G&G Lab Notes

Editors
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Atypical Freshwater CULTURED PEARLS with Damaged Nacre

Recently the East Coast laboratory received four baroque-shaped pearls ranging in size from 9.07 × 13.50 × 14.03 mm to 9.32 × 11.34 × 15.77 mm and weighing between 10.63 and 13.60 ct. On each of the four samples, the outermost surface nacre layers exhibited large holes and gaps, revealing one or more distinct nacreous layers underneath (figure 1). These underlying nacreous layers displayed good luster and overlapping platelet surface structures, and the large gaps between the inner and outermost nacre layers indicated that smaller pearls had been used as nuclei during the culturing process.

Further gemological testing by energy-dispersive X-ray fluorescence (EDXRF) and inductively coupled plasma–mass spectrometry (ICP-MS) confirmed that both the outer and inner nacre layers originated from a freshwater environment. Some of these pearls contained widely varying amounts of trace elements (i.e., Mn and Mg) between the outer and inner layers, but previous studies have found that uneven distribution of trace elements can also occur in a pearl's cross section (R. Lu et al., "Operational considerations of EDXRF, LA-ICP-MS, and photoluminescence techniques in the analysis of pearls," Summer 2011 *G&G*, pp. 149–150).

Editors' note: All items were written by staff members of GIA laboratories.

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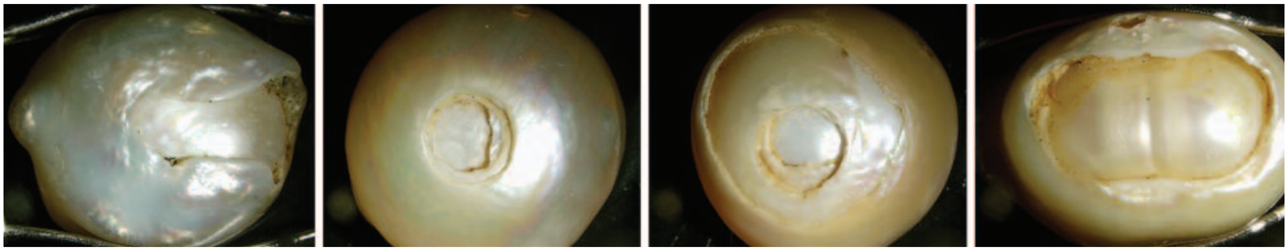
Two did not show any distinct internal demarcation when examined with X-radiography in various directions (figure 2). This lack of a clear internal boundary may be due to multiple culturing attempts using the same pearl as a nucleus, which can cause overlapped internal structures and multiple distinct nacreous layers under the surface (figure 1, sample 3). In addition, structures typical of non-bead cultured pearls were observed in these micro X-radiographs, suggesting that freshwater cultured pearls were used as nuclei during the culturing process.

Finally, photographs of the X-ray fluorescence reactions of all four pearls were taken in a dark environment (figure 3). Various intensities of green and yellowish green fluorescence could be observed within each nacreous layer, presumably due to varying concentrations of manganese between layers and among the pearls themselves (H.A. Hanni et al., "X-ray luminescence, a valuable test in pearl identification," 2005 *Journal of Gemmology*, 29, 5/6, pp. 325–329). More interestingly, reddish fluorescence was also observed around the damaged areas of the nacre in samples 3 and 4. This was possibly caused by foreign material between the "nucleus-pearl" and the outer nacre layers, which may also contribute to the lack of distinct demarcation observed in the cultured structure.

While similar types of pearls with intact outer nacre surfaces have been reported on previously (E. Strack, "Chinese freshwater cultured pearls beaded with baroque freshwater cultured pearls," Fall 2011 *G&G*, pp. 244–245), the ambiguous internal structures, interesting X-ray fluorescence properties,

Figure 1. These four atypical freshwater cultured pearls with damaged nacre reveal one or more distinct nacreous layers underneath. Left to right: samples 1–4.



