Gem Trade **LAB NOTES**

BROMELLITE

Occasionally we are fortunate to receive for identification faceted examples of minerals that are seldom if ever seen fashioned as gemstones. The West Coast laboratory had such an opportunity when three transparent colorless stones weighing 0.68, 1.92, and 2.80 ct (figure 1) were submitted by Dunil Palitha Gunasekera of Ratnapura, Sri Lanka. He suspected these stones to be johachidolite, a rare borate mineral first described as a cut gem by R. Harding et al. in 1999 (*Journal of Gemmology*, Vol. 26, No. 5, pp. 324–329).

However, the results of gemological testing did not match the properties given for the johachidolite described in the above article (R.I.= 1.717–1.724, S.G.=3.45). Instead, testing showed R.I.'s of 1.718–1.732 and an S.G. of 3.01–3.03. The stones were inert to long-wave ultraviolet (UV) radiation, showed only a very weak yellow reaction to short-wave UV, and no absorption bands were visible in the desk-model spectroscope. These properties did not match any gem material with which we were familiar.

The mystery deepened when energy-dispersive X-ray fluorescence

Figure 2. This 5.84 ct synthetic BeO crystal was grown experimentally in Russia.

Figure 1. These three transparent colorless stones (0.68, 1.92, and 2.80 ct) are believed to be the first reported faceted specimens of the mineral bromellite.

(EDXRF) analysis of the 2.80 ct sample failed to detect any elements at all. While our EDXRF spectrometers are sensitive only to the elements sodium and higher on the periodic table, this was the first time we could recall that the instrument did not detect *any* elements within a crystalline sample. GIA research gemologist Shane Elen, on hearing of these results and having just read the

Figure 3. Large, white, irregular flux inclusions were present in both samples of synthetic bromellite examined for this study. Magnified 40×.

Figure 4. All of the faceted bromellites revealed groups of transparent colorless irregular crystals similar to that shown here in the 0.68 ct sample. Magnified 40×.
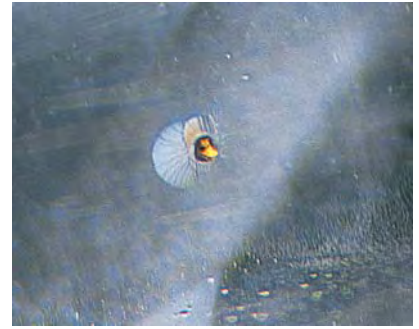

Figure 5. Also present in all of the faceted bromellites were several transparent rounded yellow crystals surrounded by haloes. Magnified 40×.

chapter on synthetic gemstones in J. E. Arem's *Color Encyclopedia of Gemstones* (2nd ed., 1987, Van Nostrand Reinhold, New York), suggested BeO as a possible chemical makeup. Arem mentions that colorless BeO crystals, with the mineral name bromellite, have been grown synthetically.

We then turned to an X-ray powder diffraction database, limiting our search to inorganic minerals with an S.G. of 2.92–3.12 and eliminating many of the elements detectable by EDXRF. The search provided a list of three possibilities (with formulas as indicated in the database): sidwillite [$MoO_3 \cdot 2H_2O$], hydroxylbastnäsite [$Nd(CO_3)(OH)$], and bromellite [BeO]. We eliminated the first two minerals because neither Mo nor Nd was detected in the chemical analysis, and the R.I.'s of both are distinctly different from those we had recorded. Published values for bromellite, however, almost exactly fit the R.I. and S.G. of the samples in question. The identification was confirmed when Raman spectra of the samples yielded a good match for bromellite from the Renishaw Raman database.

Since none of us had ever heard of bromellite being fashioned as a gemstone, and a search of the gemological literature did not reveal any mention of this mineral being cut

and polished, we next addressed the question of the natural or synthetic origin of these three samples. Synthetic bromellite has been grown for nearly four decades (see, e.g., H. W. Newkirk and D. K. Smith, "Crystal growth, morphology and twinning of synthetic macrocrystals and microcrystals of bromellite," *Acta Crystallographica*, Vol. 16, Pt. 13, 1963, p. 142). One of the authors (SFM) located two crystals of BeO that were represented as having been grown experimentally in Russia. The Raman spectra obtained on one of the crystals (figure 2) matched the spectra of the faceted stones. However, these crystals still had remnants of the wires from which they were suspended in their growth environment, as well as large, white, irregular inclusions that were probably flux trapped during growth (figure 3). These inclusions should be easily recognizable as proof of synthetic origin in a faceted stone.

