

EDITORS

Thomas M. Moses, Ilene Reinitz,
Shane F. McClure, and Mary L. Johnson
GIA Gem Laboratory

CONTRIBUTING EDITORS

G. Robert Crowningshield
GIA Gem Laboratory, East Coast

Karin N. Hurwit, John I. Koivula, and
Cheryl Y. Wentzell
GIA Gem Laboratory, West Coast

Chrysocolla "Owl" AGATE

Agates and other varieties of chalcedony are fascinating because their patterns vary so widely. While transparent single-crystal gems are much more uniform, chalcedony is one of nature's less-structured "painter's palettes," from which some quite imaginative jewelry and other decorative objects can be created. Among agates, some of the more interesting are the so-called eye agates, which develop as circular-to-semicircular

concentrically layered agate through the cyclic deposition of chalcedony around a central acicular inclusion composed of some foreign substance, usually a mineral.

Recently, the West Coast laboratory had the opportunity to examine a well-polished bevel-edged oval plate of attractively patterned eye agate, which measured approximately 21.3 × 14.8 × 4.3 mm and weighed 12.71 ct. As shown in figure 1, the gem displayed a double "eye" structure of

blue chrysocolla (identified by Raman analysis), with each eye individually surrounded by concentric bands of clear, near-colorless, and white agate. These in turn were surrounded by densely packed and interlocked macroscopic white-to-colorless quartz crystals that somewhat resembled white feathers.

This distinctive piece was cut by Leon M. Agee of Agee Lapidary, Deer Park, Washington, from an inexpensive scrap of material that reportedly was from the Ray copper mine in Pinal County, Arizona. This locality is well-known among gem and mineral collectors for its magnificent specimens of chrysocolla in chalcedony.

In the past we have encountered and documented other agates showing pleasing and interesting patterns (see, e.g., *Gem News*: Summer 1998, pp. 136–137, and Winter 1999, p. 210; *Lab Notes*, Winter 2002, p. 340). With a little imagination, it is not hard to see how this piece could be featured as the "all knowing" eyes in a one-of-a-kind snowy owl brooch.

JIK and Maha Tannous

Figure 1. This striking 21.3-mm-long Arizona "eye" agate shows an interesting double-eye pattern that could be set in a unique owl-themed brooch. Note the concentric formational banding of the agate around the chrysocolla-centered "eyes." Magnified 5×.



Editor's note: The initials at the end of each item identify the editor(s) or contributing editor(s) who provided that item. Full names are given for other GIA Gem Laboratory contributors.

*Gems & Gemology, Vol. 39, No. 4, pp. 314–321
© 2003 Gemological Institute of America*

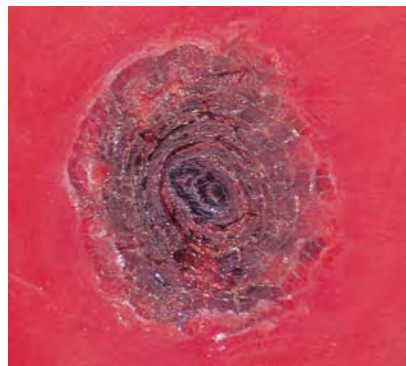


Figure 2. This dark brown area on a polished coral bead appears to be where a form of conchiolin coral was intersected and overgrown by red calcareous coral. Magnified 5x.

Interesting Red CORAL

While gems such as diamonds, rubies, sapphires, and emeralds are frequently submitted for identification, only occasionally do we see specimens fashioned from conchiolin or calcareous coral. Recently, an interesting necklace composed of what were believed to be red calcareous coral beads was submitted to the West Coast laboratory to determine if the beads were natural coral and whether they had been dyed.

Since calcareous coral has a well-known characteristic structure that is visible even with low magnification, it is relatively simple to identify this form of red coral and separate it from its various substitutes, such as dyed massive calcite, Gilson simulated coral, plastic, or glass. As is generally the practice, one bead in the necklace was randomly selected for gemological testing. Although examination with magnification easily proved it to be calcareous coral, there was still the question of whether it had been dyed or was natural color.