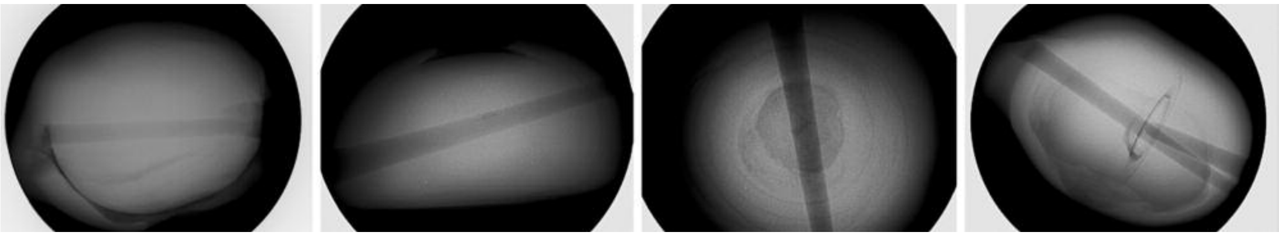


Figure 2. X-radiographs show partial demarcation between the outer and inner structures of cultured pearls 1 and 2, but fail to reveal any clear boundaries in samples 3 and 4.

and large gaps in the outer nacre layers of these four samples offer a rare view of their internal physical structure and appearance, obtained free of destructive testing.

Chunhui Zhou

DIAMOND

Calcium Fluoride Coating Found On 13 "Fancy" Pinks

Coating gemstones to alter or improve their color is the oldest known enhancement method. Early coatings, particularly on diamond, were easily detected, often visible using a 10× loupe. But advances made after World War II resulted in more durable coatings and films that were subtler and harder to detect.

A common coating was calcium fluoride (CaF_2) doped with gold (Au) nanoparticles. CaF_2 causes a broad absorption band at about 520 nm, similar to the band at about 550 nm that largely accounts for the pink color of natural diamonds.

Recently submitted to the East Coast laboratory for identification and grading was a group of 13 pink diamonds (figure 4). The round brilliants ranged from 0.42 to 0.50 ct and had a Fancy pink to Fancy Intense pink color. Figure 4 also shows the patchy appearance characteristic of coated diamonds. Microscopic examination using reflected light revealed obvious coating on the pavilion facets, with patchy colorless areas where it had been removed (figure 5).

The UV-visible spectra of all 13 diamonds exhibited a broad band centered at about 520 nm, not at 550 nm as expected. This band is responsible for the pink coloration, and the 520 nm position identified the coating as fluoride.

This example demonstrates that traditional treatments such as coating are still prevalent in the trade, and great care must be taken to identify them. Because coatings are not permanent, all 13 diamonds were issued identification reports disclosing the treatment.

Fish-Like Inclusion

It is always a pleasant surprise to find a new and interesting inclusion during routine observation. But an inclusion that reminds us of a particular shape, an effect known as *pareidolia* (Winter 2007 Lab Notes, pp. 363–364), is a geologic wonder.

The New York laboratory recently received a natural yellow (graded as Fancy yellow) "Cape" 0.51 ct rectangular faceted diamond exhibiting such a feature. Examination of the stone revealed a series of table-reaching fractures and clouds that seemed to form the outline and scale-like detail of a fish (figure 6). The inclusion is large in relation to the stone, encompassing nearly 75% of the table.

Figure 4. These face-up images of 0.45 ct and 0.5 ct pink round brilliants are representative of the 13 coated pink diamonds submitted.

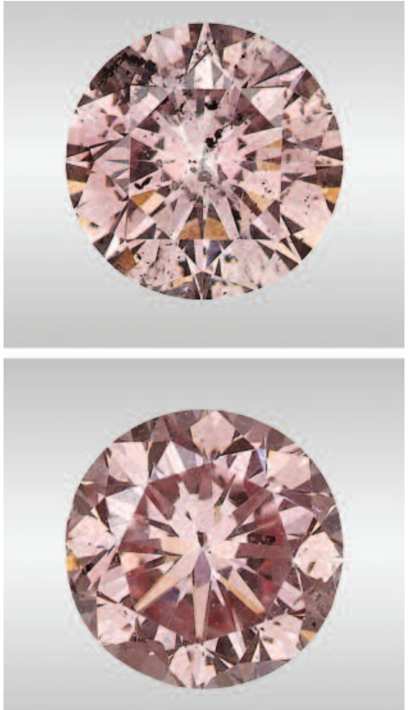
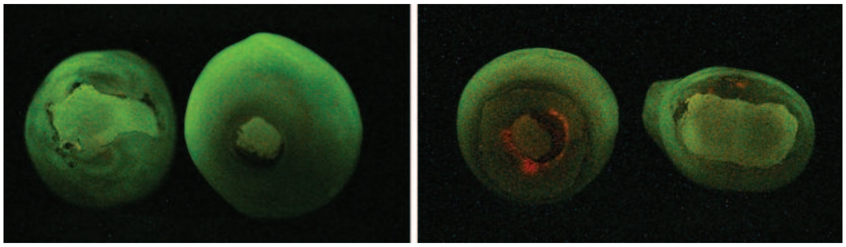


Figure 3. X-ray fluorescence images of the four cultured pearls showing various degrees of green or yellowish green fluorescence between inner and outer layers. Atypical orangy red fluorescence is also visible between the layers in samples 3 and 4.



