

EDITORS

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Figure 1. These three blue diamonds (0.42, 0.55, and 2.03 ct), which were color graded Fancy Light blue (marquise) and Fancy Intense blue (triangular and pear shapes), showed unusual phosphorescence in the DiamondView.

DIAMOND Blue Diamonds Showing Multiple Colors of Phosphorescence

Recently, three blue diamonds—a 0.42 ct marquise, a 0.55 ct triangular step cut, and a 2.03 ct pear shape (figure 1)—were submitted to the East Coast laboratory for diamond grading (0.42 and 2.03 ct) and identification and origin (0.55 ct) reports. The 0.42 ct diamond was color graded Fancy Light blue, and the 0.55 and 2.03 ct diamonds were graded Fancy Intense blue. Gemological examination and photoluminescence spectra proved that the three stones were natural-color diamonds. No color zoning was observed. There was no reaction to

long-wave ultraviolet (UV) radiation, and the 0.55 and 2.03 ct samples fluoresced very weak yellow to standard short-wave UV; no phosphorescence was observed.

Since phosphorescence in the Diamond Trading Company (DTC) DiamondView can be a useful means of separating natural from synthetic blue diamonds, as part of our examination we used this instrument to excite the samples with high-energy short-wave (<230 nm) UV radiation for five seconds at room temperature. After the UV source was turned off, individual images were taken with 1, 4, and 10.2 seconds of camera exposure time at different time delays (from 0.1 to 10 seconds) in an attempt

to record the decay of the phosphorescence. We were surprised to see different colors of phosphorescence at various time delays in all three diamonds (see, e.g., figure 2): blue phosphorescence from 0.1 to 1 second, purplish blue and pink after 2 seconds of delay, and orangy red and red between 5 and 10 seconds of delay. The latter colors

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were easier to see with longer exposure times (again, see figure 2).

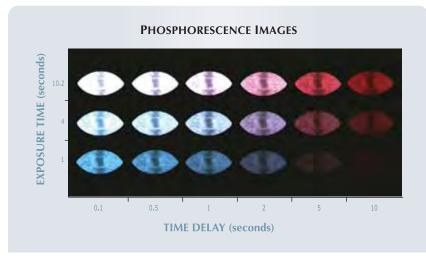
Generally, natural blue diamonds are observed to phosphoresce blue to green or, rarely, red and orangy red, whereas synthetic blue diamonds usually phosphoresce blue or sometimes orange (J. M. King et al., "Characterizing natural-color type IIb blue diamonds," Winter 1998 Gems & Gemology, pp. 246-268). S. Eaton-Magaña et al. ("Luminescence of the Hope diamond and other blue diamonds," Fall 2006 Gems & Gemology, pp. 95-96) recently reported that the phosphorescence spectra of natural blue diamonds typically show a blue-green band centered at 500 nm and an orange-red band centered at 660 nm. The variation in relative intensities of these two bands, along with their respective decay times, creates the different colors of observed phosphorescence. Thus, although orangy red phosphorescence may be seen only rarely by visual observation, it is almost always present.

K. Watanabe et al. ("Phosphorescence in high-pressure synthetic diamond," *Diamond and Related Materials*, Vol. 6, No. 1, 1997, pp. 99–106) reported observing a change in phosphorescence colors from blue to orange in synthetic blue diamonds. However, the latter hue was not the orangy red phosphorescence seen in natural diamonds; nor did the luminescence peak reported for this color (2.1 eV or 590 nm) match that for the red phosphorescence in natural diamonds (660 nm). Our ongoing DiamondView studies indicate that natural blue diamonds usually show consistent phosphorescence colors, either blue or red, although sometimes in slightly different hues of each during the decay period. However, these three blue diamonds showed multiple colors of phosphorescence: Combinations of the colors blue and red were observed as the two peaks decayed at different rates.

Interestingly, photoluminescence spectra of the three diamonds did not show the 776.5 nm peak that has been associated with red phosphorescence in natural blues (Fall 2005 Lab Notes, pp. 258–259). The phenomenon of multiple phosphorescence colors is rare in natural blue diamond, but it can be explained by the presence of the 500 and 660 nm luminescence bands, and the fact that the 500 nm band decays before the 660 nm band.

