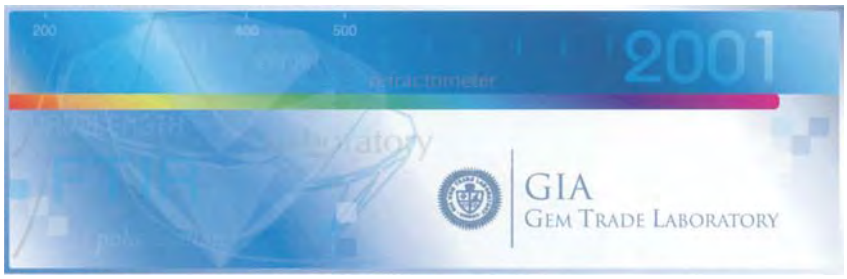
Gem Trade **LAB NOTES**



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DIAMOND

"Banded" Twinned Crystal

Diamond manufacturers commonly encounter a wide range of colors and shapes in the crystals they facet. They are routinely required to make quick cutting decisions throughout the faceting process. Occasionally, however, a crystal is so unusual that the manufacturer stops to ponder it. Such a crystal recently was brought to the attention of the East Coast laboratory by one of our clients. Although the appearance of the crystal was intriguing, we were also interested in gaining a better understanding of its growth history.

The 14.07 ct elongated diamond crystal (measuring 21.78 × 7.62 × 7.59 mm) appeared "banded," with near-colorless portions on each end and a black area in the middle (figure 1). Stepped twin boundaries (twinned according to the spinel law, that is, with the twin plane parallel to one of the octahedron's faces) were found in both near-colorless portions. The twin planes, although parallel, were shifted slightly in position. However, the two near-colorless portions had different morphologies. The portion on the left in figure 1 had a flattened triangular twinned morphology with typical re-entrant corners in the boundaries, whereas the near-colorless portion on the right had a slightly elongated triangular shape without clearly visible twin re-entrant corners. The black portion was irregular in shape, but roughly spherical, with very rough surfaces; it had a submetallic luster. The boundaries between the black center and the near-colorless ends were clearly demarcated.

Figure 1. The unusual "banded" structure of this 14.07 ct twinned diamond crystal is the result of a complicated growth history.

Because the black center section appeared so different from the end portions, we thought there might have been another material involved. However, the measured specific gravity of the piece was 3.51 (compared to 3.52 for diamond), which indicated that only diamond was present in significant amounts. Infrared spectra suggested that all three parts of the diamond were type Ia, with high levels of nitrogen. Raman spectra collected from the three portions confirmed this result. Several black inclusions with irregular shapes were observed in the near-colorless portions, but none of these was located close enough to the surface of the crystal for Raman analysis.

Detailed observations at high magnification, using an optical microscope (up to 150×) and a scanning electron microscope (up to 500×) at the California Institute of Technology (Caltech), revealed entirely different surface features for the near-colorless and black portions, which were apparently due to a combination of growth and dissolution (figure 2). On the flat surfaces of the near-colorless portions, the growth features appeared as macroscopic parallel steps and triangular hillocks, particularly in the boundary areas close to the black portion. The dissolution features included tiny etched trigons, with an orientation opposite that of the triangular hillocks, and etch figures with rhombic forms that followed the symmetry of the structure of the twin boundary. Also, the twin boundaries were slightly bent in several places. The rough surface of the black portion consisted of irregular steps and small crystallites with polygonal forms. When examined with strong transmitted light, this portion revealed a rather complex internal structure, with a near-colorless transparent core and a black rim.

