series of dipyramidal (1 121) planes, which are located characteristically at 20.1° from the optic axis direction. Photo by Shane F. McClure.

In contrast to the first (“optic axis”) method, this second (“connecting planes”) procedure does not depend on finding the optic-axis direction of the gemstone. However, it is recommended that less experienced practitioners locate and identify at least one series of growth planes initially by the optic-axis method.) What is critical to the accurate measurement of the angles is that both series of growth planes and the points at which they meet are very sharp. With our knowledge of the exact angles formed by various connecting crystal planes [see table 3 for the “interfacial” crystal angles for corundum], once one set of crystal planes has been identified, connecting crystal planes can be identified without requiring reorientation of the stone to the external eyepiece tube of the microscope.

Table 1. The primary crystal planes and angles encountered in natural and synthetic corundum.

<table>
<thead>
<tr>
<th>Crystal plane</th>
<th>Designation</th>
<th>Indices</th>
<th>a (angle from c-axis)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Second-order hexagonal prism</td>
<td>a*</td>
<td>(1130)</td>
<td>0°</td>
</tr>
<tr>
<td>Second-order hexagonal prism</td>
<td>b*</td>
<td>(14 16 38)</td>
<td>4.5°</td>
</tr>
<tr>
<td>Hexagonal dipyramids</td>
<td>c*</td>
<td>(2201)</td>
<td>10.4°</td>
</tr>
<tr>
<td>Dipyramids</td>
<td>d*</td>
<td>(4460)</td>
<td>15.4°</td>
</tr>
<tr>
<td>Vb</td>
<td>e*</td>
<td>(1311)</td>
<td>20°</td>
</tr>
<tr>
<td>Wb</td>
<td>f</td>
<td>(2210)</td>
<td>20.8°</td>
</tr>
<tr>
<td>Positive rhombohedron</td>
<td>g</td>
<td>(1011)</td>
<td>22.4°</td>
</tr>
<tr>
<td>Negative rhombohedron</td>
<td>h</td>
<td>(0172)</td>
<td>51.8°</td>
</tr>
<tr>
<td>Negative rhombohedron</td>
<td>i</td>
<td>(9115)</td>
<td>72.5°</td>
</tr>
<tr>
<td>Basal pinacoid</td>
<td>j</td>
<td>(0001)</td>
<td>90°</td>
</tr>
</tbody>
</table>

Note: a* = hexagonal crystal class h = 3.68. Adapted from Kienle and Schmetzer, 1991. More typically seen in synthetic corundum. More typically seen in synthetic corundum.
Dipyramidal

Figure 9. A second method of identifying internal growth structures uses a special eyepiece that is placed on one of the microscope oculars. It has a 360° scale around the periphery, and cross-hairs with which to measure the angle from one growth plane to a connecting plane—that is, the angle formed by the intersection of the two planes. In this illustration, the vertical lines have been identified, by the first (optic-axis) method described, as dipyramidal n (2243) planes, with a measurement of 28.8° from the optic-axis direction (see table 1). By determining the number of degrees between the n planes and the connecting planes, one can identify the remaining growth planes as positive rhombohedral (1011) planes, with an angle of 154°, and a second series of dipyramidal n planes, with an angle of 128° (see table 2).

optic axis (figure 9). When used in combination, the two methods provide a cross-check system. During growth-structure analysis, a helpful method to study the crystal forms is to examine the gemstone perpendicular to the c-axis, that is, parallel to the basal growth structures (again, see figure 3). This is also the best way to determine changes in “habit” that took place during the growth of the original crystal. For a more complete description of this procedure, the reader is referred to Peretti et al. (1995, pp. 8–9).

THE NEGATIVE d PLANE IN NATURAL RUBY

Background. The study and determination of the crystal faces and habits for corundum is certainly not new to crystallography. For the values of α, see table 1.

<table>
<thead>
<tr>
<th>Crystal planes</th>
<th>Angle (°)</th>
<th>Crystal planes</th>
<th>Angle (°)</th>
</tr>
</thead>
<tbody>
<tr>
<td>a-a</td>
<td>128.0°</td>
<td>a-a</td>
<td>147.0°</td>
</tr>
<tr>
<td>z-z</td>
<td>121.1°</td>
<td>c-z</td>
<td>122.4°</td>
</tr>
<tr>
<td>u-u</td>
<td>124.0°</td>
<td>c-u</td>
<td>121.1°</td>
</tr>
<tr>
<td>v-v</td>
<td>123.0°</td>
<td>c-v</td>
<td>122.4°</td>
</tr>
<tr>
<td>w-w</td>
<td>121.1°</td>
<td>c-w</td>
<td>122.4°</td>
</tr>
<tr>
<td>n-n</td>
<td>128.0°</td>
<td>c-n</td>
<td>119.1°</td>
</tr>
<tr>
<td>r-r</td>
<td>86.1°</td>
<td>c-r</td>
<td>148.0°</td>
</tr>
<tr>
<td>r-u</td>
<td>154.0°</td>
<td>c-u</td>
<td>154.0°</td>
</tr>
<tr>
<td>r-v</td>
<td>148.0°</td>
<td>c-v</td>
<td>174.4°</td>
</tr>
<tr>
<td>r-w</td>
<td>152.0°</td>
<td>c-w</td>
<td>174.4°</td>
</tr>
<tr>
<td>r-n</td>
<td>148.0°</td>
<td>c-n</td>
<td>174.4°</td>
</tr>
<tr>
<td>r-d</td>
<td>133.0°</td>
<td>c-d</td>
<td>174.4°</td>
</tr>
</tbody>
</table>

For the values of α, see table 1.

TABLE 2. The angles formed by the meeting of two crystal planes in natural and synthetic corundum.

sources, the reader is referred to Victor Goldschmidt’s Atlas der Krystallformen, published in 1918. Over the past decade, however, this analysis has taken the additional dimension of aiding in the separation of natural and synthetic corundums of various colors (Schmetzer, 1985, 1986a and b, 1987; Kiefert and Schmetzer, 1986, 1988, 1991c; Smith, 1992; Smith and Bosshart, 1993; Hänni et al., 1994; Schmetzer et al., 1994). Because consistent and reproducible crystal synthesis requires a controlled growth environment, most synthetic corundums display crystal habits that are typical of the specific methods (and uniform conditions) by which they are manufactured. In contrast, as a result of inconsistencies and fluctuations in the geologic conditions present during their formation, natural corundums display a much wider variety of crystal habits.

The structural differences between natural and synthetic corundums may be seen in a variety of attributes, including the presence or prominence of certain crystal planes, color-zoning characteristics, habit variations, combinations of crystal planes, and systems of twin lamellae. Of the crystal planes observed in flux-grown synthetic corundums produced by the various manufacturers (such as Chatham, Dourous, Rashin, Knisichka, and Ramaura), the negative rhombohedral d (0112) plane was found to be present with a frequency and prominence that has not been observed in natural corundums (Schmetzer, 1985, 1986a and b, Kiefert and Schmetzer, 1991a–c, Hänni et al., 1994). The negative d plane is positioned at 51.8° (again, see table 1) from the optic-axis direction. The negative d face is located at the terminations (or ends) of a
corundum crystal, extending from the basal pinacoid c (0001), between two dipyramidal or prism faces, and opposite a positive rhombohedral r (011) face (figure 10). This crystal face has been reported historically in natural corundum (Bauer, 1896; Dana and Dana, 1904; Goldschmidt, 1918; Bauer and Schlossmacher, 1932). However, it appears that Max Bauer (1896) is the only source where a ruby crystal with a negative rhombohedral d (0112) face was actually examined and illustrated. The other references appear to be citing Bauer (1896) as their source of the information. In the more modern literature, the negative d face has been described as representing a subordinate crystal form that, if present in natural corundum, would be very small (Schmetzer, 1986a). However, no references can be found in the modern literature to the observation of a negative rhombohedral d (0112) plane on or in a natural ruby. This has led to the suggestion that the presence of a more dominant negative d plane, which is commonly found in synthetic ruby, can be used as an indication of synthetic origin (Schmetzer, 1985, 1986a and b; Kiefert and Schmetzer, 1991a-c).

As part of the gemological examination of several hundred rubies, the author used internal growth-structure analysis, incorporating the combination of both methods described previously. These analyses revealed the presence of subordinate and, in some cases, dominant negative rhombohedral d (0112) planes in rubies that other tests proved were natural.

Materials and Methods. A total of eight natural rubies, between 0.15 ct and more than 25 ct, were identified with the negative rhombohedral d (0112) crystal plane as part of their original habit (table 3). Two had been purchased in northern Vietnam by Dr. Eduard J. Gübelin (Lucerne, Switzerland) and Mr. Saverio Repetto (previously director of FIN-GEMS, Chiasso, Switzerland)—1.34 and 0.15 ct, respectively—and had been represented to them as originating from the Lục Yên mining region. Two others (7.03 and 25.55 ct) were reportedly from the Mogok Stone Tract in Myanmar, according to the clients who submitted them to the Gübelin Gemmological Laboratory for testing. The remaining four were also submitted to the Gübelin Gemmological Laboratory for examination, but without any indication as to their source.

A standard refractometer, desk-model spectroscope, and electronic scale equipped with the necessary attachments for hydrostatic specific gravity measurements were used to determine that the eight samples were ruby. As part of the routine examination, as well as once the negative d planes were identified, extensive tests were conducted to confirm that the stones were indeed natural. To
establish their natural origin by means of their internal features, a binocular microscope equipped with a dark-field light source was used in conjunction with fiber-optic lighting. Both of the methods described above were used to analyze the internal growth structures and twinning characteristics. Semi-quantitative chemical analysis was performed using a Spectrace TN5000 energy-dispersive X-ray fluorescence (EDXRF) spectrometer with a special measuring routine for corundum developed by Professor W. B. Stem of the University of Basel.

**Results.** The refractive indexes ($n_p = 1.760-1.764$, $n_o = 1.769-1.772$; birefringence of 0.008-0.009; visible range spectra (Cr²⁺ absorption bands), and specific gravity [3.98–3.99]) were consistent with known properties for ruby. In addition, each of the stones had a combination of inclusion features, twinning, habit, and trace-element concentrations that provided proof of their natural origin (again, see table 3).

Among the natural inclusion features observed in these rubies were transparent, colorless, and whitish mineral inclusions; unaltered and thermally altered healed fractures; zones of dense, unaltered rutile needles (figure 11); and rutile needles that were broken as a result of heat treatment. Also noted were very fine-grained whitish clouds, “cross-hatch” or “flake-like” inclusion patterns, as well as “antennae-like” intersecting stringer formations (figure 12) and long, fine, needle-like tubules along the intersection of two or more systems of twin lamellae (commonly referred to as “boehmite needles”). When the internal growth structures provided enough information to recreate the crystal habit, typically two or more dipymidal crystal and/or prismatic planes, combined with the basal and rhombohedral crystal planes, were present (figure 13, see also figure 10). The combinations of these inclusion features and internal growth structures alone provided sufficient evidence of the natural origin of the rubies described.

**Observation of the Negative Rhombohedron $d$ Plane.** In the eight natural rubies described in this article, the negative $d$ planes ranged from subordinate through dominant, sometimes within the same stone. The negative $d$ plane in the 1.34 ct ruby varied slightly in prominence from subordinate to intermediate during the growth of the gem (figure 14). This ruby also had dipymidal $w$ planes, dipymidal $z$ planes (of varying size), positive rhombohedral $r$, and basal pinacoid $c$ planes.

<table>
<thead>
<tr>
<th>TABLE 3. Properties and internal characteristics of the eight natural rubies with the $d$ (0112) plane.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Property/ Characteristic</strong></td>
</tr>
<tr>
<td>-----------------------------</td>
</tr>
<tr>
<td><strong>Carat weight</strong></td>
</tr>
<tr>
<td><strong>Inclusions</strong></td>
</tr>
<tr>
<td><strong>Evidence of heat treatment</strong></td>
</tr>
<tr>
<td><strong>Twinning parallel to $r$</strong></td>
</tr>
<tr>
<td><strong>Growth structures</strong></td>
</tr>
<tr>
<td><strong>Chemistry</strong></td>
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</tbody>
</table>

**Internal Growth Structures**
The 25.55 ct ruby displayed dominant c planes and small negative d planes, which were present during the earlier stages of crystal growth (figure 15), as well as subordinate n planes and more dominant dipyramidal planes or v.

Negative d planes and positive r planes of approximately equal, intermediate size were recorded in the 0.15 ct ruby (figure 16), along with subordinate z planes.

In the most striking examples, three rubies had dominant negative d planes. The 7.63 ct stone showed only dominant c planes along with the dominant negative d planes (figure 17). In addition to its dominant negative d planes (figure 18), the 10.07 ct ruby had dominant positive r planes and two systems of twin lamellae, along with dominant c and a planes, with more subordinate n planes. Yet another sample (6.10 ct), revealed negative d planes that ranged from subordinate to dominant (figures 19 and 20), and also displayed c, positive r, and two series of dipyramidal—n and z—planes (refer to figure 13).

A subordinate negative d plane was present during most, but not all, of the growth observed in the 2.53 ct ruby. The dipyramidal planes or v dominated the morphology of this gemstone, with subordinate c, z, and positive r planes also present.

The 4.51 ct ruby had c, positive r, and two different dipyramidal—n and z—planes, with subordinate negative d planes having formed only during a brief period of crystal growth (refer again to figure 10).

The 4.51 ct stone was natural. Oblique fiber-optic illumination, magnified 23x.
Figure 14. The negative d planes in this 1.34 ct Vietnamese ruby varied in size slightly during the stages of growth that are visible in the faceted stone, but for the most part they were subordinate. The brownish bands following the growth structures are concentrations of short rutile needles. Also notice the sharp horizontal line, which is a twin plane parallel to a positive r plane. Immersion, magnified 15x.

DISCUSSION

Since this author first identified the negative rhombohedral d (0112) plane in a natural ruby (Smith, 1992), he has identified this crystal plane in other samples. Statistically, however, this structural feature remains very rare, having been observed in less than 1% of the several hundred corundum samples (primarily ruby and blue sapphire) measured to date by the author. The term corundum is used in this context, because the author did identify the negative d plane in a natural blue sapphire loaned by Dr. H. A. Hanni, of the Swiss Gemological Institute (SSEF), Basel; this stone also reportedly originated from northern Vietnam. Determining the conditions under which the negative d plane formed in these samples is beyond the scope of this research; we can only surmise that they must have been very special.

To use the presence of the negative d plane to separate natural and synthetic rubies, careful interpretation of the growth features present in para-

mount. The negative d plane (especially as a dominant feature) remains more common in flux-grown synthetic rubies than in natural rubies. However, the entire collection of structural features should always be used in combination with a careful analysis of the other inclusion features, as well as with the information learned from a chemical or possibly infrared spectral analysis. Whereas the basal pinacoid c and the positive rhombohedral r planes are typically seen in both natural and synthetic rubies, n is the only dipyramid seen in synthetic rubies. Natural rubies typically contain one or any combination of a, o, v, z, v, w, or n.

Aside from the various inclusion features present, it was possible to identify seven of the eight
rubies in the sample population (the exception was the 7.03 ct stone) as natural by the presence of internal growth structures (again, see table 3) that have not been seen in flux-grown synthetics. Consequently, if a negative d plane is seen in a ruby, then it is important to look also for the hexagonal dipyramidal and prism crystal planes. If, in addition to the negative d plane, only c, positive r, and/or n are observed, care must be taken: Although this suite of features is typical for flux-grown synthetic rubies, it may also occur in natural rubies. If no characteristic inclusions are present, the use of analytical techniques such as EDXRF chemical analysis (see, e.g., Stern and Hann, 1982) or infrared spectroscopy (see, e.g., Smith, 1995) is advised. If additional dipyramidal or prism planes are present, then natural ruby is indicated. In addition, the negative rhombohedral γ (0115) plane, located at 72.5° from the optic axis, is still considered indicative of a synthetic ruby in those very rare situations where it is present (Schmetzer, 1985, 1986a and b; Kiefert and Schmetzer, 1991a-c). If a negative d plane is detected in a ruby of suspicious identity, then twinning characteristics can also provide important clues (Kiefert and Schmetzer,
Figure 19. The negative d planes in this 6.10 ct natural ruby ranged from subordinate to dominant. This view shows a subordinate form of negative d plane with dominant c planes. Immersion, magnified 10x.

Figure 20. The 6.10 ct ruby also displayed dominant negative d planes as the stone was rotated to view additional growth-structure characteristics. Immersion, magnified 8x.