Kyaw Soe Moe and Paul Johnson

Figure 2. DiamondView images of the 0.42 ct blue diamond, taken with various camera exposure times and time delays, reveal a progression of phosphorescence colors. The phosphorescence color changed from blue to red as the decay time increased from 0.1 to 10 seconds.



Diamond with "Etched Dislocation Loops"

While gemologists are familiar with common diamond clarity characteristics such as cleavage cracks (feathers) and mineral inclusions (crystals and pinpoints), microscopically visible features that appear to have resulted from structural dislocations are much more unusual. The West Coast laboratory recently received for grading a nearcolorless round brilliant-cut diamond that fell into this "unusual" category. What made this diamond interesting was a formation of filamentary curved loops clearly visible through a gemological microscope (figure 3). These loops seemed to defy the monocrystalline structure of gem-quality diamonds, where angular and straightedged internal features are the norm and distinctly curved structural features are unexpected.

Dislocations are defects in the crystal lattice structure of a mineral. Whether natural or synthetic, truly perfect crystals are almost unknown, and dislocations are very common. This is as true of diamond as it is of any other crystalline material.

It is also well known that diamonds often undergo plastic deformation in the earth; linear surface graining and internal graining are visual manifestations of this. During diamond growth, plastic deformation as well as the precipitation of microscopic-to-submicroscopic foreign matter on growing surfaces can increase dislocation density, and the presence of a dislocation can itself cause the development of additional dislocations.

Most individual dislocations cannot be seen with an optical microscope unless their density in a particular area is sufficiently high. In that case, they may produce visible strain in their host or, if they are subjected to etching and become hollowed out, may scatter light and appear white. The curved features in this diamond seem to fall into the latter group, in that they appeared white when examined through a gemological microscope. This contributor welcomes any alternate hypothe-

48 LAB NOTES GEMS & GEMOLOGY SPRING 2007



Figure 3. Microscopically visible filamentary "dislocation trails," like the curved loops shown here, are very unusual internal features in diamond. Magnified 15×.

ses that might explain these unusual distinctly curved features that are sometimes seen in diamonds.

John I. Koivula

HPHT-Annealed Yellow-Orange Diamond with a Strong 480 nm Absorption Band

Diamonds with a strong absorption band centered at 480 nm usually show chameleon behavior or a yellow-orange coloration with a brown component of varying saturation. Typically, they also show strong yellow to yellow-orange fluorescence to both long- and shortwave UV radiation and weak yellow phosphorescence to short-wave UV. Little is known about the defect(s) responsible for this absorption band and the typical luminescence features, but-unlike many other defects-the artificial inducement of this defect (or defects) has never been reported. In addition, significant concentrations of this defect are only rarely seen in colortreated diamonds.

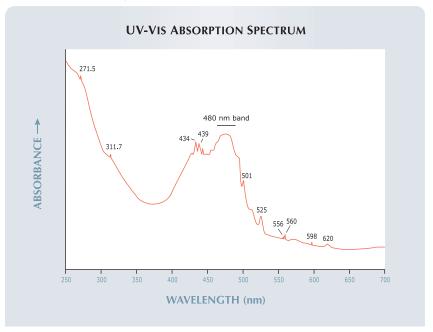
The East Coast laboratory recently examined a known high-pressure, high-temperature (HPHT) annealed diamond with a strong 480 nm absorption band, which provided a special opportunity to examine the response of this feature to HPHT annealing. This 0.88 ct emerald-cut was color graded Fancy Intense yellow-orange (figure 4). The color distribution was patchy, and some areas were more intensely colored than others, with clear and distinct boundaries. Observation with the microscope revealed many tiny pinpoint inclusions in a linear arrangement; some pinpoints had surrounding haloes. Strong linear graining dominated in one direction throughout the entire stone, a good indication that the diamond had a strong brown hue before HPHT treatment. In contrast to most diamonds with the 480 nm absorption band, this stone displayed strong orange fluorescence to long-wave UV radiation and a moderately strong orange reaction to