We concluded that this diamond was a single crystal with a complicat-

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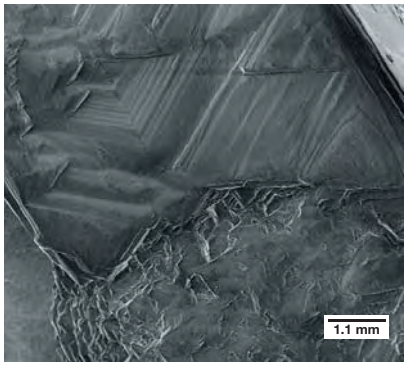


Figure 2. The surface features of this near-colorless portion (top of photo) were very different from those of the black portion (bottom of photo) of the diamond crystal. This backscattered-electron SEM image shows parallel steps, triangular growth hillocks, a twin boundary (upper right), and rhombic dissolution figures on the colorless portion, while it also reveals the rough surface with irregular steps and small crystallites in the black portion.

ed growth history. The black and near-colorless portions represent different stages and conditions of growth. The spherical central portion was formed first and grew rapidly (as evidenced by the small crystallites on its surface). The black color is probably due to numerous tiny and black inclusions located mainly on grain boundaries of the small diamond crystallites. The two flattened and twinned near-colorless portions then grew from both sides of the central portion, by means of a spiral growth mechanism.

Taijin Lu and John M. King

With a Carbonate Inclusion

Recently one of the diamond graders in the West Coast laboratory became suspicious of the appearance of a surface-reaching inclusion in a 1.43 ct diamond that could be described as a wide feather or a deep, narrow cavity. The grader thought that the crackled texture observed in the interior of the inclusion might be due to the presence of some type of artificial filling. When the diamond was brought into the Gem Identification laboratory, we immediately noticed that this texture was very similar to what we had seen in some wide cleavages that had been artificially filled.

Close examination with 15× magnification revealed that where the cavity reached the surface, the transparent material in it was slightly undercut and had a rough texture; it also contained numerous highly reflective, nearly parallel compression cracks (figure 3). The width of the cavity at the surface made it possible to analyze the filler using the laser Raman microspectrometer. The results were both immediate and surprising. The analysis showed the undeniable presence of a carbonate in the crack, although an exact match to a specific carbonate could not be made. To confirm this visually, we placed a minute droplet of 10% hydrochloric acid solution on the crack at the surface of the diamond. The resulting effervescent reaction was as expected for a positive carbonate-HCl acid spot test.

The carbonate calcite has been identified previously as an epigenetic inclusion in diamond (that is, it entered the diamond some time after its formation; see J. W. Harris, "Diamond geology," in J. E. Field, Ed., *The Properties of Natural and Synthetic Diamond*, Academic Press, London, 1992, p. 363). Therefore, the possibility of a carbonate filling the interior of a surface-reaching cavity in a diamond is not unheard of. This, however, is the first time we have identified a carbonate in a polished diamond. Whether or not it would be possible to use a molten carbonate as a diamond filler is not known, although its susceptibility to attack by acid makes this unlikely. We therefore concluded that this was a naturally occurring epigenetic inclusion.

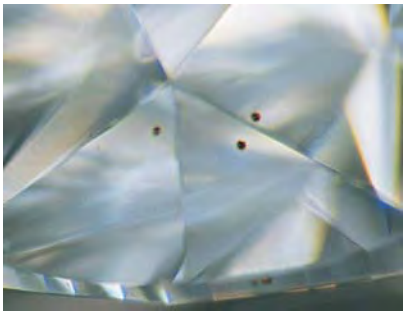
JIK and Maha Tannous

Figure 3. Numerous subparallel, reflective compression cracks in the carbonate inclusion in this diamond resemble a texture noted in some fillings in diamonds. Magnified 15×.

With Unusual Mineral Inclusions

An approximately 1 ct near-colorless round-brilliant-cut diamond recently submitted to the East Coast laboratory was found to contain some rather interesting inclusions. During the clarity grading process, three tiny (0.05 mm) dark red-brown to black inclusions, each surrounded by what appeared to be a brown radiation halo, were observed under the crown (figure 4). These inclusions looked rounded

Figure 4. Too deep in the host diamond to be analyzed by laser Raman microspectrometry, these three tiny (0.05 mm) dark red-brown to black inclusions are surrounded by what appear to be brown radiation halos. They are believed to be either strontian K-Cr loparite or Cr-chevkinite. The inclusion seen here through the girdle of the stone is a reflection artifact. Magnified 40×.



or semi-spherical in form, and proved to be too deep within the diamond host for Raman analysis.