To determine if the bead had been dyed, we used the gemological microscope to examine the surface and the drill hole for signs of dye concentration or other color irregularities. During this search, we observed two interesting and unusual structural features on the surface of the pol-



Figure 3. Surrounded by red coral, this white-to-light pink zone marks where a smaller branch of protruding coral was removed from the main mass during fashioning. Magnified 5x.

ished bead. One of these features was a dark brown, off-round area of more or less concentric rings that looked quite literally like a cross section cut through a tree branch (figure 2). Testing with a hot point yielded an odor consistent with that of burning hair or a similar organic. From the shape of the feature and its reaction to heat, we suspect that it resulted when a branch of conchiolin coral was intersected and overgrown by the red calcareous coral.

The other surface characteristic was a web- or flower-shaped white-to-light pink zone (figure 3) that showed where a smaller branch of protruding coral had been polished off the main mass during fashioning of the bead. Since no dye was detected in either of these surface features or anywhere else on the bead, we concluded that the color was natural as well.

JIK and Maha Tannous

Coated DIAMONDS

A number of new diamond treatments have appeared in the last decade. Between fracture filling and high pressure/high temperature (HPHT) treatment, one sometimes forgets about the older, once-popular treatment of diamond coating. This practice was

discussed by early GIA gemologist Eunice Miles in her groundbreaking article, "Diamond-coating techniques and methods of detection," in the Winter 1962–63 issue of *Gems & Gemology* (pp. 355–364).

Diamonds may be coated to deepen their color (in the case of fancy colors) or to diminish the amount of apparent color (in the D–Z range). Since the majority of gem diamonds are type Ia, with varying degrees of yellow due to nitrogen impurities, historically the most common coating technique has been to paint a thin blue film on diamonds of a near-colorless or lower color grade. This type of coating is typically confined to the crown and pavilion facets close to and on the girdle edge, though it can completely cover the diamond's surface. Since blue and yellow are complimentary colors, they appear to cancel each other out to produce a more colorless appearance. (Ever wonder why the flutes in most parcel papers are light blue?) It has been almost 20 years since a Lab Note on a coated diamond was last published (see Summer 1984 Lab Notes, p. 107). While, as then, it is true that only a few coated diamonds are submitted to the lab each year, the practice has not completely disappeared.

Recently, two round brilliant diamonds (1.09 and 3.03 ct) were submitted, by separate clients, to the East Coast laboratory. In the course of grading, the presence of a coating in the form of pale blue spots was observed while the diamonds were being examined with magnification. Figure 4 shows the coating on the 3.03 ct stone, which displays what Eunice Miles reported in 1962 as a faint blue "flux" that is characteristic of this treatment. Although GIA does not grade coated diamonds, for the purposes of this report we determined that the color of the smaller stone was equivalent to the "Q–R" range, while the larger appeared to be "M." In many instances, the lab has observed this treatment with diamonds in the near-colorless range, but over the years staff members have seen it applied to dia-

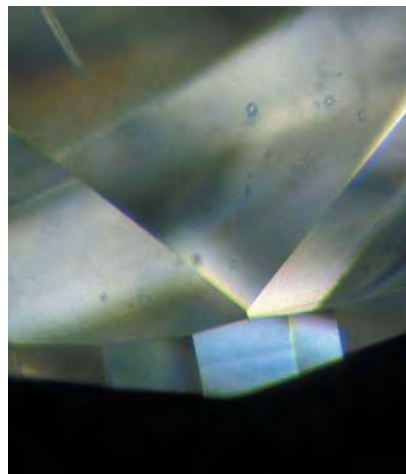


Figure 4. Seen here on the bezel and upper girdle facets of this 3.03 ct round brilliant diamond, multiple blue spots are indicative of a coating. Magnified 33 \times .

monds with color grades through light yellow. Another clue to this type of treatment is often a “grayish” overall body color evident when these stones are observed table-down in a standard color grading environment.

Upon detection of the treatment, these two diamonds were returned to the clients to remove the coating before grading could continue. Since they were not resubmitted as of this writing, we do not know their true colors.

Joshua Sheby

EMERALD

Natural, with Abundant Nail-head Spicules

Separation of natural and synthetic gems frequently requires a variety of techniques, although observation of internal features is often sufficient to make a conclusive identification. For example, curved striae or curved color banding and large gas bubbles quickly reveal flame-fusion synthetic corundum. In some instances, however, synthetics may have inclusions that resemble those found in natural stones.