Figure 4. The absorption band centered at 480 nm in the visible spectrum of this 0.88 ct HPHT-annealed Fancy Intense yelloworange diamond (figure 5) is rarely seen in a treated diamond.

short-wave UV, and no phosphorescence was observed. The fluorescence image seen when the diamond was exposed to the high-energy short-wave

Figure 5. The UV-Vis absorption spectrum of the diamond in figure 4 showed a strong, broad 480 nm band despite HPHT annealing. The band centered at ~270 nm and the peak at 271.5 nm are due to isolated nitrogen. The spectrum also showed several other sharp absorption features that could not be assigned to particular defects.



UV radiation of the DiamondView was dominated by a strong orange color with many unevenly distributed sharp, parallel green lines.

The UV-Vis absorption spectrum (taken at -196°C) showed a strong and broad band centered at 480 nm, in addition to some sharp peaks in the 430-600 nm range. There were many moderately strong and broad bands in the 420-530 nm region, as well as one at ~620 nm (figure 5). A broad band centered at ~270 nm and a sharp peak at 271.5 nm were due to trace isolated nitrogen, a common feature of this type of natural diamond that can be additionally enhanced by HPHT treatment. The infrared absorption spectrum showed a weak peak at 1332 cm⁻¹ and a relatively strong peak at 1085 cm⁻¹; the assignment of the latter peak is unclear. In contrast, the IR absorption spectra of natural-color diamonds with a 480 nm absorption band usually have some unassigned peaks in the 1330-1100 cm⁻¹ region.

Study of this interesting diamond strongly indicated that the defect(s) responsible for the 480 nm absorption band and yellow or yellow-orange fluorescence persisted through HPHT treatment. Despite the differences in the IR absorption spectra and the absence of phosphorescence, identification of a diamond showing the 480 nm band as HPHT-treated could be a challenge in the laboratory.

Wuyi Wang

Translucent Greenish Yellow Diamonds

Translucent diamonds bearing microinclusions are not encountered often in the laboratory, but they have been reported from time to time. As the popularity of colored diamonds increases, we are seeing a broader range of them with this feature (see, e.g., the following Lab Notes: Winter 2004, pp. 325–326; Summer 2005, pp. 165–167; and Spring 2006, pp. 57–59). We recently examined two large diamonds with such micro-inclusions, and it appears that both were cut from the

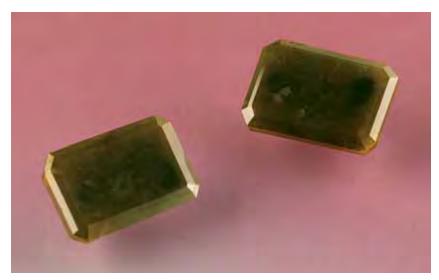


Figure 6. These two translucent greenish yellow diamonds—10.95 ct (left) and 11.40 ct (right)—have some unusual inclusions as well as gray zones on their tables that seem to mirror each other.

same piece of rough.

The two translucent greenish yellow stones (10.95 and 11.40 ct; figure 6) were submitted to the East Coast laboratory for identification reports. Microscopic examination revealed small black inclusions and numerous fractures, some of which contained orange

stains (figure 7). We also saw gray zones, which were darkest in their centers, on the table facets (again, see figures 6 and 7). When observed in the DiamondView instrument with highenergy short-wave UV radiation, these gray zones corresponded to growth sectors (figure 8). The fluorescence images

Figure 7. Numerous orange-stained fractures and small black inclusions (graphite) were present in the two diamonds (left, magnified 30×). Note how this irregularly shaped gray zone is darker in the center (right, magnified 15×).