CONCLUSION

The analysis of internal growth structures (internal indicators of crystal habit formation) in fashioned gemstones has been increasing in importance since it was first introduced to gemologists in 1985. The methods and principles involved can be learned and used by any gemologist with a minimum of specialized equipment. With experience, the study of growth structures—through the identification of individual and pairs of crystal planes—can help distinguish between fashioned natural and synthetic gems as well as help classify gemstones from various deposits. While this article focuses on the identification of growth planes, observations of color zoning and twinning characteristics can also provide key information relating to a natural/synthetic distinction or provenance determination (see, e.g., Schmetzer, 1987; Smith and Surdez, 1994; Peretti et al., 1995; Smith et al., 1995; Hanni et al., 1994).

According to earlier reports, the negative rhombohedral d (0112) plane was extremely rare in natural rubies and occurred only as a subordinate feature. However, this article has detailed for the first time in the professional literature the occurrence of negative d planes as intermediate to dominant crystal planes in natural rubies. It is potentially relevant to locality determination that to date the negative d plane has been observed only in rubies from Vietnam and Myanmar. These findings reinforce the importance of not relying on any single growth feature as conclusive proof of the natural or synthetic origin of a ruby or sapphire. Instead, identification and analysis of growth structures requires keen observation and interpretation of all the observed crystallographic features.
REFERENCES


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GOTu7
Synthetic alexandrite is being flux-grown in Russia in a molybdenum-, bismuth-, and germanium-bearing solvent by means of the reverse-temperature-gradient method. Characteristic properties include habit, twinning, growth patterns, residual flux inclusions, trace-element contents, chemical zoning, color zoning, and spectroscopic features in the visible and infrared ranges. The relationship between production technique and characteristic properties is discussed, and diagnostic properties that can be used to distinguish these synthetics from natural alexandrites are disclosed.

Fine alexandrites are even rarer than fine rubies, sapphires, and emeralds. The major factors determining price are size, clarity, brilliancy, color in daylight, color change in artificial light, and country of origin. The finest alexandrites are characterized by a lively and intense green color in daylight with a prominent color change to purplish red or reddish purple in incandescent light. The most prestigious source of natural alexandrites is Russia, specifically the Ural mountains, where they were first discovered. Fine alexandrites occasionally appear at international auctions. For example, a 31 ct alexandrite sold for almost US$185,000 at Christie’s May 1992 auction in Geneva. Dealers in Matroseein report the sale of fine, large (over 5 ct) alexandrites for $10,000-$20,000 per carat, and even higher for exceptional stones (R. Guebritz and A. Wild, pers. comm., 1996).

During the past 10 years, new deposits of natural alexandrites have been found in Minas Gerais, Brazil (Bank et al., 1987; Proctor, 1988; Casedenane and Roditi, 1993; Kofken and Wegner, 1993); in Orissa and Madhya Pradesh, India (Parsaik and Nayak, 1993; Newlay and Pashine, 1993); and, most recently, near Songea in southern Tanzania (see “News on the Songea Deposit . . . ”, 1995; Kammerling et al., 1995). In addition, gem alexandrites are still being recovered from the historic emerald deposits of the Ural Mountains (Eiczi and Krymko, 1994; Laskovkova and Zhernakov, 1995). As greater quantities of gem alexandrite enter the market, significant amounts of faceted alexandrites are being submitted to gemological laboratories for testing. Occasionally, distinguishing natural from synthetic has been difficult, notably for flux-grown synthetics, but especially when a specimen lacks diagnostic mineral inclusions (see Bank et al., 1988; Henn and Bank, 1992, Kammerling, 1995). These difficulties are caused by the similarity of growth structures and healing “feathers” in natural alexandrites from different localities to residual flux feathers in flux-grown synthetic alexandrites. In addition, hematite platelets in natural alexandrites sometimes resemble platinum inclusions in their flux-grown synthetic counterparts.
The Russian-produced synthetic alexandrites (figure 1) can imitate the highest-quality natural alexandrites—such as those from the Urals mountains. Yet some natural alexandrites have sold for up to 300 times as much as their synthetic counterparts. Consequently, those synthetic alexandrites occasionally are misrepresented in the trade as Uralsian alexandrites. One of the authors [A.P.] recently encountered two instances of synthetic alexandrites offered for sale as natural stones in Switzerland.

According to the voluminous scientific and patent literature available to the authors, synthetic alexandrite and synthetic chrysoberyl can be grown from the melt (Czochralski, Verneuil, Bridgman, and floating-zone techniques), hydrothermally, by the flux method, and by chemical vapor deposition (for a good description of most of these modern crystal-growth techniques, see Kimura and Kitamura, 1993). Because of such factors as production costs and the small crystals that result from some of these growth techniques, gem-quality synthetic alexandrites have been grown commercially and released to the market by just a few companies, which use the Czochralski, floating-zone, or flux method.

Two types of flux-grown synthetic alexandrite have been produced commercially for gem purposes. Since the 1970s, Creative Crystals Inc. of San Ramon, California, has produced synthetic alexandrite crystals by a method that uses a lithium polymolybdate flux, as described in the Cline and Patterson patent (1975).

The second type has been produced at various locations in the former USSR since the late 1970s or early 1980s, using a method originally developed at the Institute of Geology and Geophysics, Siberian Branch of the Academy of Sciences of USSR in Novosibirsk. A few crystals grown by this method were given to one of the authors [K.S.] by Drs. A. Ya. Rodionov and A. S. Lebedev in 1988 and 1991. Preliminary results from the examinations of these samples were never published because of the very small sample base. That situation has changed. Considerable quantities (many kilograms) of rough
Russian synthetic alexandrite are now commercially available, either directly from Novosibirsk or indirectly from Bangkok, and large parcels of rough are being cut in Sri Lanka, especially for the American market (E. E. Zoya, pers. comm., 1995). For the present study, the authors examined more than 200 synthetic alexandrites with properties that indicated that they were all produced by roughly the same method (i.e., using fluxes that were similar in composition). In addition, one commercial source of Russian flux-grown synthetic alexandrite is the Design Technological Institute of Monocrystals, also in Novosibirsk, which works in cooperation with the Institute of Geology and Geophysics. During a 1994 visit to Novosibirsk, one of the authors (A.P.) obtained information first hand at both institutes and saw demonstrations of part of the growth facilities, including the use of crucibles and furnaces.

The production technique used for Russian flux-grown synthetic alexandrite was briefly mentioned by Godovilzov et al. (1982) and later described in greater detail, by Rodionov and Novgorodtseva (1988) and Bukin (1993). The gemological properties of Russian flux-grown synthetic alexandrites were first described by Trosharelli (1986) and later by Henn et al. (1998), Henn (1992), and Hodgkinson (1995). The present article examines the relationship between production technique and typical properties, and identifies those characteristics that can be used to distinguish these synthetics from natural alexandrite.

### TABLE 1. Modern techniques for the flux growth of chrysoberyl and alexandrite.

<table>
<thead>
<tr>
<th>Authors</th>
<th>Inventions assigned to</th>
<th>Flux</th>
<th>Growth conditions</th>
<th>Dopant</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Farrell and Fang (1964)</td>
<td>PbO-PbF&lt;sub&gt;2&lt;/sub&gt;, PbO</td>
<td>s, sc</td>
<td>Cr</td>
<td>Chrysoberyl</td>
<td></td>
</tr>
<tr>
<td>Farrell and Fang (1965)</td>
<td>PbO-PbF&lt;sub&gt;2&lt;/sub&gt;, PbO</td>
<td>s, sc</td>
<td>Chrysoberyl</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tabata et al. (1974)</td>
<td>PbO-PbF&lt;sub&gt;2&lt;/sub&gt;, PbO</td>
<td>s, sc</td>
<td>Cr, Fe</td>
<td>Alexandrite</td>
<td></td>
</tr>
<tr>
<td>Godovilzov et al. (1982)</td>
<td>PbO-PbF&lt;sub&gt;2&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Cr, V</td>
<td>Alexandrite</td>
<td></td>
</tr>
<tr>
<td>Kurosawa and Nozorik</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s</td>
<td>Fe</td>
<td>Alexandrite</td>
<td></td>
</tr>
<tr>
<td>Takayama and Nakata (1995)</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Fe, Cr, Ni, Co</td>
<td>Alexandrite</td>
<td></td>
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<tr>
<td>Bulan (1996)</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Cr, Fe</td>
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</table>

* s = slow evaporation, s = seeded growth, c = constant growth, f = float zone, n = normal growth, sc = spontaneous crystallization, and t = temperature gradient.

The production technique used for Russian flux-grown synthetic alexandrite was briefly mentioned by Godovilzov et al. (1982) and later described, in greater detail, by Rodionov and Novgorodtseva (1988) and Bukin (1993). The gemological properties of Russian flux-grown synthetic alexandrites were first described by Trosharelli (1986) and later by Henn et al. (1998), Henn (1992), and Hodgkinson (1995). The present article examines the relationship between production technique and typical properties, and identifies those characteristics that can be used to distinguish these synthetics from natural alexandrite.

### TABLE 1. Modern techniques for the flux growth of chrysoberyl and alexandrite.

<table>
<thead>
<tr>
<th>Authors</th>
<th>Inventions assigned to</th>
<th>Flux</th>
<th>Growth conditions</th>
<th>Dopant</th>
<th>Remarks</th>
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<td>Godovilzov et al. (1982)</td>
<td>PbO-PbF&lt;sub&gt;2&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Cr, V</td>
<td>Alexandrite</td>
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<tr>
<td>Kurosawa and Nozorik</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s</td>
<td>Fe</td>
<td>Alexandrite</td>
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<tr>
<td>Takayama and Nakata (1995)</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Fe, Cr, Ni, Co</td>
<td>Alexandrite</td>
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<tr>
<td>Bulan (1996)</td>
<td>Li&lt;sub&gt;2&lt;/sub&gt;MoO&lt;sub&gt;4&lt;/sub&gt;</td>
<td>s, sc</td>
<td>Cr, Fe</td>
<td>Alexandrite</td>
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</table>

* s = slow evaporation, s = seeded growth, c = constant growth, f = float zone, n = normal growth, sc = spontaneous crystallization, and t = temperature gradient.
Figure 2. Three main processes have been described for the flux growth of beryllium-bearing oxides and silicates such as synthetic alexandrite and emerald: (A) slow cooling of a saturated melt, with seeded growth or growth by spontaneous nucleation; (B) seeded growth in a temperature gradient, with a growth zone above the dissolution zone; and (C) seeded growth or growth by spontaneous nucleation in a reverse temperature gradient, with a growth zone at the bottom of the crucible below the dissolution zone. Key: 1 = platinum crucible, 2 = flux melt, 3 = growing crystals, 4 = seed, 5 = platinum seed holder, 6 = nutrient, 7 = baffle, 8 = insulation, 9 = temperature in the upper part of the crucible, 10 = temperature in the lower part of the crucible. The circular arrows at the top of A and B indicate the possible rotation of the seed holder; arrows at the bottom of all three processes represent the possible crucible rotation. Adapted from Bukin (1993).

FLUX GROWTH OF SYNTHETIC ALEXANDRITE: HISTORY AND DEVELOPMENT

Early 19th-century experiments in the flux growth of synthetic chrysoberyl were summarized by Elwell and Scheel (1975). More modern flux techniques to grow synthetic chrysoberyl and/or alexandrite started in the 1960s (table 1). Most procedures use the slow-cooling technique (figure 2A). First, the solvent of flux and nutrient (Al₂O₃ + BeO + color-causing dopants) is heated above the point at which the flux liquifies and is then held at that temperature for a period sufficient to dissolve the nutrient oxides in the flux (Farrell and Fang 1964). Next, the melt is cooled slowly at a constant rate until the flux solidifies. Last, the crystals that grew in the flux during cooling are removed from the crucible by dissolving the flux (e.g., in hot nitric acid). Common fluxes used for chrysoberyl and alexandrite are PbO-PbF₂, Li₂MoO₄-MoO₃, and V₂O₅ (see table 1). The temperatures at which the cooling process begins and ends vary according to the composition of the flux used. They generally range from 1350°C to 800°C. Cooling rates are usually between 0.125°C and 3°C per hour. Both seeded growth and growth by spontaneous nucleation are used (again, see figure 2A). Seed crystals may be natural or synthetic chrysoberyl or alexandrite. Cr₂O₃, Fe₂O₃, and/or V₂O₅ are used as dopants.

The morphology of synthetic chrysoberyl or alexandrite depends primarily on the composition of the flux and not on the temperatures or cooling gradients used. Tabata et al. (1974) demonstrated the influence of BeO on the habit of chrysoberyl grown from PbO-PbF₂ solvents: They observed a distinct modification of habit from platy (flux without BeO) to prismatic or equi-dimensional (flux with BeO). Isolated attempts to grow synthetic chrysoberyl or alexandrite by the flux evaporation technique have had poor results (see Farrell et al., 1963). In this method, constant heating of the melt in open crucibles leads to supersaturation of the melt and the growth of crystals by spontaneous nucleation.

Godovikov et al. (1982) first mentioned the possible growth of alexandrite by the temperature-gradient method, which works by creating a convection current within the crucible. According to a Japanese patent application by Togawa (1985), the nutrient is placed at the bottom of the crucible, where a certain temperature is reached and then maintained. A seed crystal is placed into the melt in the upper part of the crucible, which is held at a temperature lower than that at the bottom of the crucible (figure 2B). As the nutrient dissolves into the solution, circulation begins between the warmer and cooler areas in the crucible (convection currents). When the solution with the nutrient reaches the cooler area, it becomes supersaturated and crystals begin to form. These different temperatures are

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maintained, with the growth zone above the dissolution zone, for weeks or even months.

Rodionov and Novgorodtseva (1988) described a reverse temperature-gradient technique—in which the growth zone is located below the dissolution zone—for the growth of synthetic alexandrite. In this method, the nutrient is placed in the upper part of the crucible, where a certain temperature is reached and then maintained, while the bottom of the crucible is held at a lower temperature (figure 2C; temperatures are in the same range as those noted above for the slow-cooling process). With this technique, synthetic alexandrite crystals grow by spontaneous nucleation, usually in contact with the bottom of the crucible (Rodionov and Novgorodtseva, 1988), although seeded growth (with a seed placed at the bottom of the crucible) is also possible (Rodionov and Novgorodtseva, 1988). The solvent used in Russia is a complex bis-molybdenum flux, mainly composed of Bi₂O₃ and MoO₃ (A. Ya. Rodionov, pers. comm., 1988; Bukin 1993; confirmed during a visit by one of the authors [A.P.] to Novosibirsk in 1994).

The basic growth technique that was developed in Novosibirsk forms synthetic alexandrite crystals with two different habits—thin and platy or more isometric and equidimensional—depending on controlled change in the growth conditions (Rodionov and Novgorodtseva, 1988). Growth rates of 0.13 to 0.35 mm per day in different crystallographic directions have been obtained. For example, a 14 x 8 x 9 mm crystal was grown by spontaneous nucleation over three months with this technique (Rodionov and Novgorodtseva, 1988). According to Bukin (1993), crystals as large as 2.3 cm (over one inch) can be grown by spontaneous nucleation in a reverse-temperature-gradient, and even larger samples are possible with seeded growth.

MATERIALS AND METHODS

In 1994, one of the authors (A.P.) purchased about 50 synthetic alexandrites that had been flux grown in Russia. These samples were selected in Bangkok from six lots, each of which contained hundreds of synthetic alexandrite crystals and represented thousands of carats of flux-grown material. In addition, R. Goerlitz of Idar-Oberstein, Germany, loaned the authors a lot of about 150 rough crystals that he had purchased in Novosibirsk in 1983. This sample of more than 200 Russian flux-grown synthetic alexandrites used for the present study also included material submitted by Novosibirsk scientists to one of the authors (R.S.) in 1988 and 1991. All of the original samples were rough crystals, the few faceted gems studied (see, e.g., figure 1) were fashioned from rough from the Bangkok and R. Goerlitz lots.

About half of the crystals were fully developed single crystals or cyclic twins with one irregular planar surface (figure 3). Compared to the smooth crystal faces, this irregular surface was somewhat rough and uneven, it probably represents the contact plane of the growing crystal with the bottom of the crucible (see the later discussion of growth conditions). Most of the balance of the samples had one irregular plane or surface that was obviously produced by sawing or breaking, probably to remove some impure, non-gem-quality material or to separate the single crystals or twins from larger clusters. Seven smaller crystals (sizes up to 4 mm) were fully developed without any irregular surface plane.