In contrast, the internal features of the three stones submitted to the lab included planes of liquid inclusions, transparent colorless irregular crystals (figure 4), transparent yellow rounded crystals (figure 5), and numerous thin needles (figure 6). The nature of these inclusions, plus the obvious difference from those seen in the known synthetics, was sufficient to prove the natural origin

of these bromellites. According to R. V. Gaines et al. (*Dana's New Mineralogy*, 8th ed., John Wiley & Sons, New York, 1997, p. 211), bromellite has been recovered from skarns in Sweden and Texas, from the emerald mines in the Ural Mountains of Russia, and from unspecified rocks in Norway.

We believe these three stones are the first reported examples of faceted gem-quality bromellite. For future reference, table 1 lists the properties of this new gem material.

SFM and Sam Muhlmeister

Figure 6. Numerous thin needles, which did not display any particular orientation within the stones, were seen in all the faceted bromellites and helped confirm their origin as natural. Magnified 29×.

TABLE 1. Properties of bromellite.

Property	Characteristic
Color	White[a] or colorless
Luster	Vitreous[a]
Hardness	9[a]
Cleavage	{10$\bar{1}$0} perfect; {0001} good[a]
Refractive indices	n_ω=1.718, n_ε=1.732
Birefringence	0.014
Crystal system	Hexagonal[a]
Optic character	Uniaxial positive
UV fluorescence	
Long-wave	Inert[b]
Short-wave	Inert to weak yellow[b]
Absorption spectrum	No features seen
Specific gravity	3.01–3.03
Inclusions	Planes of liquid inclusions, transparent colorless irregular crystals, transparent yellow rounded crystals, numerous thin needles

[a]From Gaines et al. (1997).
[b]For bromellite from Norway, Gaines et al. (1997) reported white fluorescence to short-wave UV, whereas yellowish white fluorescence to both short- and long-wave UV was reported by A. O. Larsen et al. ("Bromellite from syenite pegmatite, southern Oslo region, Norway," Canadian Mineralogist, Vol. 25, No. 3, 1987, pp. 425–428).

CHAROITE Inclusions In a Quartz-Feldspar Rock

In the Winter 2001 Lab Notes section (p. 318), we reported on the examination of an interesting 292.14 ct cabochon of charoite with tinaksite that was submitted to the West Coast laboratory by gemologist Leon M. Agee of Agee Lapidary in Deer Park, Washington. More recently, Mr. Agee sent another "charoite" cabochon for examination, which turned out to be just as interesting as the previous gem. This new cabochon weighed 73.55 ct and measured 39.12 × 27.56 × 7.20 mm (figure 7).

Although for the most part the cabochon resembled charoite in appearance, the gemological properties were consistent with quartz, not charoite. Examination with magnification and testing with laser Raman microspectrometry revealed that the cabochon consisted primarily of a mixture of transparent to semitransparent near-colorless quartz and, to a much lesser extent, feldspar. Within this host matrix, numerous lavender-colored inclusions were identified as charoite by Raman analysis. These inclusions primarily took the form of dense curved and twisted bundles of fibers and fine sprays of needles, but some individual fibers were also noted. Where the inclusions were particularly dense, the charoite gave the host quartz/feldspar a pleasing lavender color. Where the inclusions were finely disseminated, they made the host slightly translucent but did not add any appreciable color. This is the first time we have encountered charoite as a recognizable inclusion in another gem material.

JIK and Maha Tannous

DIAMOND
Altered vs. Natural Inclusions in Fancy-Color Diamonds

Characterizing gemological features has always been critical in determining the origin of color in a diamond. With the increased use of high pressure/high temperature (HPHT) technology to change the color of diamond, this endeavor has become even more important. The intense heat and pressure generated by this treatment may alter internal characteristics, thus leaving clues for the gemologist. Yet interpretation of these clues can be very challenging, as evidenced by two diamonds recently submitted to the East Coast laboratory for origin-of-color reports.