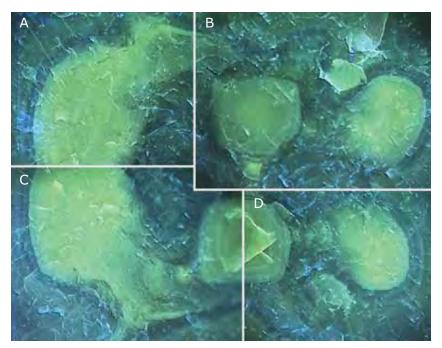


Figure 8. When the table facets were examined in the DiamondView, the gray zones (see figures 6 and 7) proved to be growth sectors. Note how they are indeed mirror images of each other in the two stones (A and B = 11.40 ct; C and D = 10.95 ct). All the growth sectors originated from cubic growth and later developed into octahedral growth.

suggests that high internal pressure was trapped in these diamonds during growth in the earth's upper mantle (see M. Schrauder and O. Navon, "Hydrous and carbonatitic mantle fluids in fibrous diamonds from Jwaneng, Botswana," *Geochimica et Cosmochimica Acta*, Vol. 58, 1994, pp. 761–771).

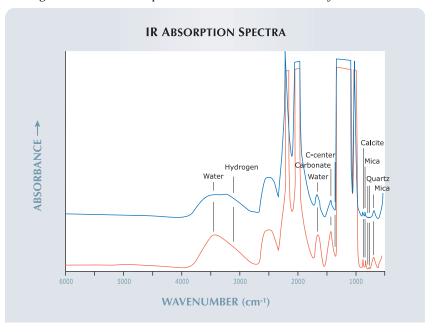
The fibrous texture is not associated with this micro-inclusion assemblage. As seen in figure 8, the cubic growth developed into octahedral growth at later growth stages. A diamond showing a similar growth sequence was reported in the Spring 2006 Lab Note mentioned above. The yellow color, which was unevenly distributed throughout the two diamonds, was caused by C-centers (i.e., single substitutional nitrogen, at 1344 cm⁻¹).

Diamonds such as these create special concerns during cutting. It is recommended that a diamond with two growth sectors be sawn between the two sectors to avoid causing cracks (see G. P. Bulanova et al., "A venture into the interior of natural

of the two diamonds actually mirrored each other, which suggests that they were cut from the same crystal.

The small black inclusions were identified as graphite by Raman spectroscopy. The micro-inclusions were identified as water, carbonates, calcite, quartz, and micas by Fourier-transform infrared spectroscopy (figure 9). The broad OH-stretching (3600-2800 cm⁻¹) and the HOH-bending (~1650 cm⁻¹) bands indicated the presence of different water phases and suggested that these diamonds were formed progressively through different growth events (see D. A. Zedgenizov et al., "Water-related IR characteristics in natural fibrous diamonds," Mineralogical Magazine, Vol. 70, 2006, pp. 219-229). The quartz bands at 808 and 783 cm⁻¹ were shifted from their normal positions of 798 and 778 cm⁻¹. The shifted positions correspond to a residual pressure of 1.5 GPa (at room temperature) inside the diamond; this

Figure 9. The mid-infrared spectra of both diamonds (top, 11.40 ct, bottom, 10.95 ct) revealed the presence of numerous micro-inclusions. The different modes of the water bands suggest that they were formed by different growth events. The spectra have been offset for clarity.



diamond: Genetic information and implications for the gem industry [Part I: The main types of internal growth structures]," *Journal of Gemmology*, Vol. 29, 2005, pp. 377–386). Likewise, the elevated temperatures used for cutting can release the high pressure trapped inside fluid-bearing micro-inclusions, which could also have contributed to the numerous fractures in these two diamonds.

The DiamondView images and infrared spectra proved that the diamonds were natural, with natural color. The micro-inclusions were formed from the trapped fluids in which the diamonds grew (again, see Schrauder and Navon, 1994). Even though these diamonds were translucent, their micro-inclusions and growth sectors provided us with valuable geologic information related to the growth environment of diamond crystals and the challenges that crystals grown in some environments can pose for the cutting process.