We performed standard geological testing on about 40 of these crystals. To identify the internal growth planes and the external crystal faces, we studied about 50 crystals with a Schneider horizontal (immersion) microscope, which had a specially designed sample holder as well as specially designed eyepieces (Schmetzer, 1986; Kiefert and Schmetzer, 1991; see also Peretti et al., 1995). In addition, we examined about 10 samples with an optical goniometer (an instrument used to measure crystal angles). By a combination of these methods, we identified all crystal faces and the most characteristic growth patterns. We studied and photographed the inclusions and internal structural features using the Schneider immersion microscope [with Zeiss optics] and an Eickhorst vertical microscope [with Nikon optics] and fiber-optic illumination.
Solid inclusions and solid phases on the surfaces of the crystals were characterized by X-ray powder diffraction analysis with a Gandolfi camera, by a Cambridge Instruments scanning electron microscope with an energy-dispersive X-ray detector (SEM-EDS), and by energy-dispersive X-ray fluorescence analysis (EDXRF) using a Tracer Northern Spectrace TN 5000 system.

We also performed qualitative chemical analysis of 23 samples using the same EDXRF instrumentation. For quantitative analysis of nine other samples, a CAMECA Camebax SX 50 electron microprobe was used. To evaluate nonhomogeneous chemical compositions of the alexandrite crystals, we measured 2-5 traverses (of 40 to 140 point analyses each) across the samples. For more detailed information, we also had one scan with 625 point analyses.

Polarized absorption spectra in the visible and ultraviolet range were recorded for nine microscopically untwinned single crystals with a Leitz-Unicam SP 800 double-beam spectrometer and a Zeiss multichannel spectrometer. Infrared spectroscopy was carried out on 11 samples using a Philips PU 9800 FTIR spectrometer.

RESULTS

Visual Appearance. The samples varied from slightly yellowish green to green and bluish green in daylight and from slightly orangy red to red and purple in incandescent light (again, see figures 1 and 5). No distinct color zoning was apparent in the crystals with one uneven face—and faceted samples.

On the faces of some crystals, we observed a fine-grained white crust. In irregular cavities of other samples, we found a fine-grained gray or yellowish gray material.

Crystallography. All samples examined revealed an equidimensional habit, which was formed by three pinacoids a, b, and c by four different rhombic prisms, designated x, y, z, and k, and by the rhombic dipyramid o (table 2). The seven small crystals that lacked rough or uneven faces were fully developed single crystals (see, e.g., figures 4 and 5).

Of the crystals with one uneven face—and those crystals that were sawn or broken—had three dominant faces: the pinacoid a, the rhombic prism k, and the rhombic dipyramid o. Frequently, the rhombic prism k was also present, and the pinacoid c was subordinate. About 90% of these crystals were cyclic twins (figures 6 and 7), which consisted of three individuals twinned by reflection across the rhombic prism (011) and forming a pseudohexagonal contact twin (figures 6 and 8). The remaining 10% of the samples were untwinned single crystals.

For those crystals that were not broken or sawn, the most frequently observed habit consisted of the a, x, and k faces (figures 6C and D). Also common was a habit formed by the three faces a, x, and o (figure 6A). Crystals with an additional c pinacoid were somewhat rarer (see table 2 and figures 6B and E). Most of the cyclic twins were somewhat distorted; that is, identical crystal faces varied in their respective sizes between the three individuals of the cyclic twin. Examples are shown in figures 6C and D, in which varying sizes were drawn for the rhombic prism x. Consequently, a typical crystal of Russian flux-grown alexandrite is a combination of the two trillings drawn in figures 6C and D, with sizes of the x faces varying within the cyclic twin.

In a few single crystals with one irregular face, the pinacoid b and the rhombic prism m were also observed (see figure 5). In one of those single crystals, an additional rhombic prism p was also present (table 2).

Gemological Properties. Table 3 summarizes the gemological properties of the Russian flux-grown synthetic alexandrites examined. The values are more or less within the ranges published by others for crystals of this material (Trossarelli, 1986; Henn et al., 1988; Rodionov and Novgorodtseva, 1988; Henn, 1990; Blikin, 1993).

Specifically, these Russian synthetic alexandrites are distinctly pleochroic. Their color change is

<table>
<thead>
<tr>
<th>TABLE 2. Morphological crystallography of Russian flux-grown synthetic alexandrites.</th>
<th>Faces</th>
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<th>Type</th>
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<td>pinacoid</td>
<td></td>
<td></td>
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<tr>
<td>b</td>
<td>010</td>
<td>pinacoid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>c</td>
<td>011</td>
<td>pinacoid</td>
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<td></td>
</tr>
<tr>
<td>m</td>
<td>012</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>n</td>
<td>013</td>
<td>rhombic prism</td>
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<tr>
<td>l</td>
<td>110</td>
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<td></td>
</tr>
<tr>
<td>k</td>
<td>021</td>
<td>rhombic prism</td>
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<td></td>
</tr>
<tr>
<td>o</td>
<td>011</td>
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<td>c</td>
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<th>c'</th>
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<td>β</td>
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<tr>
<td>γ</td>
<td>86°</td>
<td>86°</td>
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</tr>
<tr>
<td>α</td>
<td>81.5°</td>
<td>81.5°</td>
<td>81.5°</td>
<td>81.5°</td>
</tr>
</tbody>
</table>

a = c = 141°; b' = c' = 78°; α = 154°; β = 150°; γ = 86°; α' = 81.5°; (α-θ)-thyroid
caused by a change in two of the pleochroic colors—parallel to the a- and b-axes, with the latter making the greater contribution—when illumination is changed from day or fluorescent to incandescent light. The most intense color change was observed for those synthetic alexandrites that revealed a yellowish orange color in daylight and a reddish orange color in incandescent light parallel to the b-axis; a somewhat weaker color change was observed for samples that revealed a pleochroic color of greenish yellow or yellow in daylight and yellowish orange or orangy yellow color in incandescent light parallel to the b-axis (table 3; see also Schmetzer et al., 1980).

Microscopic Characteristics. Structural Properties (Growth Features and Twinning). As mentioned earlier, most of the sample Russian flux-grown synthetic alexandrites were twinned. When examined with polarized light, the three individuals of the trilling and their twin boundaries became clearly visible—especially when the polarizer was rotated (figure 8). The angle between twinned individuals is 59.88° (not quite 60°—Goldschmidt and Preiswerk, 1900; Goldschmidt, 1900), which is why the twin boundaries are not exactly planar faces (figure 9). In some samples, the twin boundary between two individuals of the cyclic twin revealed a microstructure of small inclusions of alexandrite crystals oriented parallel to the third individual of the trilling. Most of the twinned samples revealed a 141.5° re-entrant angle formed by two rhombic prism faces $k$ and $k'$ (figures 6 and 10).
The internal growth features of both the rough and faceted Russian synthetic alexandrites corresponded to the external morphology of the crystals. All single crystals or cyclic twins that showed one irregularly oriented uneven plane—that is, all samples that grew in contact with the crucible—revealed distinct internal growth planes parallel to the four dominant faces a, x, k, and o.

In a view parallel to the crystals’ a-axis (i.e., perpendicular to the a pinacoid), growth planes parallel to different k prism faces were visible in most of the samples (about two out of three; again, see figures 6 and 10). Two characteristic angles were observed in this orientation: an angle of 81.5° formed by two k prism faces of one individual, and a re-entrant angle of 141.5° formed by two k faces of two individuals of the twin (see figure 2). All of these k” characteristic growth structures also were observed in the faceted synthetic alexandrites (figure 11).

In a view parallel to the b-axis (figure 12A), a second characteristic growth pattern ax was observed in all samples. This consists of a pinacoid and x prism faces, which form a 78° angle (figure 13). By rotating the crystal approximately 29° about the a-axis (figure 12B), we saw a third characteristic growth pattern xo. Visible in all samples, it is formed by a pinacoid in combination with dipyramids (figure 14). In this case, the characteristic angle formed by the two rhombic dipyramids of measures 86°. So, three characteristic patterns of growth structures were observed in single crystals and cyclic twins of Russian synthetic alexandrites: ax and xo in all samples, and k” in approximately two-thirds of the samples.

In about 10% of the single crystals and twins examined, we saw a distinct color zoning—an intense red core (in incandescent light) and a lighter red rim—with the microscope. These areas are separated by a somewhat rounded, very intense red boundary (figure 15). This was the only color zoning seen in any of the samples.

In those small single crystals that were obviously grown without contact with the crucible (again,table 3.

<table>
<thead>
<tr>
<th>Property</th>
<th>Day (fluorescent) light</th>
<th>Incandescent light</th>
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<tbody>
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<td>Color</td>
<td>Yellowish green or green or bluish green</td>
<td>Orange red or red or purplish red</td>
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<tr>
<td>Pleochroism</td>
<td>Reddish purple, greenish yellow or yellowish orange</td>
<td>Purple red, orange yellow or yellowish orange or reddish orange</td>
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<td>Y parallel to b-axis</td>
<td>X parallel to a-axis</td>
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<tr>
<td>Z parallel to c-axis</td>
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<tr>
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<tr>
<td></td>
<td>n_y</td>
<td>1.740 - 1.755</td>
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<tr>
<td>Density (g/cm³)</td>
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</tr>
<tr>
<td>UV fluorescence</td>
<td>Long-wave</td>
<td>Bright red</td>
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<tr>
<td>Short-wave</td>
<td></td>
<td>Weak red or inert</td>
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</table>
Figure 9. The twin boundaries in this cyclic twin of synthetic alexandrite show a distinct step-like structure. View almost parallel to the a-axis, immersion, crossed polarizers, magnified 60x.

see figure 4), only very weak growth structures were found parallel to external crystal faces.

Inclusions. Various forms of residual flux were observed in many of the Russian synthetic alexandrites. These include “feathers” or “fingerprints” that consist mainly of isolated droplets or dots, which could be confused with healing features in natural alexandrites. Occasionally, we saw two-phase inclusions of residual flux and spherical bubbles, formed by shrinkage of the trapped flux as it cooled. Feathers of interconnecting tubes or channels (figure 16) also were frequently seen. In some crystals, planar, almost continuous, thin films within a net-like or web-like (i.e., emanating outward from a central point) pattern of flux were observed (figure 17). Occasionally, birefringent refractive components were also found in these flux “nets” (figure 18). In many cases, the flux inclusions took the form of wispy veils (figure 19).

In some of the samples, we observed metallic inclusions (particles or needles). On the basis of EDXRF analyses of similar-appearing solid phases on the surfaces of some of the crystals, we identified these inclusions as platinum.

Chemistry. The EDXRF spectra of the faceted samples and rough crystals with clean faces—that is, without any flux residue on the surface or in open pits or cavities—indicated varying amounts of Cr, V, Fe, Ga, Ge, Bi, and Mo (figure 20), as well as the expected Al. On the basis of these EDXRF results, we added Ge to the list of elements that we measured with the electron microprobe. X-ray fluorescence analysis also confirmed the presence of Sn, traces of which were detected in the course of the microprobe analyses.

Initial scans by electron microprobe revealed zonal variations of all minor-to-trace elements, but zoning of Cr, Fe, and Ge—and sometimes V—was particularly evident. A detailed scan (about 3 mm in length, with 625 point analyses) across one sample (see table 4, sample 7) revealed a distinct zoning and correlation of Cr, Fe, and Ge. In this sample, Ge showed a positive correlation with Fe and a negative correlation with Cr. In other words, when Ge increased, Fe also increased, but Cr decreased as
growth conditions changed. In other samples, however, a negative correlation between Ge and Fe was observed; that is, when Ge increased, Fe decreased. Two synthetic alexandrites had a positive correlation between V and Fe, and one sample showed a positive correlation between Cr and Ge.

Table 4 summarizes the analytical results. In all samples, distinct amounts of Cr and Fe were present as chromophores (see Spectroscopic Features and Discussion sections below); in two alexandrites (samples 2 and 5), minor concentrations of V were also detected. In one single crystal (sample 9), the vanadium content was distinctly higher than the chromium; EDXRF revealed a similar condition of V > Cr in two other small single crystals.

An extreme variation in CrO, FeO, and GeO, was observed in two samples (2 and 6). With the microscope, both revealed distinct color zoning: a somewhat irregular, very intensely colored boundary zone between a somewhat lighter core and a somewhat lighter rim (see figure 15). One of these synthetic alexandrites (figure 21) was sawn and polished to match the orientation in figure 12A, so that electron microprobe traverses could be made across growth zones related to a (scan 1) or a (scans 2 and 3) faces. The distribution of Cr, Fe, and Ge is shown in figure 22, which represents the analytical data obtained in scan 3. This traverse reveals inner and outer cores, a boundary area, and a rim, similar to the zoning seen with the microscope (figure 21). The inner core had the most GeO, as well as distinct amounts of CrO and FeO. In the outer core, GeO content was almost half that of the inner core, and the CrO and FeO contents were somewhat more than that of the inner core (table 5). The rim contained distinctly less Ge and Fe than the core, as well as slightly less Cr. In the boundary zone between core and rim, the Cr content jumped to an extremely high value and then dropped progressively in several steps toward the rim. Scans 1 and 3 (figure 21) revealed similar results (table 5). A scan across a second color-zoned alexandrite (sample 2) showed similar Cr zoning in the boundary area.

All samples had traces of GaO and trace-to-minor amounts of SnO (table 4). MnO and TiO were always close to the detection limit of the microprobe.

Solid Phases on the Surfaces of Rough Crystals. Several types of foreign matter were found on the surfaces of the rough synthetic alexandrites or in cavities, cracks, or pits in the surface.
Figure 14. Here, a pinacoids and a dipyramid (see figure 12B) form a growth pattern that is also characteristic for synthetic alexandrite; the dipyramid forms a characteristic angle of 86° (not inset). Immersion, magnified 30x and (inset) 50x.

White polycrystalline crusts were removed from four samples and identified as anatase (TiO₂) by a combination of X-ray powder diffraction analysis, SEM-EDS, and EDXRF. Highly reflective particles on the surface of rough alexandrites (figure 23) were identified by EDXRF as platinum, most probably originating from the crucible. On the surface of a few samples, we also observed a pattern of platinum particles: a tetrahedral particle with a smaller skeleton-like crystal that is trailed by a thin needle. Similar platinum particles occasionally were found as inclusions.

Gray, fine-grained materials in cavities, pits, and cracks were identified in several cases as molybdenum- and bismuth-bearing compounds, that is, as residual flux. In one sample, the X-ray fluorescence spectrum of a crust of this gray, fine-grained material revealed characteristic lines for tungsten as well as for Mo and Bi. The morphology of this particular sample was typical of that described earlier for other samples (see figure 6).

Spectroscopic Features. Ultraviolet-Visible Spectroscopy. The polarized absorption spectra for the single crystals of flux-grown synthetic alexandrite were consistent with data reported in the literature for chromium, vanadium, and iron as chromophores in natural and synthetic alexandrites (Farrell and Newnham, 1965; Bukin et al., 1978, 1980; Schmetzer et al., 1980; Powell et al., 1985).

Infared Spectroscopy. The infrared spectra of the rough and faceted samples showed some characteristic absorption bands in the 2800 to 3300 cm⁻¹ range (figure 24). In samples obtained from Novosibirsk (1993), the following absorption maxima were found (in cm⁻¹): 2855, 2921, 2938 (shoulder), 3205 and 3224; samples originating from Bangkok (1994) revealed maxima at 2855, 2921, 2938 (shoulder), 3025, and 3106 cm⁻¹. Because water and/or hydroxyl absorption bands—especially in the 2500 to 3000 cm⁻¹ range—are absent from the spectra of synthetic alexandrites, infrared spectroscopy is a powerful tool for separating natural and synthetic alexandrites (Leung et al., 1983, 1986; Stockton and Kane, 1988).

DISCUSSION

Growth Conditions of Russian Flux-Grown Synthetic Alexandrites. The experimental results of our study are consistent with the descriptions published by Rodionov and Novgorodtseva (1988) and Bukin (1993) of growth techniques for Russian flux-grown synthetic alexandrites. Uniform morphology and chemical properties of our samples indicate that these synthetic alexandrites were grown from a solvent consisting of molybdenum-, bismuth-, and germanium-bearing compounds, most probably oxides. The nutrient contained the main components of chrysoberyl [Al₂O₃, BeO], as well as color-causing dopants (chromium, vanadium [sometimes], and iron oxides). Cr, V, and Fe are known chromophores.
mophores of both natural and synthetic alexandrites (Farrell and Newnham, 1965; Bukin et al., 1980; see also table 1). A varying intensity of color change in different samples is caused by the absolute chromium and iron contents of the samples and by the distribution of samples between two different Al sites of the chrysoberyl lattice (Solntsev et al., 1977; Bukin et al., 1983; Schmetter et al., 1989).

We already knew that Mo and Bi were components of the fluxes used in Russia to grow synthetic alexandrite (A. Ya. Rodionov, pers. comm., 1988; Bukin, 1993). However, our chemical data do not agree with those given by Henn et al. (1988) and Henn (1992), who reported the presence of sulfur. It is possible that they misread the characteristic X-ray line of Mo (at 2.31 KeV) for the X-ray line of sulfur (at 2.29 KeV).