The West Coast laboratory recently received for identification a 9.69 \times 9.47 \times 6.30 mm green ring-set gem. The refractive index, specific gravity, fluorescence, and spectra matched those of natural or synthetic emerald. When examined with magnification, however, the sample revealed a large number of what appeared to be nail-head spicules in several parallel planes (figure 5). Nail-head spicules are commonly associated with hydrothermally grown synthetic emeralds, but they can also occur in natural stones (see Spring 1992 Lab Notes, pp. 54–55). They are formed when a small piece of material is deposited on the surface of the growing crystal, and a tapered void is created as the crystal continues to grow past the inclusion. In synthetic emeralds, the solid material is usually phenakite, beryl, chrysoberyl, or gold (the last is derived from the vessel in which the emerald is grown). In this emerald, the crystals at the base of the tapered void were flat platelets that were not easily resolved, and the voids were liquid two-phase inclusions with a gas bubble.

Because the appearance of the nail-head spicules in this particular specimen was inconclusive, we asked senior research associate Sam Muhlmeister to perform energy-dispersive X-ray fluorescence (EDXRF) analysis to test for the presence of

nickel. In Russian hydrothermally grown synthetic emeralds, NiO is found in greater concentrations than is known in natural emeralds. Our test results showed the presence of a trace amount of nickel that was more consistent with that found in natural emeralds (H. W. Schrader, “Contributions to the study of the distinction of natural and synthetic emeralds,” *Journal of Gemmology*, Vol. 18, pp. 530–543). Infrared spectroscopy also did not show any of the peaks that are considered diagnostic of synthetic emerald. Nevertheless, these tests, too, were inconclusive. Ultimately, the determination of natural vs. synthetic was made based on the refractive indices and the presence of skeletal platelets of ilmenite (figure 6).

Hydrothermally grown synthetic emeralds usually have R.I. values very similar to those of natural emeralds, particularly those from Colombia, although they generally do not exceed an upper limit of 1.585. The R.I.’s of this stone were 1.584–1.590, which is consistent with an emerald of natural origin.

Ilmenite is found more commonly in aquamarine as opaque black to dark brown dendritic or skeletal platelets, but it has not been observed in synthetic emeralds. The ilmenite in this emerald was visually identi-

Figure 5. Nail-head spicules, a common feature in hydrothermal synthetic emeralds, are also seen in natural emeralds, as shown here. Magnified 22 \times .

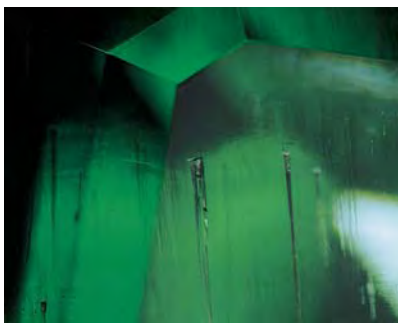
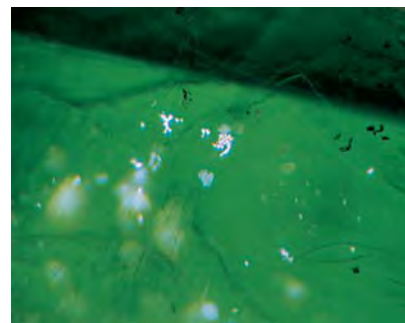


Figure 6. The presence of these skeletal platelets of ilmenite helped prove the emerald’s natural origin. The white appearance of some of the inclusions is due to light reflecting off them. Magnified 40 \times .



fied by chief research gemologist John Koivula through its distinctive skeletal or snowflake-like structure. These two pieces of evidence were enough to conclude that this emerald was indeed a natural stone.

Kimberly Rockwell

With Strong Dichroism

An unusually colored transparent bluish green pear-shaped gem (figure 7) recently arrived at the West Coast laboratory for identification from the private collection of Dale Dubin. The stone was reportedly a beryl that had been mined from Paraíso do Norte, Goiás State, Brazil. The client inquired as to whether the stone was indeed a beryl and, if so, whether the strong blue color component would prevent it from being classified as an emerald.