On the basis of visual examination of similar inclusions in about five other diamonds, as well as a search through the literature, we suspected that these inclusions were either strontian K-Cr loparite or Cr-chevkinite. These rather unusual radioactive inclusions are similar in color to those found in this particular diamond, although the latter were too small to provide a radioactivity reading. Loparite is isometric and chevkinite is monoclinic. Because the inclusions in this diamond looked somewhat spherical, loparite is perhaps the more probable choice (chevkinite tends to form as somewhat elongated, spike-shaped crystals). Mineral inclusions in diamonds, however, are often influenced by the diamond structure. Through a process known as xenomorphism, even inclusions that are not isometric, such as chrome diopside, can look as if they have an isometric form. This means that chevkinite could not be ruled out as a possibility.

Strontian K-Cr loparite and Cr-chevkinite were first discovered as inclusions in diamonds that were tiny fragments (approximately 1.5 × 1.5 mm) from the River Ranch kimberlite in Zimbabwe. These inclusions had a high content of radioactive thorium, and contained the highest amounts of rare-earth and radioactive elements ever reported in inclusions in diamonds; small, bright green radiation clouds surrounded each of the radioactive inclusions. The green color indicated that the clouds had developed at a temperature below 600°C, and had not been exposed to a temperature higher than that since they were formed. The fact that the clouds in the diamond we examined were brown indicates that the stone was heated above 600°C at some point (in nature or artificially), as illustrated in J. I. Koivula's *Microworld of Diamonds*, Gemworld International, Northbrook, Illinois, 2000. A good reference for chevkinite and loparite as inclusions in diamond is

M. G. Kopylova et al., "First occurrence of strontian K-Cr loparite and Cr-chevkinite in diamonds," *Russian Geology and Geophysics, Vol. 38, No. 2, Proceedings of the Sixth International Kimberlite Conference, Vol. 2: Diamonds: Characterization, Genesis and Exploration*, Allerton Press, New York, 1997, pp. 405–420.

JIK and Maha Tannous

FELDSPAR
With Chrome Diopside Inclusions

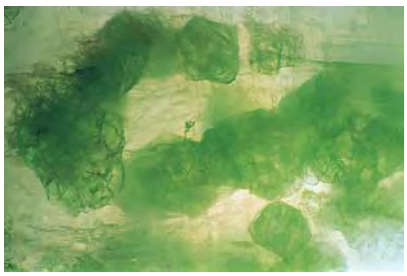
The West Coast lab recently examined an unusual double cabochon that standard gemological testing confirmed to be orthoclase feldspar. The 40.14 ct cabochon, which was 22.48 mm long (figure 5), was obtained in Mogok, Myanmar, by Mark Smith, a gemologist and gem dealer in Bangkok. The stone contained a number of relatively large green inclusions, so that in general appearance it resembled the white plagioclase feldspars from the Philippines that contain deep green uvarovite garnet inclusions. Those feldspars, however, do not show adularescence, which this Burmese moonstone did.

Examination with a microscope revealed that the randomly arranged, transparent-to-translucent green inclusions were cracked and well rounded, with no recognizable crystal faces (figure 6). These features suggested a protogenetic origin for these inclusions with respect to the feldspar; that is, they formed before their host. Raman analysis established that the inclusions were diopside, and not (as first suspected) a species of garnet. EDXRF analysis of the inclusions detected the presence of chromium, which is the probable cause of their green color. This is the first example of moonstone with chrome diopside inclusions we have encountered in the laboratory.

JIK, Maha Tannous, and Sam Muhlmeister

Figure 5. This 40.14 ct double cabochon from Myanmar is an interesting mixture of adularescent orthoclase feldspar and chrome diopside.