Ge has not been mentioned before as a trace element in Russian flux-grown synthetic alexandrites. The only reference we found to Ge in synthetic alexandrite refers to patent applications by Isogami and Nakata (published in 1985 and 1986), who described a dopant of GeO2 used to grow synthetic chrysoberyl and alexandrite cat’s-eyes. Because Ge is normally found in tetrahedral coordination in oxide structures, the zoning of Ge identified by electron microprobe suggests an isomorphic substitution of beryllium by germanium in the lattice of our samples. This disproves our preliminary assumption that the Ge came from compounds in cavities or fissures (“feathers,” “fingerprints”) within the synthetic alexandrites. It is most likely that the incorporation of Ge into the chrysoberyl lattice is very sensitive to small temperature changes and/or to small variations in the composition of the flux, as is indicated by the extreme variation in Ge in the microprobe scans (see table 5 and figure 22). Because Ge was positively correlated to Fe in some of the samples, but others revealed a negative correlation between iron and germanium, we could not prove a coupled substitution of beryllium and aluminum by...
Figure 20. This EDXRF spectrum of a rough synthetic alexandrite was taken from an optically clean crystal face. The spectrum reveals distinct amounts of the chromophores (Cr, Fe), traces of Ga, distinct Ge concentrations, and residues of the flux (Bi, Mo).

germanium and iron (for charge compensation) in all samples. Thus, we do not presently know exactly how Ge is incorporated into the chrysoberyl lattice, but it is probably due to special growth conditions. It is very likely that one of the special growth conditions—mentioned by Rodionov and Novgorodseva (1988) for the production of more-or-less equidimensional crystals (not thin platelets)—accounts for the presence of a germanium-bearing compound in the solvent, which was found in all samples obtained since 1988 (see table 4).

Our results also indicate that at least some Russian synthetic alexandrites were grown experimentally in a Mo-Bi-W-bearing flux. The use of a tungsten-bearing compound in the solvent was mentioned by Cline and Patterson (1975). Solid phases on the surfaces of the crystals we examined are representative of the major components of the flux (Mo and Bi, sometimes W) and the crucible material (Pt). At this time, however, no explanation is available for the presence of white crusts of anatase on some samples (G. Bukin, pers. comm., 1995), and our chemical analyses of the samples (table 4) do not indicate the presence of titanium in the solvent during growth.

Ga is known as a trace element in natural alexandrites (Ottemann, 1965; Ottemann et al., 1978), but small amounts of Ga recently have been observed in Czochralski- and flux-grown synthetic alexandrite (Schrader and Henn, 1986; Henn et al., 1988; Henn 1992). To the authors’ knowledge, Sn

### Table 4: Electron microprobe analyses of nine Russian flux-grown synthetic alexandrites.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Origin</th>
<th>Description</th>
<th>Growth zoning</th>
<th>Color zoning</th>
<th>Number of scans</th>
<th>Approx. length of scans</th>
<th>Analyses in wt.% (ranges)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Novosibirsk 1988</td>
<td>Single crystal</td>
<td>Yes</td>
<td>No</td>
<td>4</td>
<td>3 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>2</td>
<td>Novosibirsk 1991</td>
<td>Cyclic twin</td>
<td>Yes</td>
<td>Yes</td>
<td>2</td>
<td>2 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>3</td>
<td>Novosibirsk 1993</td>
<td>Cyclic twin</td>
<td>Yes</td>
<td>Yes</td>
<td>1</td>
<td>10 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>4</td>
<td>Novosibirsk 1993</td>
<td>Cyclic twin</td>
<td>Yes</td>
<td>Yes</td>
<td>71</td>
<td>3.5 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>5</td>
<td>Bangkok 1994</td>
<td>Cyclic twin</td>
<td>No</td>
<td>No</td>
<td>1</td>
<td>5 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>6</td>
<td>Bangkok 1994</td>
<td>Cyclic twin</td>
<td>No</td>
<td>No</td>
<td>50</td>
<td>2.5 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>7</td>
<td>Bangkok 1994</td>
<td>Cyclic twin</td>
<td>No</td>
<td>No</td>
<td>50</td>
<td>3.5 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
<tr>
<td>8</td>
<td>Bangkok 1994</td>
<td>Single crystal</td>
<td>Yes</td>
<td>Yes</td>
<td>50</td>
<td>5 mm</td>
<td>CaO 0.01-0.07, Fe 0.01-0.05, Cr 0.01-0.08, V 0.02-0.07, Mn 0.01-0.02, Ti 0.00-0.02, Fe 0.43-0.62, Cr 0.01-0.06</td>
</tr>
</tbody>
</table>

6 For this particular sample, an additional detailed scan for GeO2, Cr2O3, and FeO with 625-point analyses was performed.
7 See also table 5.
Figure 21. Visible in this color-zoned alexandrite are inner and outer red cores, a narrow, very intense, red boundary, and a light red rim (the yellow in this photo is a function of the immersion liquid and the illuminant). The three electron microprobe traverses across this section covered growth zones confined to $x$ (scan 1) and $a$ (scans 2 and 3) faces. View parallel to the $b$-axis, immersion, crossed polarizers, magnified 15x.

previously was known as a trace element only in natural alexandrites (Ottemann, 1965; Ottemann et al., 1978, Kuhlmann, 1983). Its presence in some of our samples makes it less useful than before as an indicator of natural origin.

The traces of gallium and tin came from impure chemicals used in the nutrient, and were not intentionally added to the solvent system, according to Dr. G. V. Bukin (pers. comm., 1995).

Most of the alexandrites were grown in a negative temperature gradient in contact with the bottom of a platinum crucible, as described for beryl-bearing oxides and silicates by Bukin (1993; figure 2C). The exception, the seven small single crystals that did not show an irregular surface plane, were grown by spontaneous nucleation without contact to the crucible. This is possible in a temperature-gradient system in the lower part of the crucible.
cible (figure 2C), but also in a slow-cooling system (figure 2A). A vanadium-rich nutrient is likely.

No seed crystal was observed in any of the single crystals or cyclic twins examined; that is, those crystals with an irregular contact plane and without any broken or sawn parts were grown by spontaneous nucleation from the solvent mentioned above. It is possible that those crystals that had one broken or sawn surface were produced by seeded growth, but no evidence of this was observed in any of our samples.

The properties of those samples with distinct color zoning indicate a multi-step growth process: First, growth of the core, followed by partial dissolution of the crystal during a period with a higher temperature; and then a second growth period, as the temperature was lowered again. The extremely high chromium concentrations found at the beginning of the second growth phase in these samples indicate an increase in the Cr concentration of the flux during the dissolution period.

All samples with this distinct chromium zoning revealed an intense color and a good to very good color change, which is explained by the thin intermediate layer of alexandrite with an extremely high chromium content (which could remain, at least partly, after fashioning).

Diagnostic Properties. Faceted Russian flux-grown synthetic alexandrites may show a number of features that distinguish them from natural alexandrites.

Careful microscopic examination can detect characteristic forms of residual flux and platinum particles (see also Trossarelli, 1986; Henn et al., 1988; Henn, 1992; Hodgkinson, 1995). Although some patterns of residual flux resemble the healing...
fractures seen in natural alexandrites (see, e.g., Bank et al., 1987; Henri, 1987; Bank et al., 1988), to date films of flux with a net- or web-like structure have been seen only in synthetic alexandrites.

Characteristic growth patterns—which are formed by four dominant crystal faces—a, x, k, and o in the Russian synthetic alexandrites—can be observed with immersion microscopy. However, characteristic growth patterns and growth patterns in natural gem-quality alexandrites from major localities have not yet been published. Consequently, we do not know how useful these features will be in an identification. Preliminary results indicate that the rhomboic prism (021), which is seen in most Russian flux-grown synthetic alexandrites, is extremely rare in natural samples (see, e.g., Goldschmidt, 1913). Characteristic color zoning—an intensely colored, somewhat rounded boundary between a lighter center and rim—is also diagnostic for some of the Russian samples. Traces of germanium, molybdenum, and/or bismuth, and sometimes tungsten, provide proof of synthesis. All can be readily determined by X-ray fluorescence analysis. Traces of gallium and tin can be found in natural alexandrites and in synthetic Russian samples, as can the chromophores chromium, iron, and sometimes vanadium. Therefore, these trace elements are of no diagnostic value.

Infrared spectroscopy of Russian synthetic flux-grown alexandrites shows some absorption bands in the 2000 to 3000 cm⁻¹ range that are characteristic of this material. Like other synthetic alexandrites, this material lacks the water-related absorption bands that are typical of natural alexandrites. Gemological properties—such as refractive indices, density, pleochroism, and UV-visible absorption spectra—are of no help in separating Russian flux-grown synthetic alexandrites from their natural counterparts.

CONCLUSIONS
The chemical, physical, and microscopic properties of the more than 200 Russian flux-grown synthetic alexandrites we tested are consistent with known details of the production techniques developed in Russia for this material. Platinum crucibles—with a flux containing molybdenum, bismuth, and germantnium-bearing components—are placed in a reverse temperature gradient, in which the growth zone is located below the dissolution zone. Almost equidimensional alexandrite crystals grow in this system by spontaneous nucleation in contact with the bottom of the crucible. The nutrients—which consist of Al₂O₃, BeO, and the chromophores (chromium, iron, and sometimes vanadium oxides)—are placed in the upper parts of the crucibles. The morphology of the crystals, and their chemical and physical properties, are related to the exact compositions of the fluxes and the nutrients, as well as to the temperatures in the upper and lower parts of the crucibles. The manufacturers will not reveal the specific growth conditions, and the details of these conditions cannot be deduced from study of the synthetic alexandrites that result. A careful microscopic examination of inclusions and structural characteristics (growth patterns and twinning) may be helpful—but is often not conclusive—in separating natural from synthetic alexandrite. However, modern gemological laboratories, especially those with X-ray fluorescence and infrared spectroscopy, should have no problem identifying the synthetic Russian alexandrite material currently available in the trade.

REFERENCES
202 Synthetic Alexandrite

GEMS & GEMOLOGY Fall 1996


ARAGONITE

A 55.10 x 37.80 x 9.04 mm white translucent carved pendant (Figure 1) was recently submitted to the West Coast laboratory for identification. It had been represented to the client as nephrite jade.

The piece was similar in appearance to nephrite and had a comparable specific gravity of 2.84. Magnification revealed an aggregate structure. No absorption spectrum was seen with a desk-model spectroscope. The refractive index measurements, which were vague and difficult to determine because of the poor polish, gave values of 1.50 and 1.65. There was a decided “blink” effect when the Polaroid plate was rotated.

Other gemological properties were: moderate yellowish white fluorescence to long-wave ultraviolet radiation (with yellow fluorescence in fractures), moderate light yellow fluorescence to short-wave UV, and effervescence when touched in an inconspicuous spot with a minute droplet of a 10% hydrochloric acid solution. Although these properties were not sufficient to identify the material, they proved that it was not nephrite. The effervescence, together with the R.I. “blink” effect, indicated that it was a carbonate.

X-ray powder diffraction analysis identified the carving as aragonite.

Aragonite is not often used as a gem material because of its low hardness (about 4 on the Mohs scale). It has been shown that nitrogen is often distributed unevenly in the diamond crystal see, e.g., “Fractionation of Nitrogen Isotopes in a Synthetic Diamond of Mixed Crystal Habit,” by S. R. Boyd et al., Nature, Vol. 331, February 18, 1988, pp. 604-607. Consequently, it is common that two near-colorless diamond cut from the same piece of rough will differ by a few color grades. Hydrogen impurities may also cause an uneven distribution of gray or brown color in diamond see, “Gemmological Properties of Type Ib Diamonds with an Unusually High Hydrogen Content,” by E. Fritsch and A. Scarratt, Journal of Gemmology, Vol. 23, No. 8, 1993, pp. 451-460.

Last fall, a diamond dealer shared a particularly vivid example of the consequences of such variability with staff members in the East Coast lab. Although he had noticed some unevenness to the color before sawing the rough diamond into the two pieces shown in figure 2, he had expected that the rough would produce two fancy-color stones. However, the larger of the two pieces yielded a 3.79 ct “radiant” cut with a color grade of Fancy Brownish Yellow, whereas the smaller piece finished out as a 0.78 ct pear shape of G color. Although the two pieces had some similar gemological properties...
Figure 2. These two pieces are from the same brownish yellow rough diamond. Nearly all the color ended up in the 7.32 ct portion, whereas the 1.35 ct piece is near-colorless.

ties—such as inclusions in both of small brown crystals (probably garnet, judging from their partly dodecahedral shape)—there were substantial differences in their infrared spectra, which reflect their different impurity concentrations. The larger, brownish yellow piece had strong peaks due to nitrogen and moderate hydrogen peaks; however, the near-colorless piece showed moderate nitrogen and weak hydrogen absorptions. The nitrogen in both pieces had the same aggregation state, with approximately equal amounts of A and B aggregates. Much of the color in the brownish yellow piece was concentrated in a swirling cloud in the center, a distribution sometimes seen in hydrogen-rich diamonds (again, see the Fritsch and Scarlatti reference cited above). 

**Figure 4.** Compare the appearance of these five diamond crystals (4.62-26.24 ct) to that of the imitations in figure 3.
turing, both with a microscope to detect visible inclusions and with a polariscope to detect any extraordinary strain or grain. Some diamond categories and colors, such as browns, are noted for their high degree of internal strain. In these diamonds, the presence of strain often influences how the crystal is fashioned. For example, manufacturers will sometimes opt to forgo traditional sawing and keep the stone “whole” to avoid damage. Other times, they may orient the stone so that the sawing does not intersect the center of strain.

We repositioned the two pieces in their original relative orientation to illustrate their relationship in the original piece of rough (figure 6). Figure 7 (taken with a polarizing microscope) shows the blue first-order interference color from residual strain that is present in both pieces. The fractures in both pieces relate to a single area of strain that was present in the rough crystal. Also present was a linear strain pattern along the octahedral grain near both culets, but its gray interference colors indicated that this strain was significantly lower.

Whether strain in a rough diamond will lead to spontaneous fracturing during fashioning is a matter of chance, but the presence of localized strain is an indication for concern. Sawing is thought to be the most stressful event in diamond cutting, however, in this case the stone survived sawing perpendicular to the strain region, but the pieces fractured as faces were polished subparallel to the strain region.

With natural green, blue, and red diamonds, Winter 1982, p. 238, which showed a chameleon diamond with a dramatic color change, and Summer 1988, p. 112, with a grayish purple round brilliant cut.

The 3.40 ct heart-shaped diamond in figure 8 is another addition to this list. It was graded Fancy Intense Pinkish Orange and Internally Flawless. (For a description of the GIA Gem Trade Laboratory color-grading system, see “Color Grading of Colored Diamonds in the GIA Gem Trade Laboratory,” by J. King et al., Gems & Gemology, Winter 1994, pp. 220-242.) Not only was the hue unusual for diamond, but the saturation was unusually high for such a color. In the rare instances when we have seen diamonds in this hue, the depth of color has been much weaker (less saturated). The rough reportedly came from Angola.

Infrared spectroscopy revealed that the stone was a type Ha diamond (i.e., lacking measurable amounts of nitrogen). The fluorescence was very strong range to long-wave UV radiation and moderate orange to short-wave UV. The long-wave reaction further enhanced the color of the diamond when viewed in the daylight. The absorption pattern seen with a desk-model spectroscope consisted of a broad band centered at 550 nm.

Rare Color: Fancy Intense Pinkish Orange

Over the years, we have reported on a number of diamonds notable for their rare colors (see, for example, the Winter 1965-66 Gems & Gemology, p. 362, which illustrated a ring set with natural green, blue, and red diamonds; Winter 1982, p. 238, which showed a chameleon diamond with a dramatic color change; and Summer 1988, p. 112, with a grayish purple round brilliant cut).

Whether strain in a rough diamond will lead to spontaneous fracturing during fashioning is a matter of chance, but the presence of localized strain is an indication for concern. Sawing is thought to be the most stressful event in diamond cutting, however, in this case the stone survived sawing perpendicular to the strain region, but the pieces fractured as faces were polished subparallel to the strain region.

Figure 5. These two diamonds, a 16.48 ct blocked round and an 18.15 ct blocked square, were cut from the same piece of rough. Both fractured due to strain.

Figure 6. Here the diamonds shown in figure 5 are oriented and repositioned to demonstrate their original crystallographic relationship.

Figure 7. First-order interference colors—shades of blue—seen here in the partly polished 18.15 ct piece were actually present in both halves, indicating the presence of residual strain.
A Suite of Treated-Color Pink-to-Purple Diamonds

The East Coast laboratory recently had the opportunity to examine six known treated-color pink diamonds borrowed from a local dealer. Although treated pink diamonds are relatively uncommon, we have seen and documented a number of them over the past 25 years. The diagnostic properties noted before (see Gems & Gemology, Summer 1976, p. 172; Summer 1988, p. 112, and Summer 1995, p. 121) were also observed in these diamonds; however this parcel provided a few surprises.