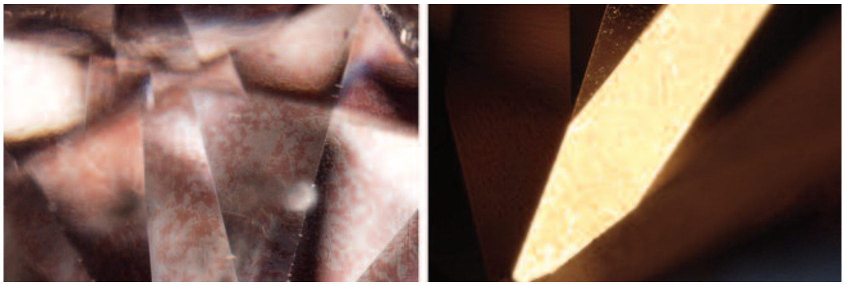


Figure 5. Photomicrographs of the diamonds from figure 4, taken with reflected light at 80× and 100× magnification, reveal the coating applied to the pavilion facets.

Figure 7. This 24.16 ct oval-cut diamond (20.47 × 14.00 × 10.72 mm) was color graded as Fancy Intense green-yellow, and identified as artificially irradiated and annealed.

Nearly all fractures in diamond result from the interaction among multiple cleavage directions (*The MicroWorld of Diamonds*, Gemworld International, 2000, p. 97). While often detrimental to the value of a stone, this inclusion serves as a useful identifier, and such findings help keep the study of gems enjoyable.

Martha Altobelli

A Large Irradiated Green-Yellow

Artificially irradiated diamonds are common in the trade, and identifying them remains a challenge. The New York lab recently identified a large diamond as artificially irradiated based on careful spectroscopic analysis and a review of the lab's database.

This 24.16 ct oval-cut diamond (20.47 × 14.00 × 10.72 mm) was color graded as Fancy Intense green-yellow (figure 7). The color was distributed evenly throughout the stone, with no color zoning detected. It showed strong yellow and blue fluorescence to long-wave UV radiation, and moderate yellow fluorescence to short-wave UV radiation. This fluorescence reaction and absence of chalkiness are seen in some natural diamonds with a similar bodycolor. Absorption spectrum in the mid-infrared region showed saturated absorptions in the one-phonon region, indicating a type Ia diamond with a high concentration of nitrogen. Also recorded were a moderate platelet peak at 1368 cm^{-1} (approximately 2.1 cm^{-1} intensity) and a weak absorption at 1433 cm^{-1}. No hydrogen-related absorption or H1b/H1c center in the near-infrared region was recorded. Absorption spectrum of this diamond in the ultraviolet/visible/near-infrared (UV-Vis-NIR) region at liquid-nitrogen temperature (figure 8) showed a moderately strong absorption from the N3 (415.2 nm) and H4 (496.0 nm) centers, as well as weak absorptions at 512.8, 594.4, and 741.2 nm (GR1). Absence of the H3 optical center was confirmed by photoluminescence spectroscopy at

Figure 6. This pavilion view of the 4.76 × 4.30 × 2.75 mm Fancy yellow diamond displays the "fish" inclusion scene. The fish appears to be facing to the right. Magnified 40×.