Figure 6. The rounded appearance and cracked texture of the chrome diopside inclusions in their orthoclase host suggest that the inclusions are protogenetic. Magnified 5×.



GIBBSITE
Dyed to Imitate Nephrite

Recently, the West Coast laboratory received a 10 × 7 × 3.5 mm oval bead for an identification report. At first glance, the "spinach" green color of the bead (figure 7) appeared to be typical for nephrite jade. However, the optical properties and structural characteristics were quite different from those expected for nephrite.

Using a standard gemological refractometer, we obtained a spot

Figure 7. This 10×7×3.5 mm bead, which looks like nephrite jade, was identified as dyed gibbsite.

R.I. of 1.58. This reading was too low for nephrite jade (which has an average R.I. of 1.61). The specific gravity, determined by the hydrostatic method, was approximately 2.09. This figure is also significantly lower than nephrite (about 2.95). When we examined the bead with a gemological microscope, we observed a dense granular structure; an even coloration; some small, irregular-shaped, opaque whitish particles; and a few dark brown needle-like inclusions. The material was very soft, and was easily scratched with a needle probe. These properties also indicated that the bead had not been fashioned from nephrite jade.

Next, using a desk-model spectroscope, we noticed strong absorption bands at 560, 600, and 650 nm. The bead fluoresced moderate yellowish green to long-wave ultraviolet radiation, but was inert to short-wave UV. Since the standard gemological tests were inconclusive, advanced testing was required to identify this material. With the client's permission, a minute scraping was taken from an inconspicuous place (the inside of the drill hole) for X-ray diffraction analysis. The pattern obtained matched gibbsite, a clay-like aluminum hydroxide. The material had been dyed to simulate nephrite jade. This was the first jade imitation of this type we have seen in the Gem Trade Laboratory, although on a number of occasions we have encountered dyed gibbsite as a realistic imitation for turquoise (see Lab Notes: Summer 1983, p.117; Spring 1988, p. 52). *KNH*

Devitrified GLASS Resembling Jade

Although jadeite is one of the more common gem materials to be imitated, seldom is the imitation mounted in an ornate setting made with quality craftsmanship. The East Coast lab recently received just such a piece in the form of a pendant.

The fully backed white metal pendant was stamped "18K." It included numerous channel-set transparent near-colorless baguettes, as well as some transparent near-colorless marquise brilliants, all bordered by mill-grain detailing. The pendant showcased a large, thin, semi-transparent to translucent green tablet—carved with the image of a stork—which measured approximately 33.40 × 40.25 × 1.65 mm (figure 8).

The carved material showed an uneven "patchy" green color throughout, which to the untrained observer might have appeared similar to the aggregate structure of jadeite. We obtained a spot refractive index of 1.60 using a standard gemological refractometer. Patches of the tablet fluoresced a medium chalky yellow to long-wave UV radiation and a very weak chalky yellow to short-wave UV. Using a standard gemological microscope (50×) and fiber-optic lighting, we observed patches of tiny gas bubbles. Higher magnification (200×) revealed a fern-like structure that is commonly seen in the manufactured glass known in the trade as "Meta-jade." The partially devitrified structure confirmed that the carving was a manufactured glass.

Ann-Marie Walker
and Wendi M. Mayerson

Figure 8. The 33.40×40.25×1.65 mm green carving in this pendant proved to be devitrified glass.

Figure 9. Assembled cultured blister pearls such as the ones in this ring (left) were first produced by Kokichi Mikimoto in 1890. Note on the back the concave cups into which they fit precisely (right).

PEARLS

Early Japanese Assembled Cultured Blister Pearls

The East Coast lab recently received for identification an intricate white metal ring set with three pearls (figure 9, left). The setting was stamped "18KT WHITE GOLD" and included numerous old European- and single-cut stones. The ring had a concave cup setting for each of the three partially drilled pearls (figure 9, right), which measured an average of 5.18 mm in diameter.