Two of the diamonds showed strikingly unusual colors, which are immediately apparent in figure 9. Although GIA GTL maintains a policy of not issuing color grades for stones of treated color, these stones were put through the color-grading process in order to locate their positions in color space and to compare them to the range of colors seen in natural-color diamonds. Treated pink diamonds are usually highly saturated, but dark in tone. The 0.05 carat orangy pink diamond, comparable to a grade of Fancy Vivid, was surprisingly light in tone for a treated-color pink, as well as being highly saturated. The pink-purple color of the 0.07 carat diamond is truly extraordinary, comparable to a grade of Fancy Vivid. Purple is an extremely rare color in diamond, and seldom has the lab seen a purple of this high saturation in either a treated- or natural-color diamond. The orangy pink stone is at least as saturated as any natural-color diamonds we have seen of similar hue. The colors of the other four diamonds lie on the edges of the distribution we have seen for natural-color diamonds, with the high saturations and medium-to-dark tones that are typical of treated-color pink stones.

In a desk-model spectroscope, all six showed the diagnostic spectrum for treated-color pink (or purple or red) diamonds: sharp absorption lines at 575, 595, and 637 nm. Most of these diamonds showed emission lines at 595 and 637 nm as well. They also luminesced the characteristic strong, bright orange to both long-wave and short-wave UV that is associated with the 637 nm color center. Last, although graining was present in all six diamonds, it was phantom graining (for the meaning of this term, see "The Elusive Nature of Graining in Gem Quality Diamonds," by R. E. Kane, Gems & Gemology, Summer 1980, pp. 294-314) with no relationship to the distribution of color within the gems. (By contrast, most natural-color pink diamonds show most, or all, of their color as colored graining.) One stone had a "streaky" color distribution (figure 10), but magnification combined with immersion revealed that the color is largely confined to the surface.

Although the face-up appearance of both natural- and treated-color pink, purple, or red diamonds can be similar, the causes of the color are entirely different. Natural-color pink diamonds are type Ia or Iia and show a smooth, broad band in the visible spectrum at about 550 nm. (See E. Fritsch and K. Scurratt, "Natural-Color Nonconductive Gray-to-Blue Diamonds," Gems & Gemology, Vol. 28, No. 1, Spring 1995, pp. 38-39, for a complete discussion of diamond type.) Treated-color pink diamonds must contain at least some type Ib component, and are sometimes pure type Ib. Their visible spectra show the three sharp lines mentioned in figure 8. The Fancy Intense Pinkish Orange color of this 3.40 ct Internally Flawless heart shape is rare.

Figure 8. The Fancy Intense Pinkish Orange color of this 3.40 ct Internally Flawless heart shape is rare.

Figure 9. Among the colors displayed by this suite of treated diamonds are some that match or exceed the brightest natural pink colors seen in the lab to date. The 0.05 ct orange pink and the 0.07 ct pink-purple stones, right, are particularly noticeable for their high saturation.

Figure 10. The streaky pink color seen in this 0.05 ct treated-color pink diamond might be confused with the pink graining seen in natural-color diamonds. Examination under 10x to 100x while the stone was immersed in methylene iodide reveals that the color is largely confined to the surface.
above, with a broad feature formed from the overlap of their sidebands. Vacancies in the atomic structure are created in these diamonds by laboratory irradiation, and then mobilized during an annealing process. Some of the vacancies become trapped at single substitutional nitrogen atoms, creating the NV center with its strong absorption at 637 nm.

In examining these six stones, we found that four were type Ib, as expected, but one had some additional type IaA component and one of the paler stones had what appeared to be a pure type IaA spectrum and an H1b peak (due to a vacancy trapped at an A aggregate, a common feature in treated yellow diamonds; again, see the A. T. Collins reference cited in a previous entry). We had not seen such a mid-infrared spectrum in a treated pink stone before. Although the infrared spectrum implied that this diamond had only A aggregates, the visible spectrum showed the 637 nm peak, so there must have been at least some single substitutional nitrogen to trap the vacancies.

The nitrogen content of all six of these diamonds was extremely low, and some of the initial infrared results suggested that a few of them might have no nitrogen at all. For the stone that showed type IaA, it is probable that there was not enough single substitutional nitrogen to produce a detectable signal in the infrared spectrum. These diamonds demonstrate the time-honored gemological principle that identification must rely on all of the observed properties. A cursory examination might lead one to conclude that the paler stones are natural-color type IIa pink diamonds with orange fluorescence and faint pink graining. However, in our experience, such stones rarely show the 637 nm line, and never show the 595 nm line, in the hand spectroscope.

John King, Elizabeth Doyle, and IR

**HERDERITE**

In spring 1996, the West Coast lab received for identification a large [25.86 x 18.55 x 13.31 mm] pear-shaped stone that was transparent yellowish green [figure 11]. The 38.91 ct stone proved to be an extraordinarily large herderite.

We recorded the following gemological properties for this stone: biaxial negative, with R.I. values of $\alpha = 1.580$, $\gamma = 1.611$, and $\beta$ between 1.600 and 1.606; S.G. [measured hydrostatically] of 3.04; no reaction (inert) to long-wave UV radiation, but faint yellowish green fluorescence to short-wave UV. With a desk-model spectroscope, we observed an absorption line at 585 nm.

Microscopic examination revealed a small feather under the table, as well as scattered “pinpoint” inclusions in the stone. However, the most pronounced feature seen with magnification was the strong doubling of the back facets visible through the table. This was not surprising given the strong birefringence (0.031) and the depth of the stone.

To satisfy our curiosity about this stone—and to characterize the material for our data files—we submitted it to energy-dispersive X-ray fluorescence (EDXRF) analysis. Calcium (Ca) and phosphorus (P) were the major elements found, along with minor manganese and trace amounts of lead, strontium, and yttrium. Gem herderite consists of material in the mineral series that has herderite, CaBe[P-O$_4$]F, as one end-member and hydroxylherderite, CaBe[P-O$_4$]OH, as the other. Because the elements beryllium (Be), oxygen (O), fluorine (F), and hydrogen (H) cannot be detected with our EDXRF system, we could not determine which end-member in the herderite-hydroxylherderite series is dominant in this stone. The manganese is probably responsible for the yellowish green color, which is similar to the color caused by manganese in some spodumenes.

**JADEITE**

An intricately carved hairpin [figure 12] with a dragon motif was submitted to the East Coast laboratory to determine if it had been treated. Reportedly, this ornate piece was from the Chinese Qing dynasty (1644–1912), which popularized the use of jadeite jewelry and objets d’art.

Standard gemological tests revealed properties that were consis-
tent with jadeite jade. The strength of the “chrome” lines in the absorption spectrum varied with the intensity of the green color. There was no reaction with a Chelsea filter. Even with magnification, no visible reaction was observed when a “hot point” was brought near the surface. In addition, the carving revealed a compact intergrowth pattern very different from the “honeycomb” structure sometimes seen in bleached and impregnated jadeite. Infrared spectroscopy showed no evidence of impregnation.

Unlike the simple Western definition of “Imperial jade” [jadeite of uniform intense green color and exceptional translucency], Asian criteria are more complex and take into account a combination of factors, including the highest quality green color and translucency, as well as the carving’s quality and style (see “Asian News Section: Jadeite Jewellery in the Qing Imperial Court,” Christie’s International Magazine, May 1996, p. 73). We suspect that this hairpin might be considered “Imperial” by either standard.

The difficulty in detecting that a piece of jadeite has been bleached and polymer impregnated, which is now a common practice in the marketplace, has made it necessary to test all important pieces, regardless of their purported history.

GRC and TM

**OPAL, an Assemblage**

An opaque gray free-form cabochon, displaying primarily green and blue play-of-color, was sent to the East Coast laboratory for identification. Since the stone was bezel set in a closed-back pendant mounting (figure 13), the client wanted us to determine if this opal was a quartz-top doublet or triplet, as is frequently encountered in the trade.

Visual examination showed that the top portion of the cabochon was obviously transparent. The play-of-color was not visible near the surface of the stone, but appeared to be confined to a deeper, somewhat opaque layer. The refractive index of the top, obtained by the spot method, was 1.52, which is lower than the 1.54-1.55 of quartz. Because of the irregular shape and the bezel mounting, it was difficult to determine an optic character between crossed polarized plates, but the transparent top appeared to be singly refractive with little anomalous double refraction. When exposed to long-wave UV radiation, the cabochon fluoresced a
strong bluish white with a distinct phosphorescence. However, when the stone was exposed to short-wave UV, we noticed a faint chalky yellow fluorescence, which is not characteristic of colorless quartz. These properties proved that the transparent top portion was not quartz, but a different material.

Magnification did not reveal any obvious inclusions. However, using standard overhead illumination, we did locate a tiny gas bubble deep inside the transparent layer. This confirmed that the top was glass. Further examination of the underly-

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**Figure 14.** Numerous thread-like "inclusions" were seen in these gray-blue assembled cultured blister pearls, which each measured about 6.35 mm in diameter.

**Assembled Cultured Blister PEARLS, With Thread-Like Inclusion:**
Although gemologists do not expect to observe inclusions when examining pearls, staff members at the West Coast lab noticed some interesting internal features in cultured pearls that arrived last year for identification. The assembled cultured blister pearls, each of which measured about 6.35 mm in diameter, were an attractive light gray-blue. They were surrounded by old European brilliants, all set in a pair of earrings (figure 14).

The fact that these pieces were assembled was readily apparent with microscopic examination. Fiber-optic illumination revealed gas bubbles in the curved interface between the nacre and the bead nucleus. The flat bases of the cultured pearls had soft, waxlike backings attached. Not only was the gray-blue color unnatural, but fiber-optic illumination also revealed vivid color concentrations reflecting along hairline fractures in the nacre. Although these features indicated an artificial coloring process, we could not prove that one had been used.

The most interesting characteristic of these assembled cultured blister pearls were the thread-like inclusions in the nacre, which were again best observed with fiber-optic illumination. For the most part, these abundant inclusions appeared to be randomly oriented; they were long, curved, and continuous, with some shorter, more kinked threads splitting off to the sides (figure 15). The laboratory was unable to identify the composition or origin of these inclusions.

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**Figure 15.** The long curved fibers in the assembled cultured blister pearls of figure 14 seemed, for the most part, to be randomly orient-

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**Figure 16.** Numerous thread-like "inclusions" were seen in these gray-blue assembled cultured blister pearls, which each measured about 6.35 mm in diameter.

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**Figure 15.** This assemblage, which measures about 30 x 22 mm, has a colorless glass top over natural opal.

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**QUARTZ, Single-Crystal Green**

Last winter, the East Coast lab had the opportunity to examine a turn-of-the-century ring, stamped "Tiffany & Co.," set with an oval mixed-cut green quartz that was surrounded by fine-quality old-European brilliant cuts (figure 16). Both the setting and the company name indicated more respect for this gem than is typical today for similar single-crystal quartz of grayish green color. The ring may well represent an example of Tiffany's interest in promoting American gemstones, as green quartz was known at that time to occur in several locations (see G. F. Kunz, Gems and Precious Stones of North America, 2nd ed., 1968, Dover Publications, New York, pp. 285, 286). If so, it joins other American gems as Montana sapphires, chrome pyrop garnets, and American freshwater pearls, among others (see Gem Trade Lab Notes, Spring 1989, p. 37, and The Tiffany Touch by J. Purrall, 1971, Random House, New York).

This color of single-crystal quartz was rare until the mid-1950s, when it was discovered that amethyst from the Montezuma mine in Minas Gerais, Brazil, would turn green with...
heat treatment. Such treated-color quartz is commonly known by the trade name “prasiolite.” In recent years, the lab has also seen a few pieces of synthetic quartz with this grayish green color.

The stone in the ring proved to be natural quartz, as it demonstrated both Brazil twinning and parallel color zoning. However, it is not currently possible to distinguish naturally green quartz from heat-treated material.

SAPPHIRE, Unusual Treated Natural Sapphire

In the Spring 1996 issue of Gems & Gemology, we reported on flame-fusion synthetic corundum oddities. Earlier this year, within a short period of time, the East Coast lab received two rather odd natural sapphires of treated blue color. The first was a 70.20 ct oval cabochon (figure 17) with an incised design on the back. Microscopic examination revealed numerous fluid-filled “fingerprints” and unidentified crystals that were altered in ways consistent with heat treatment. Exposure to long-wave UV radiation produced little reaction, but the stone fluoresced a patchy chalky blue to short-wave UV, further supporting the conclusion that it had been heat treated.

When examined with magnification in diffused light, several of the cavities and shallow fractures revealed blue color concentrations reminiscent of the effect sometimes seen in diffusion-treated stones (see, e.g., R. E. Kane et al., “The Identification of Blue Diffusion-Treated Sapphires,” Gems & Gemology, Summer 1990, p. 124, figure 10). In this stone, the apparent color “bleeding” was actually a blue “dye,” as was evident when the stone was immersed in methylene iodide (di-iodomethane; see figure 18). When we re-examined the stone with short-wave UV radiation and low magnification, the areas that were luminescent corresponded to those with the blue dye. This treatment could easily have been confused with diffusion treatment because of the shallow penetration of the blue color in some areas.

The second stone (figure 19) was a diffusion-treated natural sapphire that had been quench crackled, making it more challenging to identify. Quench crackling, a relatively common procedure, is most often seen in quartz and beryl, which are quench crackled and then dyed green to imitate emerald. In the Summer 1996 Lab Notes section (pp. 125–126), however, we reported on a parcel of quench-crackled synthetic rubies. The crackling in this sapphire made it difficult to see the very small crystals and silk, which had been reduced from needles to lines of small unconnected dots. More importantly, it also disguised the surface diffusion treatment. Immersion again revealed the telltale outlined facet junctions and uneven surface coloration, GRC and TR.

Flame-Fusion SYNTHETIC SAPPHIRE

When GIA was founded in the early 1930s, gemology was relatively uncomplicated and most identifications could be made with basic instrumentation. However, then as now, the identification of synthetics was particularly worrisome. When we consider the primitive gemological microscopes available at that time, the great concern for the proper identification of Verneuil flame-fusion synthetics—the only type then available—is understandable.

Pioneering work in Europe had established that the main identifying features in these synthetic sapphires and rubies were curved striae or growth lines, as well as gas bubbles, large or small. One of the first indications that features believed to be characteristic of natural sapphires were also seen in synthetics was reported in 1920 by Mr. E. G. Sandmeier of Locarno, Switzerland, and confirmed by Mr. W. Plato of Frankfurt, Germany: the discovery of polysynthetic twinning in synthetic corundum. It is significant that one of the first gemological notes written
by Robert Shipley Jr., in the second issue of the (then) new Gems of Gemology—March-April 1934—concerned his observation of nearly straight striae in a synthetic sapphire. Significant, too, is the fact that the very first (Summer 1942) of many well-illustrated feature articles in this journal by Dr. Eduard Gubelin addressed “Genuine Type Inclusions in New European Synthetic.” Occasionally, other similarities between synthetic and natural gems have appeared in the gemological literature. More recently, synthetics with characteristics introduced to make them look natural, such as natural-appearing “fingerprints,” have been seen on the market (see, e.g., J. I. Koivula, “Induced Fingerprints,” Gems of Gemology, Winter 1983, pp. 220-227).

Another example of a natural-appearing inclusion in a synthetic stone was recently seen in the East Coast lab (figure 20). Needle-like inclusions near the cleft of this heart shape had convinced the client that the piece was natural. However, using only a loupe, he thought that he also saw curved growth lines. His suspicion proved to be well founded, as the curved growth lines were easily resolved when this synthetic sapphire was immersed in methylene iodide (figure 21).
DIAMONDS

Chatham synthetic "white" diamonds at JCK show.

Three years ago, Tom Chatham of Chatham Created Gems, San Francisco, California, announced that he would be marketing "white" synthetic diamonds from Russia for jewelry use. Although it took longer than he originally anticipated, Mr. Chatham offered for sale a number of near-colorless synthetic diamond crystals at the June 1996 JCK show in Las Vegas, Nevada.

Mr. Chatham offered GIA Research a brief opportunity to examine about 100 of these synthetic diamond crystals (which ranged from about 10 points to almost a carat) before the show. Most were of small size and had too many inclusions for faceting. Research Associate Sam Muhlmeister and Research Gemologist Shane Elen focused their testing on four crystals of slightly better-than-average quality. These weighed between 0.41 and 0.51 ct, and were cubo-octahedral in crystal habit. All four had eye-visible metallic inclusions (figure 1), one had a typical white "cloud," one had relatively large stepped cavities (which resembled the hopper-growth cavities in, for instance, salt crystals, but are unusual in natural diamonds), and two had surface structures that looked like trigons. All four crystals were attracted to a strong magnet.