The 3.38 ct Fancy Intense blue marquise and 1.20 ct Fancy Vivid orange-yellow pear shape exhibited very similar inclusions that looked suspiciously like those often found in HPHT-annealed stones (see, e.g., T. M. Moses et al., "Observations on GE-processed diamonds: A photographic record," Fall 1999 *Gems & Gemology*, pp. 14–22; and I. M. Reinitz et al., "Identification of HPHT-treated yellow to green diamonds," Summer 2000 *Gems & Gemology*, pp. 128–137). In figure 8, the blue diamond displays a large, flat, apparently graphitized inclusion surrounded by a colorless halo. Similarly, in figure 9, the yellow diamond also has a large, flat inclusion

Figure 7. Consisting primarily of quartz and, to a much lesser extent, feldspar, this 73.55 ct cabochon was decorated internally with fibrous bundles and fine sprays of charoite.

Figure 8. The tension halo in this 3.38 ct blue diamond appears similar to inclusions seen in HPHT-annealed diamonds. However, the smooth, shiny appearance of the halo suggests that the color in this diamond is natural, as was confirmed by spectroscopy. Magnified 45×.

Figure 9. The tension halo in this 1.20 ct orange-yellow diamond appears similar to the one in figure 8. In this case, however, the sugary texture and undulating nature of the halo suggests that this diamond has been exposed to an HPHT process, which was confirmed by spectroscopic analysis. Magnified 45×.

that appears to be graphitized, with a colorless halo surrounding it.

After further examination, we noted subtle but significant differences between these two inclusions and their associated tension halos. The halo in the blue diamond was quite flat with little or no relief. The halo in the yellow diamond appeared to undulate as it went around the dark center. Also, the surface of the halo in the blue diamond was very shiny in reflected light, whereas the surface of its counterpart in the yellow diamond revealed a less reflective, sugary appearance.

Black inclusions are relatively common in natural-color type IIb blue diamonds (see J. M. King et al., "Characterizing natural-color type IIb blue diamonds," Winter 1998 *Gems & Gemology*, pp. 246–268). They are less common in natural-color yellow diamonds, but they are not unknown. Tension haloes can form naturally around inclusions in any diamond.

The conditions of HPHT annealing also can create tension haloes around existing inclusions while stimulating graphite formation along the crystal faces of those inclusions. The result may be a large, flat inclusion with a dark center, which is surrounded by a transparent/translucent halo.

However, there are subtle differences in tension haloes produced naturally and those resulting from HPHT processing. This led us to suspect that the orange-yellow diamond had in fact been color enhanced by HPHT annealing, while the blue one had not. Because the HPHT process subjects a diamond to intense heat and pressure for a relatively short period of time, the damage it often causes to existing features may be severe. From our experience, the sugary texture and undulating nature of the tension halo in the orange-yellow pear shape suggested that this diamond was subjected to a rapid change in heat and pressure, whereas the smooth, shiny appearance of the halo in the blue diamond indicated that it was caused by a slower, natural process. Spectroscopic analysis confirmed the observations we had made with magnification: The blue diamond had not been HPHT annealed, and the orange-yellow diamond had. These examples further validate the need for both careful gemological observations and advanced analytical testing to arrive at a correct origin-of-color determination.

Thomas Gelb and Matthew Hall

**With "Compact Disc" Inclusion**

During the process of clarity grading a diamond, graders will occasionally come across inclusions that are reminiscent of some familiar object that adds interest to the diamond even if it also lowers the clarity grade of the host. Such was the case when a near-colorless 0.97 ct round brilliant was submitted to the West Coast laboratory for a diamond grading report.

The dark portion of the inclusion observed in this diamond was too deep to be identified by Raman analysis. Through the microscope, however, and in darkfield illumination, the inclusion appeared to be composed of an opaque dark gray to black graphite and/or sulfide center that was surrounded by an almost perfectly circular cleavage disk.

When the inclusion was examined at a slight angle to its plane with a fiber-optic illuminator, the cleavage disk took on a reflective, silvery appearance, with what looked like numerous fine lines arranged in a relatively uniform pattern around the dark center. These grooves appeared to be minute steps in the primary cleavage plane. They were probably extensions of the other three directions of octahedral cleavage that is typical of diamond.

When the fiber-optic illuminator was placed in one specific position, as shown in figure 10, the light scattered

Figure 10. Extending from a dark inclusion in a near-colorless diamond, this cleavage disk takes on the appearance of a compact disc when it is illuminated with a fiber-optic light. Magnified 20×.
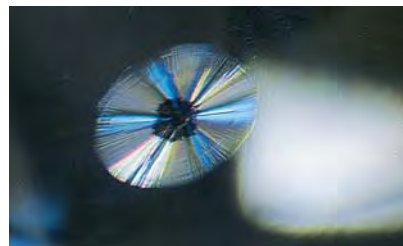
and reflected by the grooves on the cleavage disk formed a distinct radial pattern, with the "flares" varying from silvery white to a slightly grayish blue. To those who viewed the unusual feature, this created the illusion of light reflecting from a compact disc.