The 1.12 ct stone (8.52 × 6.44 × 4.04 mm) was moderately to heavily included, with numerous internal characteristics typical of emerald. Fourier-transform infrared (FTIR) and Raman spectroscopy both revealed spectra that were consistent with those of emeralds we have on file. There was no luminescence to long- or short-wave UV radiation, and the stone displayed a uniaxial optic figure in the polariscope.

Additional examination and testing revealed several unusual characteristics. The R.I.'s (~ 1.595–1.601) and S.G. (~ 2.76, by the hydrostatic

Figure 7. This 1.12 ct bluish green emerald, reportedly from Brazil, displayed several unusual gem properties.



Figure 8. The dichroism of the emerald in figure 7 was unusually strong and distinct, with a very saturated blue component.



method) were both high compared to other Brazilian emeralds we have seen. These high properties are more typical of emeralds from African countries such as Zambia (R. Webster, *Gems*, 5th ed., Butterworth-Heinemann, London, 1994, pp. 117–118).

However, the most unusual quality of this stone was its strong and distinct dichroism, of medium yellowish green and vivid blue (figure 8). The absorption spectrum as seen with a desk-model spectroscope revealed chrome lines typical of emerald and a 427 nm line typical of aquamarine, indicating the presence of iron as a coloring agent in addition to chromium. This seemed to explain the presence of both the green and blue components of the color.

In an attempt to confirm the elemental coloring agents, Sam Muhlmeister performed EDXRF analysis as well as UV-Vis spectroscopy. Trace elements revealed by EDXRF were V, Cr, Fe, and Rb. UV-Vis analysis corroborated the presence of Cr and significant amounts of Fe. This, along with the 427 nm line visible in the desk-model spectroscope, led us to conclude that iron was responsible, at least in part, for the strong blue component of the color.

Several properties confirmed that the material was indeed beryl, and the predominantly green color of the stone was sufficient to classify it as an emerald. The owner stated that he had seen a few other similar stones from his supplier, but none as blue as

this one. Indeed, this emerald displayed perhaps the most intensely saturated blue dichroic color component we at the laboratory had ever seen in a Brazilian emerald.

CYW

High-R.I. GLASS Imitation of Tanzanite

Glass has long been used to imitate a wide variety of gem materials. In recent years, we have seen an increased amount of blue to violet glass being used to simulate the very popular gem tanzanite (see, e.g., L. Kiefert and S. T. Schmidt, "Some tanzanite imitations," Winter 1996 *Gems & Gemology*, pp. 270–276). As shown in figure 9, the West Coast

Figure 9. The high R.I. of this 5.42 ct violetish blue specimen of manufactured glass, very close to that of tanzanite, could lead to a misidentification.



laboratory recently received a 5.42 ct transparent violetish blue oval modified brilliant for identification. The specimen, submitted to us by Martinek's Jewelers of Traverse City, Michigan, was brought to them by one of their customers, who had purchased it in Mexico as tanzanite. Upon recording the gemological properties, we could see how a quick examination could lead to a misidentification.

The most interesting characteristic of this material was its high refractive index, which was very close to that of tanzanite. We recorded a single R.I. of 1.700, which is the high value for tanzanite (1.690–1.700). The glass imitations of tanzanite we have examined in the past have seldom had refractive index readings that exceeded 1.66. The fact that the specimen was singly refractive (with weak anomalous double refraction) was the main distinguishing property separating it from tan-

zanite, and this also meant that it lacked tanzanite's characteristic pleochroism. Additional features of the stone included a specific gravity of 4.11; weak blue fluorescence to long-wave UV radiation, and weak-to-moderate chalky yellow and blue fluorescence to short-wave UV; and weak bands seen at approximately 500 and 600 nm with a desk-model spectroscope. The stone was fairly free of inclusions, and magnification revealed only a few pinpoints.

Both FTIR and Raman spectroscopy provided spectra similar to those previously recorded for manufactured glass, which confirmed the identification. EDXRF analysis by Sam Muhlmeister showed silicon as a major constituent with numerous trace elements, including calcium, zinc, strontium, barium, zirconium, antimony, and lanthanum.