The crystals were inert to long-wave ultraviolet radiation, but fluoresced a very faint yellow or orange to short-wave UV. The UV fluorescence was typical, both in color and intensity, of that seen thus far in near-colorless synthetic diamonds; it is rare for a natural diamond to have a stronger fluorescence to short-wave than long-wave UV. However, no cross-shaped or octagonal pattern was visible in the UV fluorescence reaction—unlike the patterns reported previously for some synthetic diamonds [see, e.g., J. E. Shigley et al., "A Chart for the Separation of Natural and Synthetic Diamonds," Winter 1995, pp. 256-264]. The crystals phosphoresced blue for at least one minute after exposure to short-wave UV; phosphorescence was much fainter in one than in the other three.

On the basis of their infrared spectra, we determined that these four samples were all type IIa (essentially nitrogen-free) synthetic diamonds. Near-colorless synthetic diamonds are typically type IIa or mixed type IIa plus other types, whereas type IIa near-colorless natural diamonds are relatively rare. Energy-dispersive X-ray fluorescence (EDXRF) spectroscopy disclosed both iron (Fe) and germanium (Ge) in all four crystals. GIA Research had not previously detected germanium in any diamond, natural or synthetic. They suspect that Ge is being added to the growth environment of these crystals to prevent nitrogen from incorporating into the crystal structure, of the synthetic diamonds, as the nitrogen would color them yellow.

Although all of these properties indicated that these samples were synthetics, conclusive proof was provided most readily by the metallic inclusions, which were eye-visible in all four samples, and were easily identified with magnification. All four proved to be magnetic. The presence of Ge in these samples also provides proof of synthesis, but trace-element determination requires the use of equipment beyond the reach of the average gemologist. Mr. Muhlmeister and Mr. Elen cautioned that this preliminary study involved only four crystals, the properties of other Chatham synthetic "white" diamonds may differ.

Figure 1. Chatham's "white" synthetic diamonds were recently offered for sale at the Las Vegas JCK Show. Note the numerous metallic inclusions—characteristic of synthetic diamonds—in this 0.51 ct example. Photo by Shane Elen.
Figure 2. This 2.14 ct diamond (9.40 x 8.46 x 5.26 mm) is an example of the “Buddha Cut.” Photo by Maha DeMaggio.

Diamond novelty-cut as a seated Buddha. New forms of fashioned diamonds are designed either for novel light-transmission and weight-retention properties, or to resemble other items. Examples of the former cuts include the “Flanders Brilliant” [Gem News, Summer 1993, pp. 130-131] and the “Context” and “Spirit Sun” cuts [Gem News, Spring 1995, pp. 59-60]. Examples of the latter include diamonds cut to resemble dice [Lab Notes, Fall 1985, p. 172] and letters of the alphabet [Lab Notes, Spring 1986, p. 47]. We recently had the opportunity to examine a new cut, the “Buddha Cut” (figure 2), which is being marketed in the United States by J. Kleinhaus and Sons of New York City. The distribution of facets was reminiscent of triangular modified brilliants, with 33 crown facets and 21 pavilion facets (no culet). The girdle was also faceted; two GIA Gem Trade Laboratory (GTL) graders assessed the girdle as thick to extremely thick.

Some consumers wear “Buddha Cut” diamonds in necklaces, while others treat them as objets d’art, according to a J. Kleinhaus spokesperson. (The unmounted sample we examined did “sit up” by itself.) Because of the potential religious implications, the cutter reportedly has strict criteria governing cut symmetry and what constitutes a diamond appropriate for this cut. For example, the “head” region should be free of unsightly inclusions.

COLORED STONES

Nonphenomenal vanadium-bearing chrysoberyl. Attractive examples of green chrysoberyl, lacking change of color, were seen at the February 1996 Tucson gem shows, some material was marketed as “mint” chrysoberyl. Six months earlier, the editors had examined a 3.48 ct stone cut from similar material. The gemological properties (see below) confirmed that this cushion-shaped modified brilliant cut was a natural chrysoberyl. We were somewhat surprised that there was no perceptible change of color (aleurodite effect) between incandescent and fluorescent light in a stone of such saturated green. With magnification, we noted a small brown-red crystal in the pavilion and growth zoning. EDSXRF spectroscopy showed that the stone contained Al, Fe, V, Ti, Ga, and Sn. We had seen synthetic nonphenomenal green chrysoberyls colored by vanadium, but we had not previously examined natural vanadium-bearing chrysoberyls. For comparison, we borrowed from Malhotra Inc., New York City, four faceted nonphenomenal green chrysoberyls (figure 3) that reportedly had been mined in Tanzania. Three had properties similar to the chrysoberyl in question. These included pleochroism—duo-chromatic colors of dull green to blue-green/yellow-green to green/neon-colorless to yellow, optic character—birefringent, petal aftercolor; color filter reaction—none to faint pink, refractive indices—1.743 and 1.750; birefringence—0.008-0.009; specific gravity—3.71-3.73; luminescence to UV radiation—inert to faint orange (long wave) and inert to faint yellow (short wave); no luminescence to visible radiation; absorption spectrum in the desk-model spectroscope—440-445 nm band; infrared spectrum—strong features at 9225 and 2975 cm⁻¹, weaker features at 3140, 2850, and 2650 cm⁻¹; UV-visible spectrum—bands at 415 and 608 nm, peaks at 505, 365, 375, and 980 nm. Again, EDSXRF spectroscopy revealed Al, Fe, V, Ga, and Sn from the fourth stone (1.47 ct), which was bluish green, showed additional features typically associated with chromium (including a red reaction to the Chelsea color filter; moderate red luminescence to UV radiation).
visible light, and a 590-665 nm absorption—and 670 nm emission line—seen with the handheld spectroscope, as well as Cr in the EDXRF spectrum; however, there still was no perceptible change of color.

Contributing editor Henry A. Hanni had the opportunity to examine an 11.14 ct "intense" bluish green chrysoberyl (figure 4) that was reportedly from Tunduru, Tanzania. An associate at the University of Basel, M. Krzemnicki, performed electron microprobe analyses on this stone and on two samples of Russian hydrothermal synthetic nonphenomenal chrysoberyl that had been purchased at Tucson. The natural stone contained 0.4 wt.% V₂O₅, 0.2 wt.% Fe₂O₃, and trace amounts of Cr, Sn, and Ga, whereas the synthetic chrysoberyls revealed more vanadium (1.8 wt.% V₂O₅), more chromium (0.2 wt.% Cr₂O₃), and no appreciable Fe, Sn, or Ga.

Large faceted chrome diopside. At one of the 1996 Tucson shows, Alex Grizenko of the Russian Colored Stone Company, Genesee, Colorado, showed an editor two dark green oval modified brilliants: chrome diopside from the Inagly mine, Yakutsk, Siberia, north of Lake Baikal. The larger oval, at 26.17 ct, may be the largest known example of this faceted material; however, the 25.33 ct stone (figure 5) was brighter. Although this is not a new find (the rough for these pieces was probably mined 30-40 years ago), this material is increasingly popular in the colored stone market. Mr. Grizenko kindly loaned us these samples for closer examination.

We recorded the following gemological properties for the two diopside: color—dark green, with even distribution; pleochroism—weak, green to brownish green; diaphaneity—transparent; optic character—biaxial positive; Chelsea filter reaction—none. Reflective indices for the 25.33 ct stone were α = 1.670, β = 1.680, and γ = 1.699, with a birefringence of 0.029; for the 26.17 ct stone, β was not determined, but α = 1.672, γ = 1.700, and birefringence was 0.028. Both stones had an SG of 3.30, were inert to both short- and long-wave UV radiation, and had very dark spectra as seen with a handheld spectroscope. The cut-off was at about 520 nm, with "chrome bands" at 630, 660, and 690 nm. Both stones contained scattered small crystals and "fingerprints," visible with magnification but not particularly distinctive. EDXRF spectra of the two stones showed major Mg, Al, and Ca, with smaller amounts of Fe, Cr, Ni, Ti, and Sr.

"Rose"-colored plagioclase-corundum rock from Sri Lanka. In March 1995, contributing editor Henry Hanni received two small "rose" pink translucent pebbles (figure 6) that each had one small polished face. These stones reportedly came from a locality near Pallekele, Ratnapura district, Sri Lanka. The sender could not identify them on the basis of the R.I. (1.576) and S.G. (2.98) values that he determined. He also noted weak chromic lines in the absorption spectrum.

At the SSEF in Basel, microscopic observation of the surface in reflected light revealed a granular groundmass (90%) with occasional interpersed aggregates of ilmenorhomic crystals, which ranged from 20 to 70 microns (10%). The pink crystals had a hardness greater than that of the groundmass, as seen by their relatively higher relief. EDXRF analysis of the entire pebble indicated that Si, Al, and Ca were the main constituents, with Fe and Sr present as trace elements. On the basis of SEM-EDS analysis of individual mineral grains within the pebbles, the main (granulorhomic) mineral component was identified as Ca-rich plagioclase feldspar, and the interpersed harder grains were identified as corundum. The Cr was contained in the corundum, which explained the rock's pink.
Figure 6. Mixtures of feldspar and corundum, these two pebbles from Sri Lanka have some gem properties common to both materials. The largest pebble is about 1 cm across. Photo courtesy of SSEF.

Dr. Hämni doubts that this material will have great gemstone potential, but he considers it an interesting gemological puzzle: As a mixture of feldspar and corundum, it has the R.I. of one, the spectrum of the other, and an S.G. intermediate between the two.

Quartz-magnesite rock, so-called “lemon chrysoprase,” from Australia. In past Gem News items, we have reported on various yellow-green materials from Australia, including the cryptocrystalline quartz variety chrysoprase (Summer 1994, pp. 125–126; Fall 1994, pp. 193–194), colored by nickel, and the nickel-carbonate mineral gaspeite (Summer 1994, pp. 125–126), sometimes marketed as “Allura.” Another Australian material was seen throughout the 1996 Tucson gem shows, marketed as “lemon chrysoprase.” After a cursory initial inspection, the curiosities of contributing editors Dino DeChionno and Shane McClure were piqued; they decided to investigate further.

They acquired several samples, including a strand of 7 mm beads and a heart-shaped pendant (figure 7), and selected one 2.27 ct bead for detailed examination. The semi-translucent light yellowish green bead had a specific gravity of 2.83 and refractive indices of 1.51–1.68, with a pronounced carbonate “blink.” Fluorescence to both long- and short-wave UV radiation was unevenly distributed—weak yellow in scattered areas. The absorption spectrum viewed with a desk-model spectroscope showed a lower wavelength cutoff at 450 nm; a dark band between 490 and 510 nm; lines at 600, 615, and 630 nm; and an upper-wavelength cutoff at 640 nm. With magnification, distinct areas of a lighter colored, more opaque material and a brighter yellowish green, more translucent material could be seen in the bead; the more opaque material had higher relief (that is, it was harder than the greener material).

EDXRF analysis revealed silicon, magnesium, and nickel. X-ray diffraction powder patterns were consistent with two phases being present: quartz and a carbonate with the calcite structure. The unit-cell spacings determined for the carbonate mineral were consistent with magnesite (MgCO₃) rather than gaspeite (NiCO₃); however, there is a solid solution between these two minerals, so that a phase can have up to 50% gaspeite and still be considered magnesite mineralogically. One identification question remained: Was the quartz material colored green by nickel (that is, was it chrysoprase?), or was it merely intergrown with the yellow-green carbonate? To answer this, the bead was submerged in a hydrochloric acid solution. The bead was selectively dissolved and turned white; a diffraction pattern showed only quartz. Consequently, we concluded that the “lemon chrysoprase” was not chrysoprase at all, but rather a rock consisting of (white) quartz and (yellowish green) magnesite.

Update on Madagascar sapphires. The Summer 1996 Gems & Gemology featured an article on sapphires from Madagascar’s Andranondambo region (by D. Schwarz et al., pp. 80–99). Since then we have received additional information about significant discoveries of sapphire elsewhere in this region. Thomas Banker, of Gem Essence Co., Bangkok, Thailand, writes that large quantities of sapphires have also been found at Antsiemeure, about 10–12 km north of Andranondambo. The “sapphire rush” at Antsiemeure began not long before Mr. Banker’s April 1995 visit. Mining resembled that at Andranondambo, with each individual digging accompanied by his own small tailings pile of white plagioclase-rich rock. By his second visit in November 1995, a considerable shanty town (with about five streets) had developed next to the diggings (figure 8): Mr. Banker estimated...
that 2,000–3,000 miners inhabited this town at the time of his visit, and others commuted from Andranondambo. The diggings covered about 3–5 km².

Madagascar is also the source of a large (17.97 kg; 29 x 19 x 16 cm) crystal that was recovered late last year from an undisclosed region. The deep blue crystal (figure 9) was the subject of a short report in the June–July 1996 *JewelSiam* (“The Find of a Lifetime,” pp. 86–87).

**Figure 9.** By November 1995, a large shanty town, with a population in the thousands, had grown up adjacent to the Antsiermene sapphire diggings. Photo courtesy of Thomas Banker.

**Spessartine from Pakistan.** Late last year, the Gem News editors were loaned samples of spessartine from a relatively new locality, Azad Kashmir, in northeastern Pakistan. Dr. Lon C. Ruedisili of Ruedisili Inc., Sylvania, Ohio, provided a 22.31 ct rough crystal and four faceted examples (figure 10) of this garnet, which is being marketed as “Kashmirine.” Geometric investigation of the faceted stones revealed: color—slightly yellowish orange to brownish orange or red-orange; transparency—translucent; color distribution—even; optic character— singly refractive, with weak anomalous double refraction; color filter reaction—orange; refractive index—1.800, specific gravity (measured hydrostatically)–4.19 to 4.20, inert to both long- and short-wave UV radiation; and a typical spessartine absorption spectrum, with bands at 410, 420, and 430 nm visible with strong transmitted light in a desk-model spectroscope. Using magnification (and for some stones, polarized light), we saw internal growth zoning (figure 11) in all samples. In addition, the 2.31 ct stone contained acicular inclusions, possibly birefringent (figure 12), and the 1.91 ct stone contained two small “fingerprint” inclusions. Dr. Ruedisili also provided UV-visible absorption spectra of some of this material—obtained by Dr. Eric Findson of the University of Toledo, Ohio—which showed absorption maxima at 408.5, 422, 430, 461, 482.5, 504.5, 522.5, 565.5, and 612 nm. These gemological properties are similar to those given in a short article on this material by Dr. U. Henn ("Spessartine aus Pakistan," *Ztschrift der Deutschen Gemmologischen Gesellschaft*, Vol. 45, No. 2, 1996, pp. 93–94). Dr. Henn reports the composition of one stone (determined by electron microprobe analysis) as 85.26 mol.% spessartine, 10.13 mol.% almandine, and 4.61 mol.% garnet.
According to Dr. Ruedisili, this material first became known when a 19.30 gram specimen was discovered in the summer of 1993. A representative of the Remmy Company, Pakistan (partners with Ruedisili Inc.), saw some of the material at the Mineral and Industrial Development Corporation (MIDC), Azad Kashmir, government auction in April 1994. The “Kashmirine” garnets are found in a pegmatite in the Jauwai-Chojaw region (Neelum Valley) of Azad Kashmir. The deposit is about 177-200 km (110-124 miles) northeast of Muzaffarabad. Seven pegmatites are being explored for garnets and other gem materials. The main mine is in Jandranwala Nari pegmatite no. 1, which lies about 1.5 km northeast of Folawai Village, at an altitude of 2,590 m. The pegmatite occurs in a pegmatite complex, and averages 15 m by 2 m, so far, 143 m³ have been excavated. In addition to the spessartines, quartz and greenish black tourmaline have been found in the gem zone of this pegmatite. These same minerals occur in the neighboring Dongi Nari pegmatite, but the spessartite is not of gem quality.

Thirteen kilograms of spessartine were mined in 1994, and 16 kg in 1995. Approximately 20% of this production was suitable for cutting as cabochons or faceted stones. Most of the fashioned “Kashmirines” seen to date are in the 0.75-7.50 ct range, with a very few stones above 7.5 ct. The largest to date is about 30 ct. A joint venture between Ruedisili Inc., the Remmy Company, and the Azad Kashmir MIDC is being planned, in order to exploit the spessartines and other gem materials— including morganite, aquamarine, "mint green" and bicolored tourmaline, and topaz—from pegmatites in this region.