Kyaw Soe Moe, Paul Johnson, and HyeJin Jang-Green

Unusual Natural-Color Black Diamond

Black diamonds in the market today commonly receive their color from heat treatment at ambient pressure or intense irradiation. These treatments create either graphite inclusions or absorption centers (from the damaged diamond lattice) that strongly absorb visible light and result in a black appearance. In comparison, we see very few gem-quality black diamonds of natural color in the lab, and the natural color is usually ascribed to graphite inclusions (R. C. Kammerling et al., "An investigation of a suite of black diamond jewelry," Winter 1990 Gems & Gemology, pp. 282-287) or metallic inclusions such as magnetite (S. V. Titkov et al., "An investigation into the cause of color in natural black diamonds from Siberia," Fall 2003 Gems & Gemology, pp. 200-209). Recently, the East Coast laboratory examined a 3.14 ct natural-color black

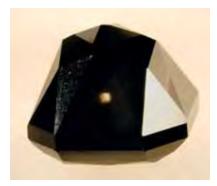


Figure 10. This 3.14 ct black diamond is colored by extremely dense clouds of micro-inclusions. The small "window" on the pavilion is an area that did not contain inclusions.

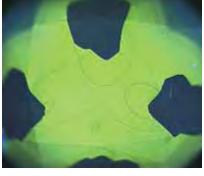


Figure 11. When exposed to highenergy short-wave UV radiation in the DiamondView, the black diamond in figure 10 exhibited typical greenish yellow fluorescence in its cubic growth sectors.

cut-cornered rectangular brilliant diamond (figure 10) that showed some interesting gemological features and yet another cause of color.

Unlike most natural diamonds, in which octahedral growth is well developed, this black diamond was dominated by cubic growth and had a high concentration of hydrogen. These cubic growth sectors exhibited greenish yellow fluorescence to short-wave UV radiation in the DiamondView instrument (figure 11). They also contained dense clouds of tiny inclusions that were readily visible with magnification. These small inclusions were evenly distributed in the cubic growth sectors and absorbed almost all the light entering the diamond. As a result, it was graded Fancy black.

Micro-inclusions occur frequently in natural diamonds and normally induce an overall brownish or grayish hue (see Winter 2004 Lab Notes, pp. 325–326; Spring 2006 Lab Notes, pp. 57–59; and Spring 2006 Gem News International, pp. 73–74), but a resulting black appearance is rare (when examined with very strong lighting, areas along the edges of the octahedral growth sectors were seen to be dark brown). The spectrum in the midinfrared region for this diamond displayed extremely strong hydrogen-related absorptions (e.g., 3107 cm⁻¹), a com-

mon feature of natural diamonds with micro-inclusions that are restricted to certain growth sectors. The mineralogy of these tiny inclusions is not well known.

Another unusual feature of this diamond was its poorly developed octahedral growth sectors. These were much smaller than the cubic sectors, and contained almost none of the micro-inclusions. As a result, these transparent areas formed small, symmetrical colorless "windows" in the diamond (figures 10 and 12).

The well-developed cubic growth

Figure 12. Unlike the cubic growth sectors, the black diamond's octahedral sectors contained no micro-inclusions, and these areas consequently formed transparent "windows" through the stone. Magnified 45×.



pattern, unusual inclusions, and interesting color distribution combined to make this natural-color black diamond gemologically interesting.

Chincheung Cheung and QianWen (Mandy) Liu

SYNTHETIC DIAMOND, Possibly Grown at Higher Temperatures

The gemological features of most synthetic diamonds show little variety. Many articles have been written on the subject, and most of the features are quite familiar to gemologists, which is why a synthetic diamond recently submitted to the East Coast laboratory was so interesting.

Standard testing procedures established that the 1.35 ct cut-cornered rectangular modified brilliant in figure 13 was a synthetic diamond. The sample was color graded Vivid yellow, which in itself was unusual, as most HPHT synthetic diamonds we see have an orangy component to their color. Examination with 60× magnification did not reveal any large metallic inclusions, but numerous pinpoint-like micro-inclusions were present. Also seen was a subtle yellowish patchy color zoning in a very limited region in the center; this is not common in yellow synthetic diamonds. When illuminated with a fiber-optic light, the sample showed unevenly distributed moderate green luminescence that corresponded to the color zoning (figure 14). Notably absent was the characteristic hourglass-like color zoning seen in most HPHT-grown synthetic diamonds (see J. E. Shigley et al., "An updated chart on the characteristics of HPHT-grown synthetic diamonds," Winter 2004 Gems & Gemology, pp. 303-311). Also, in contrast to type Ib synthetic diamonds, which are inert or display weak fluorescence when exposed to long-wave UV radiation, this synthetic diamond displayed unevenly distributed moderate-tostrong green fluorescence. Furthermore, also unlike most other synthet-



Figure 13. The Vivid yellow color of this 1.35 ct cut-cornered rectangular modified brilliant is unusual for a synthetic diamond.

ic diamonds, the intensity of the fluorescence was *weaker* when exposed to short-wave UV.