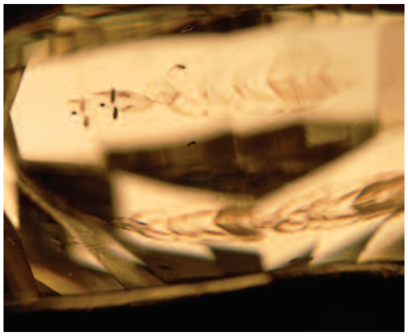
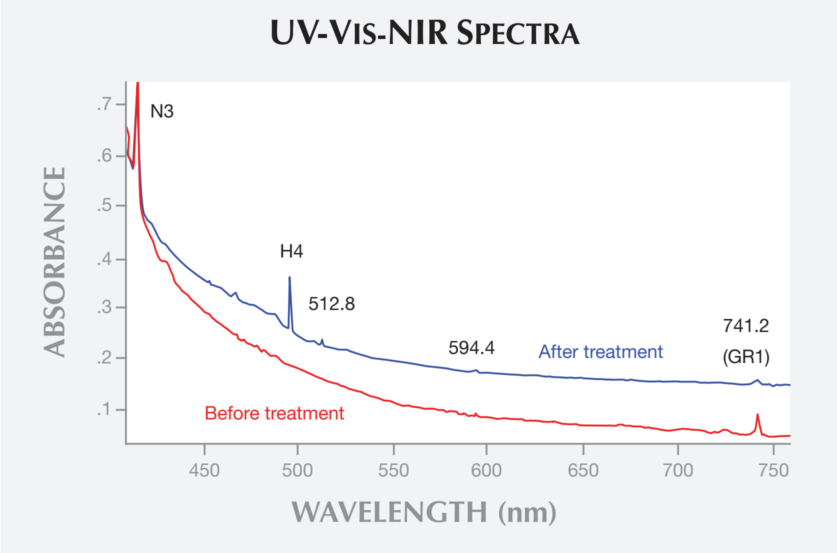


Figure 8. After irradiation and annealing treatment, a significant amount of optical center H4 was introduced. This is mainly responsible for the color improvement from Fancy brownish greenish yellow to Fancy Intense green-yellow. N3 absorptions in two spectra in this plot were normalized to the same intensity.



liquid-nitrogen temperature with 488 nm laser excitation. A fluorescence image collected using the DiamondView showed a gradual change from a predominantly blue table region to a mostly green upper pavilion and girdle area. The spectral features observed in this large diamond are rarely encountered in natural diamonds, raising the possibility of artificial irradiation treatment.

A search of the GIA database found a very good match between this stone and a previously tested diamond. The two diamonds were identical in measurements and weight, as well as their infrared absorption spectroscopic features, though the previous grade was Fancy brownish greenish yellow. This significant improvement in color appearance is attributed to the relatively high concentration of the H4 center produced by the artificial irradiation/annealing treatment confirmed in this study.

Despite its suspicious spectroscopic features, this irradiated diamond showed some gemological features normally observed in diamonds with similar natural color. This example demonstrates the value of careful spectral and gemological analysis, combined with a review of the GIA database, in gem identification.

Wuyi Wang, Paul Johnson, and Emiko Yazawa

Strong Color Zoning Reflects Complex Growth Environment

A 2.08 ct emerald-cut diamond recently submitted to the Carlsbad laboratory received a color grade of Fancy Dark orangy brown. Its strong color zoning, visible without magnification (figure 9), consisted of a dark orangy brown section with a few near-colorless bands and a near-colorless area. The orangy brown section showed numerous very small oriented reflective inclusions, visible in figure 9 only as dark clouds, that are typically associated with type IaA diamonds colored by 480 nm visible absorption bands. Both the near-colorless side and the colorless bands displayed very few oriented reflective inclusions.

The diamond's unusual fluorescence under both long- and short-wave UV illumination can be seen in figures 10 and 11, respectively. Under long-wave UV (handheld lamp) the near-colorless section showed typical blue fluorescence. The orangy brown section displayed a yellowish fluorescence, which is common in type IaA yellow diamonds. Under the higher-energy, shorter-wavelength UV excitation of the DiamondView, the distinction between the colors became even more pronounced, and the blue fluorescence of the near-colorless bands was clearly visible within the yellowish green fluorescence of the orangy brown section. Yellow phosphorescence, common in type IaA diamonds colored by 480 nm visible absorption bands, was also observed in the orangy brown area.

Infrared spectroscopy revealed a type Ia diamond with both A and B aggregates. When IR spectra were collected separately from the near-colorless and orangy brown sections, distinct differences were noted. The orangy brown section contained ~33 ppm of A-aggregated nitrogen and ~15 ppm of B-aggregated nitrogen, whereas the near-colorless section showed significantly higher concentrations: approximately 139 ppm of A aggregates and 100 ppm of B aggregates. In addition to the higher overall nitrogen content, the near-colorless portion contained a higher proportion of B-aggregated nitrogen impurities.