Activity continues at the Capiio do Lana “Imperial” topaz mine. Two mining sites near the historic city of Ouro Preto in Minas Gerais, Brazil, are famous for their production of fine Imperial topaz: Capiio do Lana and Vermelho. Currently, operations at the Vermelho/HCC/Alcan mining complex, on the outskirts of Ouro Preto, are temporarily halted because of a landslide on the boundary between two concessions, according to geologist Daniel Sauer, of Amsterdam Sauer, Rio de Janeiro, Brazil. However, the Capiio do Lana mine, in the Rodrigo Silva district, is fully operational.
Figure 13. Draglines stretch across the large open pit at the Capiio do Lana Imperial topaz mine. Large buckets suspended from the two lines scoop up the gem-bearing slurry from the bottom of the pit and drag it to the top for processing. Photo by Daniel A. Sauer, Amsterdam Sauer, Rio de Janeiro.

During an August 1996 visit to Capiio do Lana, Mr. Sauer and Gems & Gemology editor Alice Keller observed the mining operations at what are now two large open pits (approximately 30 m at the deepest point, covering a total area of about 5 ha, or 12 acres) next to one another in the 800-1,000 ha (2,000-2,500 acre) concession area. Using draglines (figure 13), bulldozers, and water cannons, miners recover an average of 11,000 m³ of mineralized rocks a month, which are processed to yield about 100 kg of topaz crystals. The estimated yield of fashioned stones from this material is 5,500 ct.

Approximately 50 people are currently involved in mining and processing the ore.

Typically, the material is heavily included, so totally clean crystals are quite rare. At the mine office, Mr. Sauer and Ms. Keller saw topazes in a wide range of colors—light yellow, orange-yellow, brownish orange, pinkish orange ("salmon" or "peach"), pink, reddish orange, orange-red, and purple or violet (figure 14)—all of which are marketed as Imperial topaz. The most sought-after are the intense pink, red, and purple stones, which—according to mine director Wagner Colombaroli—represent less than one-half of one percent of the total recoverable material. Although these colors occur naturally, many of the pink and red stones on the market today were produced by heat treating the original rough to remove the yellow component.

An article on gem topaz from the Capiio do Lana deposit is in preparation for an upcoming issue of Gems & Gemology.

ígems from Vietnam. Items on gems from Vietnam regularly appear in Gem News (see, e.g., Winter 1992, pp. 376-379; Summer 1993, p. 37; Fall 1993, pp. 211-212; Winter 1993, p. 285; and Fall 1994, p. 197). Vietnamese gems from many provinces were available at the 1996 Tucson shows. Mary Nguyen of Van Sa Inter national, Marina del Rey, California, showed one of the editors (MLJ) several examples of gem rough from Vietnam, including aquamarine and topaz from Thanh Hoa, topaz from the Darkel plateau, ruby and sapphire rough from Lai Yen, zircon and opal from Pleiku, unakite and citrine from Phan Thiet, and several materials from Lam Dong, among them tourmaline, peridot, petrified wood, chalcedony, and tektites. This extensive assortment suggests that Vietnam—a region with little previous history or lore in gemology—promises to be a rich source of gem materials for many years to come.

TREATMENTS
Coated quartz in "natural" colors. One of the techniques commonly used to change the apparent color of quartz is that of coating the sample with a colored material (see, e.g., "Tavalite," cubic zirconia colored by an optical coating," Summer 1996 Gem News, pp. 139-140). The most prominent example of this technology is so-called "aquas..."
aura” quartz, which owes its blue color to a thin surface coating of gold (Gem News, Winter 1988, p. 251; Fall 1990, pp. 234–235). At one of the 1996 Tucson shows, mineral dealer Rock Currier of Jewel Tunnel Imports, Baldwin Park, California, showed one of the editors (MLJ) several examples of coated quartz crystals in colors resembling natural quartz varieties. Four samples (figure 15) were borrowed for further study; these resembled amethyst, citrine, green “prasiolite” (heated amethyst), and red “strawberry quartz.” The coatings contained gold, bismuth, lead, chromium, titanium, and lesser amounts of calcium, potassium, and iron, as determined by EDXRF spectroscopy. As with the “aqua-aura” quartz, the coatings on these samples were too thin to affect the 1.54 R.I. value expected for quartz.

SYNTHETICS AND SIMULANTS

Flexible “crystal” fabric. A new product from the Swarovski Company of Wattens, Austria, is a flexible mesh fabric of faceted glass “crystals” (figure 16). The editors recently examined a sample of this “Crystal Mesh,” which consisted of 238 mounted foil-backed single-cuts, each about 2.7–2.8 mm in diameter, which (in our sample) occurred in three colors:
- Near-colorless (which had a weak-to-moderate, even light blue fluorescence to long-wave UV radiation)
- Light blue (fluorescing a slightly chalky, weak-to-moderate, even yellow-orange to long-wave UV and a moderate-to-strong, even light blue to short-wave UV)
- Medium dark blue (fluorescing a weak-to-moderate, even blue to long-wave UV and a moderate-to-strong, even blue to short wave UV)

All had the same refractive index, 1.578. When examined with the microscope, these single-cuts were mostly free of inclusions, although some contained scattered gas bubbles. Each single-cut was foil-backed and...
fastened with black glue into a black-painted metal cup; these cups alternated with black-painted metal rings, with one of four prongs on each cup folded through the adjacent rings and then glued shut with a brown rubbery material (figure 17). According to product information supplied by the manufacturer, "Crystal Mesh" can be machine-washed but not dry cleaned. Additional colors and patterns are available, and "Crystal Mesh" is available in pieces as large as 20 x 50 cm (about 8 x 20 inches).

Figure 18. Optical fiber glass—like the 2.85 ct cabochon (10.03 x 7.86 x 4.09 mm) on the left—is being produced in colors such as this specifically for gem use. A natural (heat-treated) tiger's-eye cabochon (1.83 ct, 9.61 x 7.96 x 3.67 mm) is shown on the right for comparison. Photo by Maha DcMaggio.

Fiber-optic glass imitation of tiger's-eye. Entries on fiber-optic glass (marketed under the names "Catseyte," "Cathaystone," and "Fiber Eye") appeared in the Summer 1991 and Summer 1994 Gem News sections. In the latter section, gray fiber-optic glass was discussed as a simulant of cat's-eye sillimanite from Orissa, India. Fiber-optic glass, which is most often created in fashioning to produce an extremely sharp chatoyant band, has been commercially available for some time in two colors: white and brown.

During the last few years, we have seen additional colors on the market: saturated yellow, pink, purple, black, and blue, as well as a more subdued "gummetal"-grayish blue that yields cabochons reminiscent of hawk's-eye quartz, a bright green similar to some cat's-eye diopside now coming from India, and a reddish orange similar to some heat-treated tiger's-eye (figure 18). The most recent material seen was "striped" in red/white/green, red/blue/green, and red/white/blue. According to a representative of Teton Gems, Boise, Idaho, the white and brown material previously available had been produced and rejected for laser or other technical applications. The newer colors, however, were produced specifically for their gem potential.

Gemological investigation of the 2.85 ct reddish orange glass cabochon shown in figure 18 revealed: 1.55 spot R.I.; 3.09 S.G.; moderate red appearance through the Chelsea filter; weak, dull red fluorescence to long-wave UV and strontium, chalky whitish yellow fluorescence to short-wave UV. Magnification revealed that the optic fibers have hexagonal outlines with a "honeycomb-like" structure similar to that seen in other fiber-optic glasses. Also seen, when looking parallel to the fibers, was a "speckled" color appearance, apparently the result of orange fibers intermixed with colorless ones.

Some of the cabochons displayed a somewhat less distinct chatoyant band than what has typically been encountered. According to the vendor, these stones had been cut as lower-domed cabochons to give the "eye" a more natural appearance.

MISCELLANEOUS JEWELRY

Versatile jewelry. A recurring trend in jewelry design is that of creating pieces in which the stones are interchangeable, enabling the wearer to adjust color schemes or "looks" with minimum effort. One sensitive contemporary example of this trend is a brooch and stickpin combination (figure 19) designed by Barbara Berk, of Foster City, Califonia. With this set of combination jewelry, the overall appearance can be easily altered by changing stickpins, in addition to the tourmaline, diamond, pearl, and gold wire pins pictured, the set that we saw contained citrine- and onyx-headed stickpins. Virtually any gem material could be adapted for this use.
THE DEALER'S BOOKS OF GEMS & DIAMONDS

By Menahem Sevdermish and Albert Mashiah, 1004 pp., illus., Kalmsl Printing House, Israel, 1996. US$98.00*

This two-volume work was first published in Hebrew in 1986 by Mada company. It is the first English edition.

Menahem Sevdermish is a Fellow of the Gemological Association of Great Britain, a founder and president of the Gemological Association of Israel, and the owner of a gemstone manufacturing and trading company. He has particular expertise in gemstone cutting and selling. Co-author Albert Mashiah also has impressive credentials. A long-time member of the Israeli precious stones industry, he has been a vice-president of the Israel Precious Stones and Diamonds Exchange, as well as vice-president of the Israel Emerald Cutters Association and the Gemological Institute of Precious Stones and Diamonds.

This book presents an interesting and difficult approach to a gemological text, in that it is written from the gem dealer’s point of view. The authors seem totally at home with the business aspects of diamonds and colored stones, as well as with assessing rough and cutting it for the market. In general, however, the two volumes of this work differ in effectiveness and value. The chapters on diamond are up-to-date and probably the best exposition of current practices in diamond cutting to be found anywhere in book form. The chapters on diamond grading and on judging value in colored-stone rough are very well planned and executed. However, the chapters on colored-stone identification leave something to be desired. Although many of the comments made from a dealer’s viewpoint are germane and useful to the reader, these chapters suggest that the authors have had little experience in colored-stone identification in a laboratory—or that the copyediting and proofreading were inadequate.

For example, in a discussion of spinel identification, the authors mention that glass would be distinguishable because it is amorphous and, thus, isotropic. As I am sure the authors are aware that both spinel and glass are isotropic, this is undoubtedly a fault of inadequate proofreading. They also state that “it is sometimes very difficult to decide if a stone is peridot or sinhalite, and only a chemical test will distinguish between them.” It is true, certainly, that for many years sinhalite was thought to be brown peridot, but the difference in spectra and the nature of the birefringence readily separates the two. In the identification table for the peridot section, they list the birefringence of sinhalite—which they refer to as DR—at exactly the same as peridot, and they also give the refractive indices, specific gravity, and hardness of the two as identical. Both the R.I. and S.G. for sinhalite are higher than those for peridot, and the beta index is closer to that of gamma.

Elsewhere, the explanation of what causes play-of-color in opal leaves the reader bewildered. It states, “When the spheres are uneven in size, the light reaching them is dispersed differently in each sphere, so that different colors are reflected. But, since the human eye is unable to see the minute spheres, the color coming from this layer will appear white because the human eye interprets a mixture of the spectrum as white.” These are just a few of a significant number of examples where careful editing would have been useful. Editing in the chapters related to diamond seems to have been more rigorous.

Yet there are also interesting chapters on subjects not found in other gem books, such as “A Commercial Analysis of the Geminestone” and “A Pre-purchase Cost Analysis.” In addition, there is a local account of the ingenious Robogem, a new instrument that analyzes rough to obtain maximum yield.

On the whole, the book is useful because it offers not only the exceptionally good diamond chapters, but also unique insights into other areas of the gem field. Although slightly flawed, the book seems, to this reviewer, a very worthwhile addition to any gemological library.

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Chairman of the Board
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NEW FRONTIERS IN DIAMONDS—THE MINING REVOLUTION

By David Duval, Timothy Green, and Ross Louthean, 175 pp., illus., pub. by Rosendale Press, London, 1996. US$55.00

History has shown that no single factor—other than the world economy—has affected the orderly functioning of the diamond industry more...
than the production and availability of rough. Today, exploration for diam-
monds is proceeding at a feverish pace on all continents except Ant-
arctica. In addition to De Beers, dozens of mining companies have
entered the arena. These include some of the world’s largest, who tra-
ditionally have been involved pri-
marily with metals. A very few appear to have been ostentatiously suc-
cessful, as evidenced by the antici-
pated 3 to 5 million carats of produc-
tion from one major new mine
[RHP/Dia Met] in Canada by the end
of this century.

Responsive to the interest that all
of this activity has generated, three
prestigious resource journals—from
two widely separated parts of the
world—have teamed up to review the
highlights of diamond exploration dur-
ing the last decade. The result is this
small book, which is divided into four
parts. Information is remarkably cur-
nent, as the February 1996 “agreement”
between De Beers and Russia is men-
tioned several times.

Part 1 [Green, London], “The
Global View,” reviews the special
and national, Northern Miner,
February 1996]. Nevertheless, this conveniently sized
book, which is divided into four
parts, is a short, statistics-based discussion
of the world retail trade (there is a
seemingly relentless rise in diamond
prices, and it is hoped—India).

Part 2 [Duval, Vancouver], “Can-
da: Joining the Big League,” is a nar-
tive-style review of the history of
diamond exploration in Kalimantan,
the island of Borneo. However, the ques-
tion as to who (if anyone) will be
Argyle’s heir is not answered.

Part 4 [Green, London], “Who
Sells Diamonds?” is a short, statistics-based discussion
of the world retail trade (there is a
seemingly relentless rise in diamond
jewelry sales). Green concludes that
the markets for diamonds in the
United States and Japan have matured.
Future additional production will be
consumed in East Asia (Taiwan,
South Korea, Thailand, eventually
China, and—it is hoped—India).

It must be noted that every
major topic in this book [including
most of the figures and all three
tables] can be found in articles that
have appeared recently in such
sources as [CK, Diamond Inter-
national, Norther Miner, literature
available from two investment (secu-
rity) companies, and this journal
Gemology J]. Those gemolo-
gists and jewelers who have kept
abreast of the professional literature
will have a sense of this era when they
read this fairly expensive review.

Nevertheless, this conveniently sized
compendium will be appreciated by
those who are just entering the field
or have not kept up with recent
events.

ALEXANDER D. LEVINSON, PH.D.
University of Calgary, Alberta, Canada

PHOTOGRAPHING MINERALS, FOSSILS AND LAPI 
DARY MATERIALS

By Jeffrey A. Scovil, 224 pp., illus., Connoisseur Press, Tucson, AZ, 1996, $40.00.

This important “how-to” book will be useful to anyone—beginners or profes-
sional—who wants to improve their photography of gems and min-
erals. It explains in plain language, for those not trained in this type of pho-
tography, the equipment and proce-
dures necessary. Line illustrations and photographic examples clearly
illustrate these lessons.

Specifically, the book describes
teritories, which led to the dis-
coration of the Lac de Gras Kimberlite
field by Charles Fuge in 1991. From
Duval’s discussion of the cur-
tent exploration activity by about a dozen
companies (most small and special-
tizing), it is clear that only the RHP/
Dia Met mine mentioned above
approaches commercial viability at
this time. The difficulties of explor-
ing for diamonds—especially the
heavy investments in time and
time necessary—become evident in
this chapter and the next.

Part 3 [Lockhary, Perth], “Aus-
talia: Any Heirs to Argyle,” is simi-
lar in scope to Part 2, except that it
places heavy emphasis on the Argyle
mine (its discovery, development,
corporate structure, marketing strate-
gies, and the crucial question of
whether it will be able to operate after
about 2000). Also discussed are many
of the companies presently exploring
in Western Australia, New South
Wales, and offshore, where rivers
drain the Argyle region empty into
the sea. Brief mention is also made of
exploration in Kalimantan, on the
island of Borneo. However, the ques-
tion as to who (if anyone) will be
Argyle’s heir is not answered.

Part 4 [Green, London], “Who
Will Buy My Beautiful Diamonds?”
is a short, statistics-based discussion
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COLORED STONES AND
ORGANIC MATERIALS


Recently, two mushrooms were discovered in amber of Tunennan age (90-94 million years old, mid-Cretaceous) in central New Jersey. One specimen is nearly complete, with an intact cap, distinct gills, and a central stalk (it is the oldest known such mushroom, by about 60 million years); the other is a wedge-shaped fragment of a mushroom cap. Both mushrooms resemble modern common leaf-litter and wood-decayer species, and both were growing on a cedar (a member of the Cupressaceae family).

Opal. extraLapis, No. 10, 1996, 96 pp. [In German].

Opal is the subject of another extraLapis, an issue of Lapis magazine that is devoted entirely to one gemstone. Following a comprehensive introduction by Edward Gubelin, a series of articles provide information on all important aspects of this colorful gem. An article by Max Weibel explains the origin of play-of-color. Two other papers describe Queensland's boulder opals, their forms, production, and prospecting methods (Wilson Cooper and Barry J. Neville); and the geologic setting and processes that led to opal formation in the sediments of Australia's Great Artesian Basin (Jack Townsvill). In disagreement with traditional theories of opal formation, Len Cram offers a surprising new model, based on ion exchange, that he demonstrated by growing synthetic opal out of "opal dirt" in a bottle in just three months. Jürgen Schütz describes the long history of Mexican opals, their varieties, and the present mining situation. Jochen Knigge recounts the history and production of opals from Pedro II, Piaui, Brazil. Klaus Eberhard Wild portrays another important locality—Kochshekler, near Ile-Otobong—with which [and perhaps still is] one of the most important centers of opal fashioning and trade worldwide. Jürgen Schütz explains the factors that determine the price of an opal (locality of origin, body color, play-of-color, etc.).