JIK and Maha Tannous

HEMIMORPHITE,
A Rarely Encountered Gem Material

At first look, the medium-tone, intensely colored greenish blue oval cabochon shown in figure 11 reminded us of high-quality turquoise or possibly chalcedony. The principal refractive indices, measured on the base of the 9.80 ct cabochon, were approximately 1.613–1.635. These quickly ruled out chalcedony, but not turquoise. However, the specific gravity (determined hydrostatically) was approximately 3.45, significantly higher than turquoise.

The cabochon was inert to both long- and short-wave UV fluorescence, and no absorption lines were visible with a desk-model spectroscope. The combination of properties indicated that the cabochon might be hemimorphite [$Zn_4Si_2O_7(OH)_2 \cdot H_2O$]. To confirm this identification, we turned to the advanced techniques of

Figure 11. This 9.80 ct oval cabochon (12.90 × 11.00 × 7.50 mm) was identified as hemimorphite, which is seen only rarely as a fashioned gem.

laser Raman microspectrometry and qualitative chemical analysis using EDXRF spectrometry. With Raman analysis, we observed a strong peak at 932 cm⁻¹, a moderate peak at 682 cm⁻¹, and relatively weak peaks at 333, 404, and 455 cm⁻¹; this spectrum matched well that of our reference sample of hemimorphite. As a further confirmation, EDXRF analysis showed silicon and zinc in high concentrations (along with lower amounts of copper, sodium, and lead). Hemimorphite is a common mineral in the oxidized zone of zinc-bearing mineral deposits. However, we have encountered gems fashioned from hemimorphite only rarely in the laboratory.

For the most part, these observations are consistent with those reported for hemimorphite in the Spring 1998 Lab Notes section (pp. 44–45). Unlike the hemimorphite cabochon reported by R. T. Liddicoat in the Winter 1971–72 Lab section (pp. 383–384), the current cabochon did not show distinctive agate-like banding and it had a more fibrous structure, similar to the cabochon and rough sample reported in 1998.

TM and Wuyi Wang

SAPPHIRE
Bulk or Lattice Diffusion Treated

Over the last several months, there has been extensive discussion and research related to the color-enhanced yellow and orange-to-orangy pink sapphires that have been treated mainly in Thailand. The cause of color in these stones has been a major focus of the investigation.

A few key characteristics have been observed in many of the samples examined to date, both by GIA and by other laboratories throughout the world. The first is that the yellow-to-orange color produced by this treatment generally does not penetrate completely through the stone, but is confined to varying depths relative to the outline of the original facets. The second, observed in a smaller number

Figure 12. When immersed in methylene iodide, this nearly 20 ct sapphire shows an orangy yellow layer of color following the outline of the facets, which indicates that it was subjected to a bulk-diffusion process.

of cases, is that there has been recrystallization or new crystal growth on the surface or just below the surface. The third characteristic, observed in samples that have been sectioned and analyzed by sophisticated techniques, is the presence of beryllium in higher concentrations near the surface, or corresponding to the depth of the treated color layer.

A large stone submitted for an identification report in the East Coast laboratory demonstrated how these characteristics could be used to identify this new treatment, which scientifically is referred to as bulk or lattice diffusion. The orangy yellow cut-cornered rectangular mixed cut measured 14.85 × 12.61 × 10.71 mm and weighed almost 20 ct. The gemological properties recorded were consistent with sapphire. Examination with a desk-model spectroscope did not reveal any sharp absorption features. However, the stone fluoresced weak orange to long-wave UV radiation, and a moderately chalky but very weak patchy yellow to short-wave UV, which is consistent with heat treatment. Preliminary inspection of this exceptionally clean stone with magnification revealed only a few small chips around the girdle and no diagnostic inclusions to indicate if

the stone was natural or synthetic. However, when the stone was examined while immersed in methylene iodide, a clear demarcation could be seen between a near-colorless core and an orangy yellow zone that followed the outline of the faceted sapphire (figure 12). This was the feature that indicated the sapphire had been subjected to a bulk-diffusion process.