The important point with an imitation such as this is one that we continue to mention on a regular

basis: Always use all the available information in making an identification. A quick R.I. reading coupled with the violetish blue color could easily lead to the wrong conclusion. However, if tanzanite is suspected, the characteristically strong pleochroism should be readily detectable if the material is genuine.

Elizabeth Quinn

Large Clam "PEARL"

An antique brooch received in the East Coast laboratory generated great interest among our staff members. It showcased a single large clam "pearl" that was bezel set in a yellow and white metal mounting and surrounded by a single row of brilliant old mine and old European cut diamonds (figure 10). The almost round button-shaped mottled purple-and-white clam "pearl" measured approximately 20 mm in diameter and showed a prominent alveolar (honeycomb) growth pattern readily visible with the naked eye. This distinct growth structure is predominantly found in such concretions as clam pearls. On closer scrutiny, we noticed another feature commonly found in other non-nacreous pearls: Each individual honeycomb cell also displayed a microstructure similar in appearance to the flame-like structure that characterizes so many concretions from various mollusks. Even though this microstructure was only moderately developed, it still produced a sheen-like effect in those areas where the surface was still smooth and had not been slightly worn off over time. Such a remarkable non-nacreous "pearl"—with an alveolar structure and a flame-like microstructure—is indeed rare. G. F. Kunz and C. H. Stevenson report in *The Book of the Pearl* (Dover Publications, New York, 1908) that the common hard clam *Venus mercenaria* from the Atlantic Coast of the U.S. can, on rare occasions, produce such large "pearls."

KNH

Figure 10. This antique brooch showcases a very rare clam "pearl" that measured approximately 20 mm in diameter.





Figure 11. These five sapphires, 3.07–9.22 ct, all have the unusual color zoning associated with what may represent a “new” treatment.

Blue SAPPHIRES with Unusual Color Zoning

A possible new treatment for blue sapphires has begun raising questions throughout the industry. The appearance of this treatment has already been reported on the Internet and in a few trade publications (see, e.g., K. Scarratt, “A review of preliminary data on heated blue sapphires with an unusual color distribution—A work in progress,” <http://www.agta.org/consumer/news/20031112gemstone->

[update.htm](http://www.agta.org/consumer/news/20031112gemstone-update.htm), Nov. 12, 2003; D. Federman, “Blue devils,” *Modern Jeweler*, November 2003, p. 9).

The concern with these stones, which typically are an attractive blue (figure 11), focuses on the presence of unnatural color zoning, which consists of a dark blue central zone with curved irregular edges that is surrounded by a light blue to near-colorless zone (figure 12). The pale outer zone varies dramatically in depth and does not conform to the outside surface of the faceted stone. Rather, it

appears to conform to the surface of the original rough. This feature has been seen primarily in sapphires of Sri Lankan and Madagascar origin that show evidence of exposure to high temperatures.

The GIA laboratory has examined dozens of these treated sapphires since they were first noticed on the market in the beginning of this year. When this zoning was first observed, it had so many characteristics similar to those seen in some Be-diffused blue sapphires that we expected it to be, in fact, the result of Be diffusion. We have seen several known Be-diffused stones with surface-conformal colorless rims surrounding blue cores, at times with blue color bands ending abruptly in a scalloped pattern (figure 13). Although the pale (near-colorless to light blue) rims around the new sapphires tended to be significantly deeper and usually did not conform to the faceted shape of the stone, the similarities were nonetheless striking.

To date, however, no significant amounts of beryllium have been found in these suspicious stones. Closer inspection of the zoning revealed other interesting distinctions from known Be-diffused corundums.

When viewed in certain directions, many of these sapphires showed a scalloped appearance at the interface between the darker center and the lighter rim (figure 14). This

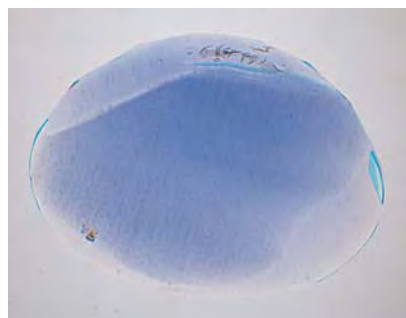


Figure 12. Sapphires subjected to this treatment typically have a dark blue central zone that is separated from a paler outer zone by curved edges, as can be seen in this 6.55 ct stone at 10× magnification with immersion.