Observation with the Diamond-View instrument revealed an internal growth-sector arrangement showing well-developed octahedral growth zonation and a four-fold cross-shaped pattern from very poorly developed cubic growth sectors (figure 15).

In further contrast to typical HPHT-grown synthetic yellow diamonds, which are dominated by isolated nitrogen, the nitrogen in this sample was highly aggregated. Its midinfrared absorption spectrum was dominated by A-form nitrogen (>200 ppm as shown by the 1282 cm⁻¹ peak), and only an extremely weak absorption due to isolated nitrogen at 1344 cm⁻¹ was detected (figure 16). When viewed with a handheld spectroscope, a weak but sharp band at 546 nm was observed, which is evidence of nickel impurities. Many other Ni-related defects were present in the sample's photoluminescence spectrum.

These observations indicated that this synthetic diamond likely was grown at a relatively high temperature, $\sim 1700^{\circ}$ C, compared to the 1400–1500°C for typical synthetic diamonds (see, e.g., J. E. Shigley et al., "The gemological properties of Russian gem-quality synthetic yellow diamonds,"

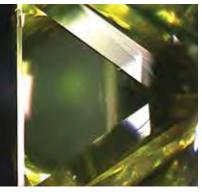


Figure 14. When the synthetic diamond in figure 13 was illuminated using a fiber-optic light, unevenly distributed moderate green luminescence was seen to correspond to the patchy color zoning.

Winter 1993 Gems & Gemology, pp. 228–248). At very high temperatures, octahedral growth sectors are selectively developed and cubic growth sectors are minimized (H. Sumiya et al., "Growth rate of high-quality large diamond crystals," Diamond and Related Materials, Vol. 237/239, 2002, pp. 1281–1285). Most nitrogen in synthetic diamonds grown at such a high temperature is aggregated.

Another possibility is prolonged

Figure 15. In the DiamondView instrument, the synthetic diamond showed green fluorescence with a cross-shaped pattern.



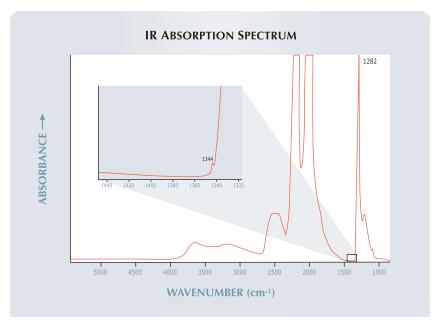


Figure 16. While many HPHT-grown synthetic yellow diamonds are predominantly type Ib due to isolated nitrogen, this sample proved to be type IaA with a trace of type Ib (the small peak at 1344 cm⁻¹); the peak intensity at 1282 cm⁻¹ indicated a nitrogen concentration of >200 ppm in the A centers.

annealing at high temperatures after initial growth, but the very limited development of cubic growth in this synthetic diamond indicated that it was more likely to have crystallized at higher temperatures directly. While HPHT-grown synthetic yellow diamond is becoming more common in the jewelry market, samples such as this one are encountered only rarely.

QianWen (Mandy) Liu

Heat-Treated Blue SAPPHIRE with Unusual Dendrites

The laboratory routinely receives large numbers of blue sapphires for examination. Many of these stones show relatively typical signs of heat treatment in the form of melted and resolidified mineral inclusions, heat-decrepitated primary and secondary fluid inclusions, and color zoning attributed to internal diffusion.

It has also been demonstrated through experimentation with synthetic corundum that the precipitation of inclusions formed by exsolution, such as fine needles of silk-like rutile, can be controlled during the growth process. As would be expected, this is true of natural corundum as well.