From dozens of past (destructive) chemical analyses, we have confirmed the presence of beryllium in the yellow-to-orange color layer of the bulk diffusion-treated sapphires. Previously, beryllium alone was not known to act as a chromophore; it was believed to be interacting with elements/defects intrinsic to the sapphire to produce the yellow-to-orange color. However, Dr. John Emmett of Crystal Chemistry in Brush Prairie, Washington, reports that recent experiments have demonstrated that beryllium, diffused into the stone in an oxygen atmosphere, can in fact cause color on its own (see http://www.agta.org/consumer/gtclab/treatedsapps04.htm).

Although we found compelling evidence that this sapphire had been subjected to a bulk-diffusion process, we still had to prove its origin—natural or synthetic. The absence of Plato lines suggested it was not a melt-process synthetic. Observation of the stone with magnification in diffused illumination revealed a weak straight, parallel growth structure under the table, which is consistent with natural sapphire. Chemical analysis using EDXRF showed, as expected, large amounts of Al with lesser amounts of Fe and Ti and a significant quantity of Ga. The relatively high amounts of gallium and titanium are also indicative of natural origin.   *TM*

Bulk Diffusion–Treated Sapphire with Synthetic Overgrowth

Not all of the discussion concerning bulk diffusion of yellow coloration into sapphire has revolved around the source of color. At the extreme tem-
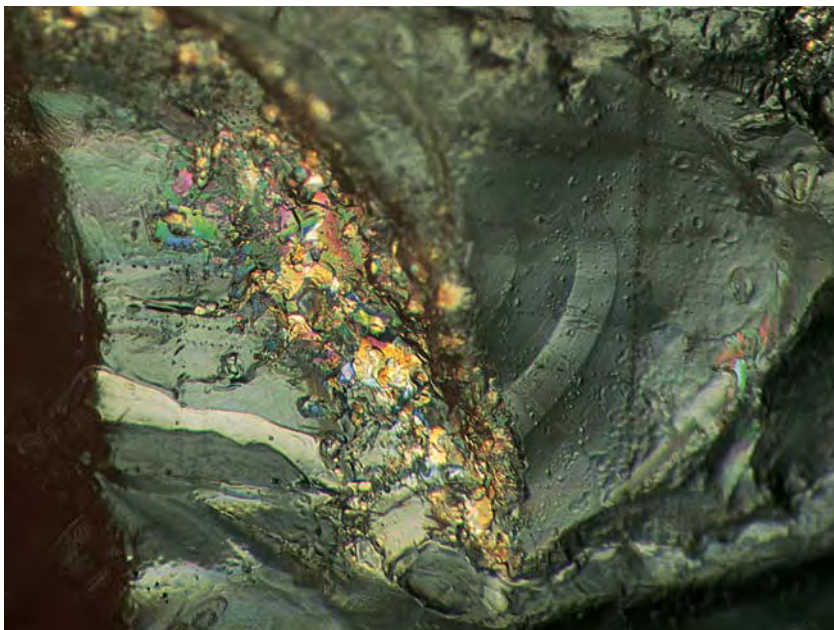

Figure 13. Much of the synthetic corundum growth on the surface of bulk-diffused sapphires is in the form of randomly oriented clusters of crystals, here seen with polarized light. Magnified 20×.

peratures required for this treatment, many other changes can take place. One of these is the partial dissolution of the corundum in the crucible (and potentially of the walls of the alumina crucible itself) through contact with fluxes, which can create an environment where synthetic corundum grows on the surface of stones on cooling. During our research on this new treatment, we have seen many examples of corundum "redeposition" on treated rough and preformed samples, often as groups of minute flat hexagonal platelets. Most of this synthetic corundum is removed when the stones are finished, but we have seen a number of examples—most commonly the treated yellow sapphires—where some of this material was left on the polished stone. Ken Scarratt, of the AGTA Gemological Testing Center, first published his observations on this phenomenon April 19, 2002, on the AGTA Web site (http://www.agta.org).

The examples we have seen of synthetic growth on bulk-diffused sapphires have been quite different from

past cases where the synthetic overgrowth has been intentional, such as with Lechleitner synthetic overgrowth on beryl or corundum. In those gem materials, the crystallographic orientation of the overgrowth has been consistent with the orientation of the base material. Synthetic corundum growth on the bulk-diffused sapphires is entirely random in orientation and tends to form clusters of tiny crystals (figure 13). After the stones are polished, this random orientation manifests itself in two ways that make identification of the presence of synthetic material relatively easy.