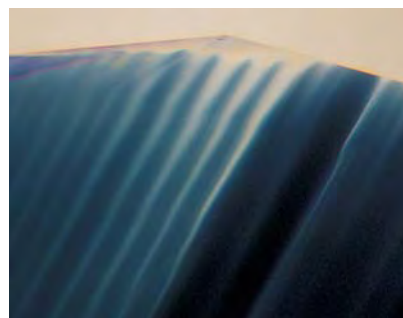


Figure 13. Be-diffused blue sapphires also typically have blue cores, but the pale rims are surface conformal, and the blue color bands in the core end abruptly at the outer rim. Immersion, magnified 40×.

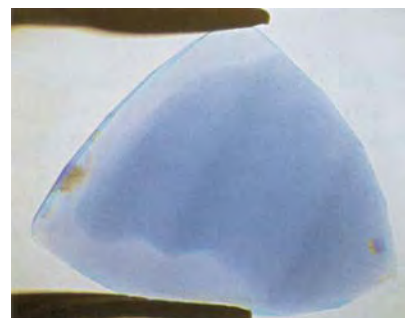


Figure 14. Note the scalloped edge characteristically seen at the interface of the darker core and paler rim in these “new” treated sapphires. Immersion, magnified 10×.



Figure 15. In many of these treated sapphires, when observed from certain orientations, the interface between the light and dark areas was marked by a colorless line all along the dark core. Immersion, magnified 12 \times .

scalloped edge seemed to be directly related to the tone of the color banding: The pale rim extended deeper into the lighter blue bands of the core than into the darker bands. This seemed to suggest that, as with Be diffusion, the depth of penetration of this treatment is dependent on the inherent chemistry of the stone.

The color banding of these stones often did not simply disappear at the light/dark interface. Rather, this interface usually manifested itself as a very narrow, colorless line extending all the way around the darker blue core (figure 15). In many examples, the color banding abruptly became much lighter on the other side of this line, and appeared distinctly curved (figure 16), but continued on, making the outer area light blue rather than colorless.

As stated earlier, the depth of this outer portion varied considerably from stone to stone. It sometimes encompassed the majority of the sapphire, leaving an area of darker color

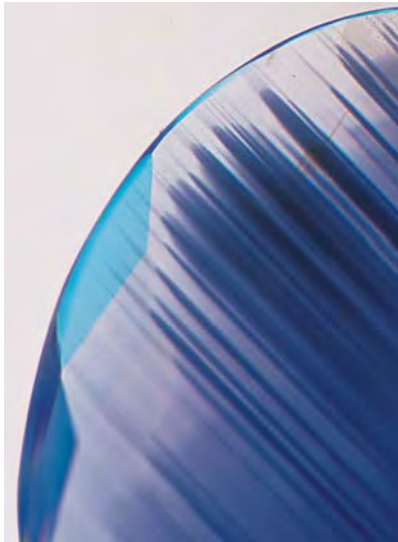
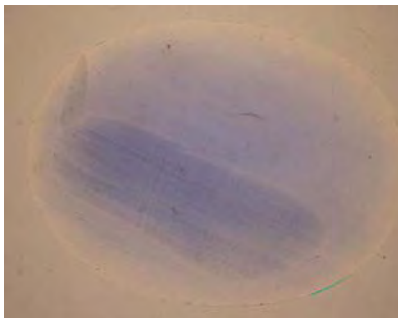


Figure 16. Often the color bands in the possibly new treated sapphires do not end at the light blue or near-colorless rim, but rather get abruptly lighter and appear curved. Immersion, magnified 10 \times .

that represented only 25–30% of the whole (figure 17).

The exact nature of this treatment, as well as its specific effect on the stones treated, is still not understood.

Figure 17. In some samples, such as this 4.80 ct sapphire, the light area encompassed most of the stone. This sapphire, the center one in the back row of figure 11, was also lighter than those we examined that had a smaller light area. Immersion, magnified 10 \times .