During the heat treatment of nat-

ural corundum, preexisting exsolved rutile can be dissolved back into the corundum lattice structure to clarify a rutile-clouded stone. This "breakdown" begins to occur at approximately 1250°C and progresses more rapidly as the temperature and time-at-temperature both increase.

In some instances, however, the rutile is not completely reabsorbed, leaving behind tiny crystallite remnants aligned in typical corundum exsolution patterns; these are sometimes accompanied by clouds of color resulting from internal diffusion. The rutile may also partially recrystallize back into the corundum host. When this occurs, the rutile usually reappears as very fine, short needles or extremely small dust-like, light-scattering particles. Good examples of this type of controlled exsolution can be found in both flame-fusion (Verneuil) grown star sapphires and rubies and cabochoncut natural stones that have asterism induced by lattice diffusion.

Recently the laboratory received a relatively large (20+ ct), faceted, transparent blue sapphire for identification. The many inclusions, all of which showed evidence of heat treatment, made the identification as a natural but heat-treated stone relatively easy.

Figure 17. The rows of unusual tree-like inclusions in this sapphire probably formed through exsolution as a result of the heat-treatment process. Their composition is unknown. Magnified $10\times$.



54 LAB NOTES GEMS & GEMOLOGY SPRING 2007

However, the examination also revealed an inclusion with a dendritic pattern that was unique in our experience. This feature was very elusive in that it was only visible when a fiberoptic light was directed at a specific angle oblique to the plane of the table facet. These dendrites were arranged in consecutive rows and displayed an obvious arborescent habit, as illustrated in figure 17. They also appeared to occupy the same plane in their host. This, combined with their reflective thin-film behavior and crucial directional visibility, led to the conclusion that they were the result of post-treatment exsolution. The fact that they were composed of numerous tiny individual disk-like crystallites also suggested that they originally formed as continuous branching dendrites that then contracted down into individual droplets as the corundum cooled before they finally solidified.

This is only one theory, of course. Readers are invited to convey their own thoughts as to the origin of these unusual inclusions.

John I. Koivula



Figure 18. This unusual elongated dislocation spiral was discovered in a faceted green YAG. It appears to consist of numerous minute particles of growth residue, as well as gas bubbles that were captured as the spiral developed. Magnified 10×.

YTTRIUM ALUMINUM GARNET (YAG) with a Dislocation Spiral

Crystalline compounds, whether natural or synthetic, can show two main types of structural dislocations. The first, *edge dislocations*, are offsets within atomic planes; these can be thought of as an extra half-plane of atoms inserted between two parallel planes in an otherwise "perfect" crystal structure. The other type, *screw dislocations*, are helical in nature; these can be compared to a spiral

staircase or the auger-like pattern on a screw, hence the name. As far as gemstones are concerned, the screw dislocation is visually the most interesting of the two types.

A screw dislocation forms in a gem crystal when a helical pattern develops around a linear defect that starts at a point source, such as an inclusion or a strain knot. The spiral form becomes visible when there is sufficient strain to cause small fractures to develop along the central dislocation line

and/or when fluid inclusions are captured along it as the crystal grows.

While we occasionally encounter beautifully formed dislocation spirals decorated by fluid inclusions and minute fracture patterns in gems such as topaz (orthorhombic) and beryl (hexagonal), these are somewhat expected, since both minerals have central axes of symmetry around which a spiral structure might form. However, using a gemological microscope, we had never seen an inclusion spiral in a material formed in the isometric (cubic) crystal system. It was, therefore, somewhat surprising when just such a spiral-shaped inclusion formation (figure 18) was observed during the otherwise routine identification of a faceted transparent green yttrium aluminum garnet (YAG).

Whether it is a doubly refractive synthetic such as corundum or chrysoberyl, or a singly refractive material such as spinel or YAG, if a melt-grown crystalline rod is sawn in two lengthwise and both halves are polished, the sawn sections will sometimes display centrally located strain oriented parallel to their length. During growth, this central strain zone could develop as a visible spiral in the host, which would explain this unusual inclusion in a YAG. From that, we surmised the green YAG was probably grown by a melt technique rather than from a flux.

John I. Koivula

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