The first of these is the appearance of the overgrowth in a microscope. With darkfield illumination, these areas look roiled, somewhat like areas of graining, although they are much more isolated than one would expect for natural graining. With transmitted light, however, it is possible to see the edges of many of the individual crystals within this roiled zone (figure 14, left).

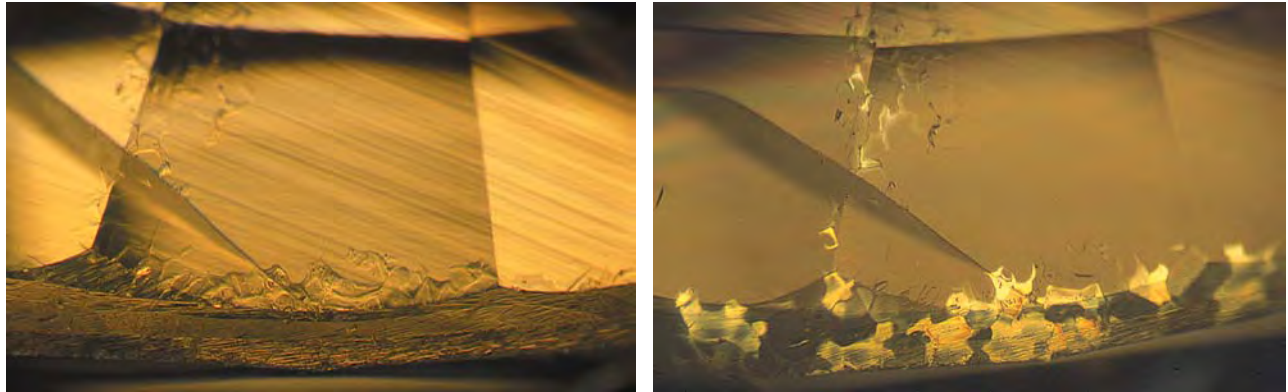The second manifestation is the reaction of the areas of overgrowth to

Figure 14. On finished stones, the synthetic corundum growth may show up as roiled areas, with some crystal faces becoming visible in transmitted light (left). When viewed in cross-polarized light (right), the individual crystals of synthetic corundum may become visible as bright areas against a dark background due to differences in their orientation. Both images magnified 40×.

cross-polarized light. Because the synthetic crystals are randomly oriented, their directions of extinction should be different from one another, as well as from the host material. With proper orientation (which may require changing the position of the Polaroid plates or the stone), and with the host stone in the dark position, many of the synthetic crystals will be in their light position, which can be seen easily in the microscope (figure 14, right).

To help meet this identification challenge, gemologists must be aware of the possible presence of a synthetic overgrowth and know what it looks like. However, with increasing trade awareness of the overgrowth, the cutters of these stones will probably make every effort to remove all the synthetic material after treatment.

SFM

Synthetic Sapphire Treated by "Traditional" Bulk Diffusion with Transition Elements

Coincidentally, only a few days after the West Coast lab received the orangy yellow bulk-diffused sapphire described above, a client submitted the earring shown in figure 15 for an identification report. Routine gemological testing indicated that the center stone was a sapphire. Like the

orangy yellow sapphire, however, this one was free of any readily apparent inclusions—even with high magnification. It revealed only "wear" marks—small chips and abrasions. It did not show any absorption features with a desk-model spectroscope. There was no visible

Figure 15. The 8.00 × 6.20 × 3.80 mm blue stone in the center of this yellow-metal cluster earring set with "old cut" diamonds proved to be a bulk-diffused synthetic sapphire.

reaction to long-wave UV radiation, and the reaction to short-wave UV was a moderately strong chalky yellow or white. This type of short-wave reaction in a sapphire suggests that it has been heat treated or, in the case of a blue sapphire, that it may be synthetic.

Figure 16. As seen with immersion and the earring viewed from the back, the shallow and patchy blue coloration, plus the "outlining" of the facet junctions, serves as proof that the blue color in the synthetic sapphire was produced by bulk diffusion.

When the earring was immersed in methylene iodide, the true identity of the blue stone became clear. The patchy, shallow blue coloration and the concentration of color along facet junctions (or "outlining"; see figure 16) indicated that this stone owed its blue color to a diffusion (now referred to as "bulk diffusion") process. (For a review of these characteristics, see R. E. Kane et al., "The identification of blue diffusion-treated sapphires," Summer 1990 *Gems & Gemology*, pp. 115–133.) As the stone was moved and inspected in several positions, we observed an even more interesting feature: Subtle curved blue color banding extended across its width. This confirmed that it was a bulk-diffused synthetic sapphire. The pale blue of the curved banding suggests that this was the color of the starting material.