One theory that has received considerable attention involves the partial dissolution of the original sapphire during this “new” heating process and subsequent regrowth (i.e., of synthetic sapphire) over the remaining, original stone. To investigate this new treatment further, in mid-December GIA researchers Christopher P. Smith and Matthew Hall traveled to Sri Lanka, where they attended experiments by Tennakoon Punsiri of Punsiri Gems, the man who developed this process. The heat treatment procedure (which is proprietary to Mr. Punsiri) had a dramatic effect on the color of most of the samples they had treated during their visit (figure 18), and initial observation suggested that the unusual color concentrations indicated above were produced in more than two-thirds of them.

On the basis of detailed microscopic examination of the samples after treatment, Smith and Hall concluded unequivocally that no synthetic sapphire growth had taken place. Furthermore, they did not witness the use of any fluxing agents during the heating experiments, and no evidence of flux was present on or around the samples brought back to the U.S. for additional analyses. To further our understanding of this

Figure 18. This 9.74 ct sample (approximately 16.1 \times 10.3 \times 6.3 mm) was sawn in two prior to the heat treatment experiment, and the half on the left was retained as a control. Note the dramatic change in color in the half on the right following treatment.





Figure 19. This sequence of photomicrographs shows two resin-filled cavities on the pavilion of a Sri Lankan spinel under different lighting conditions. In the left and center images—taken with darkfield and surface-reflected fiber-optic illumination, respectively—excess filler on the surface and its relationship to the filled cavities is evident. The image on the far right clearly shows the largest apatite-shaped cavity as well as the gas bubbles in the fillers in both pits. Magnified 10 \times .

treatment, a cooperative effort between GIA researchers, colleagues from other laboratories, and outside experts has brought full resources to bear on this problem. We will continue to report significant developments as they occur.

SFM

SPINEL with Filled Cavities

While the filling of cavities and surface-reaching fissures in emerald, ruby, and sapphire with resin or glass is relatively common and always suspected by gemologists, this type of treatment is virtually unknown in spinel. During a buying trip to Sri Lanka, gemologist Mark Smith of Thai Lanka Trading Ltd., Bangkok, encountered an oval mixed-cut spinel that appeared to have been treated in this manner. A cursory field examination with a 10 \times hand loupe revealed the presence of several rounded crystal inclusions, as well as two pits on the pavilion that obviously had been filled.

Routine gemological testing of this transparent 4.79 ct stone at the

West Coast laboratory confirmed the identification as spinel. Examination with a gemological microscope and a fiber-optic illuminator also confirmed the presence of several slightly rounded (corroded) near-colorless transparent-to-translucent mineral inclusions, which had the general morphology of those that have previously been identified as apatite in Sri Lankan spinel. In addition, the two cavities clearly visible on the pavilion apparently had been filled after polishing, since no attempt had been made to clean the excess filler from the surface (see figure 19, left and center). Both pits contained gas bubbles in the filler, and the largest of the two (as shown in figure 19, right) had the general shape of an apatite crystal, so we suspect that the pits were created when some of the mineral inclusions were plucked out during faceting.

Since spinel is a very durable gem material with a high melting temperature, some form of glass filling was suspected, as this type of treatment is frequently encountered for pits and surface-reaching cracks in rubies and sapphires. Micro-hardness and inden-

tation testing, however, revealed that the filler was much softer than glass. When a small spot of the filler was tested with a thermal reaction tester (hot point), the immediate reaction and odor produced were similar to what would be expected for some type of clear-drying organic resin.

In this instance, the identification of these resin-filled cavities was made relatively easy by the presence of the gas bubbles and the complete lack of repolishing to remove the excess filler. Such obvious surface evidence is rarely encountered even in stones that are routinely filled, such as rubies and sapphires, since they are commonly repolished after treatment. This is the first spinel we have seen treated in such a manner.

JIK and Maha Tannous

PHOTO CREDITS

Maha Tannous—1, 7, 9, and 11; John I. Koivula—2, 3, 8, and 19; Vincent Cracco—4; Shane F. McClure—5, 6, and 12–17; Elizabeth Schrader—10; Matthew Hall—18.

For regular updates from the world of **GEMS & GEMOLOGY**, visit our website at:

www.gia.edu/gandg