Magnification revealed a nacreous appearance consistent with natural and cultured pearls. X-radiography, however, suggested that the internal structure was not typical of the natural or cultured pearls generally seen in the lab today. Each "pearl" had three components: a layer of nacre, a shell bead with a square notch cut into it, and an oddly shaped mother-of-pearl backing cut to fit into the square notch (figure 10). Most unusual was the fact that the mother-of-pearl backing was rounded on the bottom to give the overall appearance of a fully round pearl. We therefore concluded that these were assembled cultured blister pearls.

Figure 10. This X-radiograph reveals the unusual assemblage of the cultured blister pearls shown in figure 9.

We have previously reported on similar cultured pearls in the Lab Notes section (Summer 1983, p. 116; Spring 1988, pp. 49–50; Spring 1994, pp. 44–45). They were produced in Japan prior to the successful culturing of solid round pearls. Although the lab has not seen very many of them, we know that Kokichi Mikimoto was marketing assembled cultured blister pearls as early as 1890, with production exceeding 50,000 annually.

While one might at first assume that these assembled cultured blister pearls were set in the ring to replace lost or worn natural pearls, the fact that they probably represent early Mikimoto production makes it more likely that they were original to the ring itself. In addition, they fit precisely into the cupped backs, another indication that they were original to the ring. Knowledge of their history can aid in dating this piece of estate jewelry.

Ann-Marie Walker
and Wendi M. Mayerson

Faceted Cultured Pearls, Dyed Black

An 8 mm black faceted pearl was sent to our West Coast laboratory for an identification report. The client had noticed unusual "color spots" that made him question whether it was indeed a natural-color Tahitian cultured pearl. Although we cannot determine a pearl's provenance, we can determine its identity, whether it is natural or cultured, and whether or not it has been treated.

The item had an attractive black bodycolor and showed strong purplish pink overtones (figure 11). Most of the polygonal facets had been well placed and polished, leaving the surface with a smooth finish. X-radiography revealed a fairly large bead nucleus, which confirmed that this was a cultured pearl. Using 10× magnification with strong overhead illumination, we observed that the nacre was actually dark brown. This color was unevenly distributed, and concentrations in some areas gave the surface a slightly spotted appearance. Magnification also revealed that some facets had been damaged extensively during fashioning. Fairly deep grooves (some straight and others circular) were noticeable. On some facets the top nacre layer had been removed, exposing an almost colorless layer of nacre (figure 12). Along the rims of those exposed areas was an opaque, light brown deposit of unknown identity.

The reaction to long-wave UV radiation was quite peculiar. The exposed near-colorless nacre layer fluoresced chalky yellowish white, similar in appearance to natural-color light nacre shell layers. The black nacre surface, however, did not show

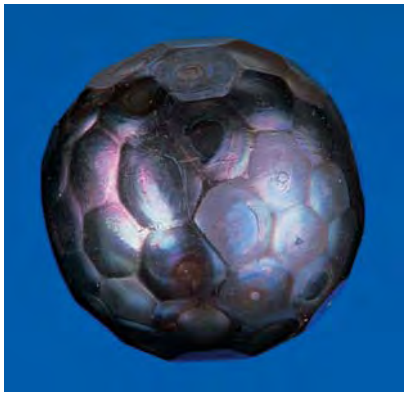


Figure 11. This 8 mm black faceted cultured pearl revealed evidence of treatment. Note the different color of the lower nacre layer that was exposed on some facets during fashioning.

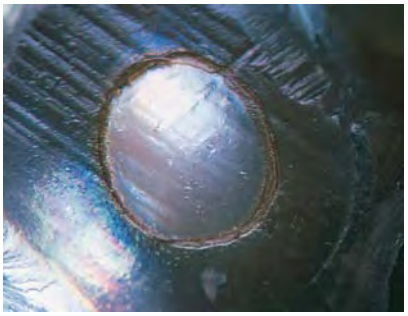


Figure 12. With magnification (here, 15×) a second, almost colorless, nacre layer was revealed, and a light brown deposit was seen where the top nacre layer had been removed during fashioning.

any fluorescence. Natural-color black nacre fluoresces either reddish brown or red to long-wave UV. The absence of this type of fluorescence in the top nacre layer proved that it had been treated. EDXRF chemical analysis revealed the presence of silver, which further substantiated our conclusion that the cultured pearl had been treated to attain its black color. As to the cause and nature of the brown deposit around the rims of exposed areas, we could only speculate that it was a byproduct of the faceting and treatment process. *KNH*

Figure 13. This strand of 34 "baroque" imitation pearls was sold as "shell pearls."