This section is designed to provide as complete a record as practical of the recent literature on gems and gemology. Articles are selected for abstracting solely at the discretion of the section editor and his reviewers, and space limitations may require that we include only those articles that we feel will be of greatest interest to our readership. Inquiries for reprints of articles abstracted must be addressed to the author or publisher of the original material. Opinions expressed in an abstract belong to the abstracter and in no way reflect the position of Gems & Gemology or GIA.

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It has generally been believed that agate formation in rhyolite intrusions (Bogen Schütz and Manfred Szykora); dote- bles, triplets, and opal mosaics [Kellar Fischer] and synthetic- ic opals and opal simulants [Martinez Szykora]. The volume concludes with a description of opalized fossils, amber- mosses, belemnites, and even dinosaurs [Alex Ritchie and Britigne Szykora].

This encyclopedia also contains short descriptions of smaller opal sources [Denmark, Vietnam, Indonesia, Mali], Saxony, Slovakia (formerly Hungary), Turkey, and the United States) and opal glasswears and the stories of the Hope and El Agua Aztita opals [by John S. White]. Two stories tell us how Australian aborig- ines explain the origin of opals.

A volume about opals with words only, in this case, German would be utterly frustrating. To this edition, however, the stunning beauty and incredible variability of opals is illustrated throughout the entire volume by wonderful color photographs.


It has generally been believed that agate formation in rhyolite and pumice glasses takes place at high temperature. Recent research by M. Landweber has shown that agate may form in other types of rocks at lower temperatures. In this article, Mr. Rykart proposes a new theory for the for- mation of star-shaped agates in lithophytes (e.g., thundereggs) in volcanic volcanites. The basic idea is that the gas bubbles in the magma contract to form polygonal cavities because of the high gas pressure during cooling near the Earth's surface. Subsequently, monomer H4Si04, dissolved in the water that invades the cavity, fills them with clays, diatoms, and quartz. The surrounding rhyolites devitrifies and hardens, forming the well-known quartz porphyry balls. RT

DIAMONDS

Aber Resources Ltd. Diamond Industry Week, February 26, 1996, p. 3.

Aber Resources has announced results from drill cores at A-418, core of its kimberlite pipes in the Northwest Territories, Canada. Nineteen tons of core in a large disseminated 10 inches, about 15 cm) core drilled through 367 m of kimberlite yielded 83.1 carats of diamonds—4.3 carats per ton of ore—with individual stones between 0.025 and 3 carats. The largest "petty" diamond weighed 2.2 carats. The A-418 pipe is estimated to contain 15 million tons of ore, at a valuation of $58.17 per carat, bulk sampling is proceeding at the A-154 South, A-154 North, and A21 kimberlites. MLJ


On Earth, diamonds usually occur in rocks derived from the mantle. They are thought to have formed from fluids or melts in the upper mantle at "immense" temperatures and pressures, probably—according to Mr. Koeberl—during several diamond-forming events early in the Earth's history. Diamonds have also been produced, directly on the Earth's surface, during meteorite impacts. Such dia- monds may have formed, at least in one case (Ries Crater), from the vapor phase. Diamonds found in iron meteorites and ureilites (another variety of meteorite) were formed by shock from graphic amorphous car- bon, probably in the meteorite and not after it arrived on the Earth. Nanometer-size diamonds have been found in chondritic meteorites, associated with noble gases (argon, xenon, etc.) with unusual isotopic compositions, these came from interstellar or presolar events very early in the history of the solar system. Small diamonds have also been found in clays marking the boundary of the Cretaceous and Tertiary periods, which has evidence of a large impact. Their carbon and nitrogen isotope point to an origin within the impact event or the resulting fireball. Polycrystalline diamonds up to 1 cm, discovered in impactites at a few Russian and Ukrainian impact struc- tures, also appear to be crustal in origin. Impact-produced diamonds are very different from microdiamonds found in high-grade metamorphic rocks. Also rare and unusual are polycrystalline black dia- monds, called carbonados. No carbonados diamond has ever been found in situ in a rock. Possible origins include carbon subduction in the mantle, shock metamorphism during impact, or irradiation of organic matter. Vapor- deposition may also be a candidate. MLJ


The first diamond mine in Wyoming is about to begin pro- duction, and many more kimberlites and lacustrines in the tectons may contain diamonds, according to Dr. Dan Hazard of the Wyoming State Geological Survey. Redaurum Red Lake has just finished construction of a 1/2-ton-per-hour ore-processing plant for the company's Kelsey Lake diamond property, along the Colorado-Wyoming state line; several gem diamonds, up to 14.2 ct, already have been recovered from this property. The Colorado-Wyoming kimberlite province includes more than 100 kimberlite intrusions, one of the world's largest lamproite fields, and dozens of unex- plained geophysical and geochemical anomalies. More than 12,000,000 diamonds have been recovered from this area in the last 20 years.

No history of diamond mining in Wyoming is complete without mention of the "Great Diamond Fuss of 1872." A
sandstone outcrop was salted with 10 pounds of uncut dia-
monds and rubies (purchased in London), plus another 50
pounds of garnets and chrome-rich diopsides from Arizona.
(At the time, sandstone was thought to be a host rock for dia-
monds, no doubt based on the many alluvial diamond
soiLs then known.) This hoax helped provoke passage of the
Mining Law of 1872, which established the first mine-
patenting regulations in this country. Full details of the
story may be found in the Wyoming Geological Association's

Are euhedral microdiamonds formed during ascent and
decompression of kimberlite magma? Implications
for use of microdiamonds in diamond grade esti-
mation. D. R. M. Pattison and A. A. Levinson,
The authors answer this question with "Yes, quite likely
in some cases." Before they proceed with their explana-
tion, they first state clearly that this paper only discusses
the generation (at different times) resorption of existing macro-
diamonds (resorbed to extinction) and only euhe-
dral microdiamonds. Thus, the relationship between the
population of macrodiamonds and that of euhedral microdia-
monds is not simple and direct.

The implication is that the use of microdiamonds in
estimating the overall diamond content in a pipe (which
is widely done in Canada, where pipes are hauled and only
drift core samples are available for testing) is not straight-
forward. In several cases, this method can give an incor-
rect answer. Diamond exploration companies' argument
that the method does work rests on their use of the ration
of euhedral, resorbed, and cubic microdiamonds, as well
as broken and fragmented crystals. However, this privi-
leged information is not readily available in published form, so it
is impossible to confirm their arguments.

The authors note the paucity of published research
into nitrogen content and the aggregation and carbon-isot-
otope composition of microdiamonds, by which their
hypotheses could be tested. Very recently, a number of
papers (by researchers such as Milledge, Mendelsohn,
Taylor, and Pillinger, among others) have addressed these
subjects, but not the specific issue of euhedral microdia-
monds. However, this interesting review and thought-pro-
voking hypothesis may give a welcome impetus to the release of more information on microdiamonds and their
usefulness in estimating diamond content.

Bram Janse

French Guiana diamonds. Mining Journal, London,

Diamonds have been found in a metamorphosed ultra-
omatic rock in the Dachone permit area, Iniria, French
Guiana. Golden Star Resources, Curionite Resources SA,
and Lakefield Research have recovered 3,748 microdia-
monds from over 113 kg of host rock, also, 8 microdia-
monds (the largest being 2.4 mm) have been recovered
from 1.8 tons of rock altered to saprolite.

ODM: Namibian gem. Mining Journal, London,

Several companies have licenses to mine the diamond-
rich offshore deposits along the Namibian and South
African coasts: De Beers Maritie (which produces the
most stones at this time, about 0.5 million carats),
Namibian Minerals Corp., Diamond Fields Resources,
BHP, and Ocean Diamond Mining [ODM], plus a few oth-
er. Copyrite-based ODM is in its "important, but under-
reported" company operating in the region. ODM cur-
rently works in two main areas: It has production around
the 12 Penguin Islands, and exploitation along South
African concession 7B and deep-water concessions 6c and
14c [in a joint venture with Benguela Concessions].
Production in fiscal 1995 was about 40,000 carats at an
average price of US$300 per carat.

GEM LOCALITIES

Alkaline basalts and associated volcaniclastic rocks as a source
of sapphire in eastern Australia. G. M. Oakes, L. M.
Barron, and S. R. Lishman, Australian Journal of
The major sapphire deposits in eastern Australia are allo-
tial, they occur in recent drainage systems where Tertiary
alkali basaltic volcanic rocks dominate the present surface.
As a result, it has been generally accepted that these
basalts are the immediate source of the sapphires. The
enigma is that sapphires are only rarely found in these
rocks. This paper discusses recent developments in the
understanding of sapphire occurrences in eastern
Australia and reviews several possible origins for these
deposits.

During field studies, it was observed that the sapph-
ires are associated with volcaniclastic rock units, now
mostly altered to clay minerals, that are common within
Australian diamonds are unusual; at present, their origin rising through the mantle transition zone. Eastern Australia and possibly in similar geologic provinces elsewhere (e.g., Southeast Asia). Alkaline rocks and gemstones, Australia: A review and synthesis. F. L. Sutherland, Australian Journal of Earth Sciences, Vol. 43, No. 3, 1996, pp. 323-343.

Valuable gemstones that occur in Australian alkaline rocks include diamonds in lamproites and kimberlites, sapphires, zircons, and rubies in alkali basalts and one gem zircon prospect in carbonatite. This paper reviews prime exploration targets for alluvial sapphires (and associated minerals such as zircon and sapphire) in eastern Australia and possibly in similar geologic provinces elsewhere.

There are marked contrasts between diamond and sapphire-zircon associations across the continent. Most western cratonic areas exhibit episodic, sparse, deep-alka-line activity from the diamond zone (2 billion-20 million years old). However, in eastern fold-belt areas, pro- lific Mesozoic/Cenozoic basaltic volcanism carried up considerable amounts of sapphire and zircon (since 170 My). Some South Australian Mesozoic lamproilite d–zircon associations across the eastern Australian craton are unexplained by present-day processes. Several different models compete to explain sapphire/zircon formation in eastern Australia. These range from eruptive plucking of metamorphosed sub–continental crust to fractionation of felsic melts to carbonatitic reactions. Pb-U isotopic zircon ages favor formation from fractionated basaltic melts, but no production figures have been found in the eastern Australian craton. Furthermore, the tectonic settings and origins of Australia's gem-bearing alkaline rocks are in dispute. Several different models compete to explain sapphire/zircon formation in eastern Australia. There are marked contrasts between diamond and sapphire-zircon associations across the continent. Most western cratonic areas exhibit episodic, sparse, deep-alka-line activity from the diamond zone (2 billion-20 million years old). However, in eastern fold-belt areas, pro- lific Mesozoic/Cenozoic basaltic volcanism carried up considerable amounts of sapphire and zircon (since 170 My). Some South Australian Mesozoic lamproilite d–zircon associations across the eastern Australian craton are unexplained by present-day processes. Several different models compete to explain sapphire/zircon formation in eastern Australia. These range from eruptive plucking of metamorphosed sub–continental crust to fractionation of felsic melts to carbonatitic reactions. Pb-U isotopic zircon ages favor formation from fractionated basaltic melts, but no production figures have been found in the eastern Australian craton.

AAI


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This paper presents the results of microthermometry, SEM, and Raman probe examination of emeralds and gangue minerals, as well as the first sulfur isotope data on pyrite, from seven Colombian emerald deposits. Also discussed is the origin of the hydrothermal fluids that carried the carbonate-pyrite-emerald vein mineralization in Colombia.

The fluid-inclusion study shows the presence of homogenous and hypersaline brines; the isotope study suggests a uniform sulfur isotopic composition for the fluids and a heavy, probably unique, small-sulfur source for emerald deposits of both the western and eastern emerald zones. Considering the presence of salt deposits and gypsum deposits in the area, the authors conclude that "the only likely and unique source of sulphur might be derived from evaporites through a sulphate reduction process in the site of mineral precipitation." These results, together with those of previous studies, support the interpretation of hydrothermal emerald mineralization in a sedimentary environment. In that respect, the Colombian emerald deposits differ dramatically from almost all other emerald sources.


Mining for black opals consists in the area of New South Wales, Australia. Although Lightning Ridge proper is the most popular tourist destination, the author visited the more remote opal fields at Grawin, Glenarrayan, and Sheep Yards. Most of the miners in the Grawin and Sheep Yards area dig down 9-12 m to reach the working level. Prospecting entails two stages: A drilling rig makes a 17.5-cm-diameter test hole down to the working level; if the hole looks promising, a 1-m-diameter shaft is drilled after the claim is registered.

Most of the opal mined is "potch," or common opal, usually gray, "amber," or black; a black-and-white variety is known locally as "magpie" potch. Only 5% shows play-of-color, and only 5% of that (0.25% altogether) is classified as "precious gem quality" opal. Gem opal sitting on black potch is the most desirable.

The Grawin opal fields were discovered in 1907, and all three opal fields (Grawin, Glenarrayan, and Sheep Yards) have been extensively mined. However, amateur collectors as "precious gem quality" opal. Gem opal sitting on black potch is the most desirable.

In June was a bumper month for London jewelry sales, Christie's and Phillips both did well. Christie's June 18 sale was the greater success, with a £4,299,97 total. Buyers from 26 counties competed actively on the phone, in the room, and in the book; more than 60% were private clients.

A pair of rare antique Indian diamond briolettes more than doubled its estimate, to sell to the trade at £115,500. An antique emerald, diamond, and pearl necklace, estimated at £30,000-£35,000 brought in £49,500, and a fancy intense yellow diamond ring—estimated at £40,000-£50,000—sold to an anonymous buyer for £122,500. A typical Belle Epoque garland-style diamond necklace brought an extraordinary £73,000. The overflow from these refined Christie's sales goes to the ever-popular Christie's South Kensington, which boosted its best-ever sale on June 18.
Phillips's fine jewelry sale on June 27 was highlighted by a group of 19th-century jewels by French gothic-revival goldsmith Louis Wiese. A heavy neo-gothic bangle, mounted with a large cabochon sapphire and flanked by leopard heads, went above estimate for £21,850. A gold lozenge-shaped brooch with matching ring, again set with cabochon sapphires and pearls, sold for £6,670. A heavy neo-Renaissance gold, cameo, and pearl locket and chain made £26,450.

Buyers also showed a good appetite for old-cut Victorian diamonds. A diamond riviere necklace from about 1880 sold for £10,350, more than doubling its estimate. A late-Victorian pearl and diamond bangle, estimated at £1,200-£1,500, sold for £3,220.

Although not mentioned in this comprehensive article, the possible consequences for gemology (e.g., the coating of gems, especially diamond simulants) should be kept in mind.


Researchers James Adair and Ravit Singh at the University of Florida (and their coworkers and industrial collaborators) have made the largest synthetic diamond known to date—about 1600 carat. It was grown by a low-temperature (to as low as 500°C) vapor-deposition technique; color and clarity were not specified, but its dimensions (11 inches [about 28 cm] in diameter and 1.5 mm thick) would seem to preclude any gemstone application. The prospects for large-scale diamond coatings look very promising, however.


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SYNTHETICS AND SIMULANTS


Theorists have predicted a structure of carbon nitride that should be harder than diamond, and experimentalists are trying to make it. The predicted material, cubic C$_3$N$_4$, has the same structure as Si$_3$N$_4$. Attempts to make this compound by vapor deposition have been hampered by the difficulty in getting enough nitrogen into the material, graphite-like structures and films with triple-bonded (acetylene-like) carbon form instead. Annealing of the latter gets rid of the triple bonds and produces a film that has diamond-like electrical properties but much lower hardness than diamond thin films. It is possible that "super-hard" C$_3$N$_4$ can be produced by high-pressure synthesis methods.


Chemical vapor deposition (CVD) of diamond films from activated gas phases was first achieved in 1952-53, but only since 1983 has it become an important and rapidly developing field of research. This paper summarizes the principles, results, and perspectives of CVD technology. It starts with a description of the conditions in which diamond thin films crystallize from gas phases and the many deposition processes now available. The growth of textured and hetero-epitaxial diamond films on different substrates, especially silicon, is discussed and illustrated with scanning electron micrographs. The author then outlines the many applications of diamond films (these high-frequency electronic devices to optical filters to membranes for loudspeakers) and gives an outlook on their future potential.