The process of bulk diffusion is not new; in fact, patents for this procedure (held by Union Carbide) date back to the mid-1970s. Typically, the blue color in such treated sapphires is created by the diffusion of titanium. As noted in the earlier entries, however, we currently are seeing the diffusion of new, light elements into the crystal lattice of corundum. Depending on the elements diffused, the chemistry of the host, and several other variables, the resulting color and properties of the treated corundum may vary widely. This is creating a significant challenge for the corundum market.

Given the identification of the center stone, we were surprised to see that the surrounding diamonds were fashioned in Old European, older Swiss, and single cuts, which are indicative of a period jewelry piece. Although we can never be certain that such a piece of jewelry is an original period piece or a modern reproduction, it added to the intrigue of the identification. If it was an original period piece, the setting obviously would predate the availability of bulk-diffused corundum. This makes one wonder what center stone may have been set in the earring originally or even what kind of stone is in the
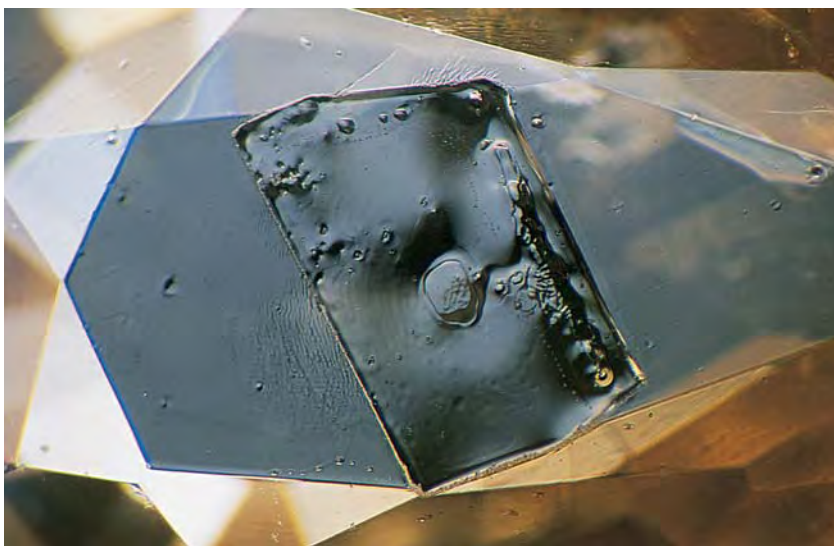

Figure 17. It is readily apparent that no effort was made to remove the surface evidence of high-temperature heat treatment from this 0.95 ct yellow sapphire. Shadowed fiber-optic illumination brings out even small details on the badly damaged table facet. Magnified 10×.

other earring (which was not submitted for identification).   *TM*

Sapphire with Surface Evidence of Heat Treatment

The West Coast laboratory recently had the opportunity to study an interesting heat-treated stone that was purchased in Bangkok from a parcel of commercial sapphires by Lab Notes co-editor Tom Moses. As determined by standard gem identification techniques, the 0.95 ct light yellow pear-shaped mixed cut (8.34 × 5.29 × 2.77 mm) was a natural sapphire.

Examination with a gemological microscope and a fiber-optic illuminator revealed small, light-scattering particles in distinct angular formation within the stone. The facets were covered with numerous solidified droplets of a transparent material that had a Mohs hardness of approximately 6 and appeared to be amorphous. The table facet in particular revealed obvious surface evidence of high-temperature treatment. As shown in figure 17, flow lines and ridges in the surface layer on the

table were clearly visible with shadowed fiber-optic illumination. Laser Raman microspectrometry confirmed that the host was a sapphire and the thick, amorphous-looking patches and droplets of residue were a glass. The pattern formed by the glass residue suggests that the table of this sapphire was in contact with another surface during treatment.

Identification of heat treatment in this instance was made relatively easy by the complete lack of re-polishing of the sapphire. Although such obvious evidence is rarely encountered, since most heat-treated sapphires are polished after treatment, occasionally remnants of the surface layer will remain on unpolished areas of the stone and aid in the identification.

JIK and Maha Tannous