A New Imitation: "Shell Pearls" with a Calcite Bead

This year in Tucson, we purchased a strand of 34 imitation "baroque" pearls (12.50–11.60 mm × 10.30–9.75 mm; figure 13), which were sold as "shell pearls" by Tiger Trading, Fresh Meadows, New York, at the GJX Show. These imitation pearls were offered in rounds ranging from 8 to 14 mm, as well as in "baroque" shapes such as those in the strand we purchased. They came in a variety of colors. Besides the "Tahitian" color scheme of our strand, "South Sea" and standard "akoya" colors were available. The seller reported that they were mother-of-pearl beads—as are sometimes used in imitation pearls—that had been coated to look like many different types of pearls.

The regularity of shape of the "baroque pearls" and the multiple layers visible at the drill holes (figure 14) seemed to support the seller's description. However, a close inspection in reflected light also revealed what appeared to be facets under the coating. Given the unusual shape and uniformity of these "pearls," we were curious to learn whether their centers were in fact shell.

To examine the core material, we decided to sacrifice one of the

Figure 14. The view down the drill hole readily revealed that this imitation pearl had multiple layers of some type of coating. Magnified 50×.

Figure 15. The coating removed from the imitation "shell" pearl was found to consist of two types of layers (left): external transparent colorless layers and internal opaque light gray metallic layers. Gemological testing identified the bead nucleus (right) as calcite.

imitation pearls in the strand. Using a procedure we normally would not perform, we immersed a light gray "shell pearl" in acetone (standard nail polish remover) in an attempt to break down the coating and expose the bead. After 30 minutes, the coating was soft enough to be squeezed with tweezers. In fact, the coating fit loosely, like a plastic bag around a hard center. When the object was squeezed with tweezers, a dark cloud or "puff" was exuded through the drill hole into the nail polish remover. More squeezing ruptured the coating and released the bead nucleus.

As can be seen in figure 15, the coating was quite thick and consisted of two different types of layers—transparent colorless external layers and opaque metallic light gray internal layers. It was part of the internal colored material that, because it was in the process of dissolving, had squirted out of the drill hole when the assemblage was squeezed in acetone.

The bead was translucent white and banded (again, see figure 15). The spot reading, taken with a standard refractometer, revealed a carbonate blink from 1.48 to 1.65, and the polariscope established that the material was a crystalline aggregate. On the basis of these findings and a specific gravity of 2.75, we identified the bead as calcite, rather than mother-of-pearl. Although both materials consist mainly of $CaCO_3$, mother-of-pearl—a shell material—contains organic matter and water in its structure as a result of being excreted by a living creature. It should be noted that shell beads, not calcite, are the standard nucleus for cultured pearls in the industry today. A strand of very similar appearing pearls was recently advertised for sale as "mother of pearl jewelry" in the catalogue of a major department store. A special note stated that "*Mother of pearl jewelry is derived from the inner lining of an oyster shell. This collection's magnificent luster is enhanced by a man-made protective coating.* Again, we say *caveat emptor*—"buyer beware."

Wendi M. Mayerson

PHOTO CREDITS

Maha Tannous—figures 1, 5, 7, 11, and 12; Taijin Lu—figure 2; John I. Koivula—figures 3, 4, and 6; Jennifer Vaccaro—figures 8 and 13; Elizabeth Schrader—figures 9 and 15; Ann-Marie Walker—figure 10; Wendi Mayerson—figure 14 .