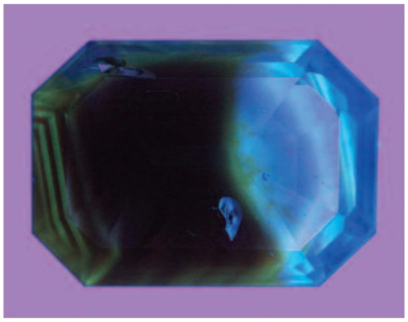


Figure 10. Under long-wave UV illumination at 10× magnification, the diamond's color zones showed blue and yellowish fluorescence.

The unique zoning observed in this diamond likely reflects changing conditions during growth. Variations in nitrogen and possibly oxygen content in the earth's interior—480 nm defects are reportedly related to oxygen impurities in diamond—could have produced the noteworthy color and fluorescence zoning that reveal the complex growth history.

Troy Ardon

Figure 9. The strong color zoning in this Fancy Dark orangy brown diamond was visible at 10× magnification.

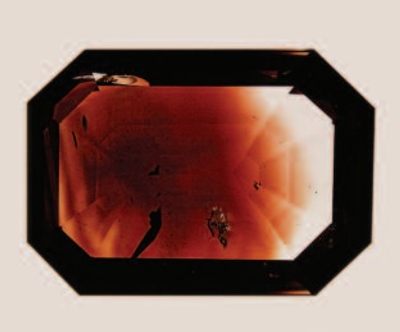
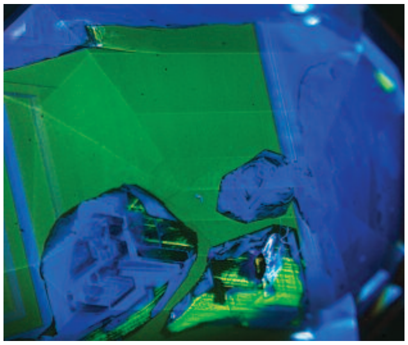


Figure 11. This DiamondView image of the color-zoned diamond shows an even more pronounced distinction between the colors.



Very Large Rough Diamond

Throughout history, the discovery of very large diamond rough has been a rare occurrence. Not only is it unlikely to survive the journey from deep in the earth to the surface, but modern extraction methods, including the use of crushers to break large rock, can reduce such specimens to smaller pieces. Therefore, the recent examination of a 1,138 ct rough diamond (62.51 × 47.61 × 45.56 mm; figure 12) provided a unique opportunity to document the largest single-crystal natural diamond ever examined by GIA. While it is possible there have been larger unreported specimens, a search of public sources (e.g., I. Balfour, *Famous Diamonds*, 5th ed., Antique Collectors Club, 2008) suggests this could be the second-largest rough diamond in history—between the 3,106 ct Cullinan and the 995 ct Excelsior, both of gem quality.

Known to be from the Democratic Republic of the Congo, this stone displayed Raman spectroscopic features characteristic of diamond. The sheer size presented a challenge with IR absorption spectroscopy analysis, as it was too large for the regular sample chamber, beam condenser, or DRIFT units. Instead, we used an infrared microscope with reflective mode and obtained a high-quality absorption spectrum. The rough was identified as a natural type IaA diamond with very high nitrogen concentration. We also detected a weak hydrogen-related absorption peak at 3107 cm^{-1}, a common feature in natural diamond. Also recorded were strong absorption bands at approximately 3300, 2920, 1700, and 1550 cm^{-1}, attributed to micron and sub-micron inclusions. Photoluminescence spectra were recorded at liquid-nitrogen temperature with various laser excitations. The main emission features were broad bands at 700 and 787 nm. The crystal also displayed a very weak blue fluorescence under long- and short-wave ultraviolet radiation, another typical feature.

Figure 12. This 1,138 ct single-crystal natural diamond (62.51 × 47.61 × 45.56 mm) is the largest ever examined by GIA.

The irregular morphology of the 1,138 ct rough was quite interesting, as diamond usually occurs in octahedral or dodecahedral forms. But such a morphology is less likely to be maintained as a large crystal forms. Numerous randomly shaped and distributed inclusions may have contributed to the irregular crystal growth. These inclusions also caused the gray color appearance.

The specimen's strong surface dissolution provided a visual document of its journey from deep in the earth. At the high temperatures in the earth's interior, diamond can remain stable only under very high pressure. As the diamond was transported to the surface, the decrease in pressure would have dissolved the stone unless it made the journey rapidly. The surface of the 1,138 ct crystal speaks to the change that took place during its journey and represents the fine line between our experiencing this rare item and never having known it at all.

John M. King and Wuyi Wang

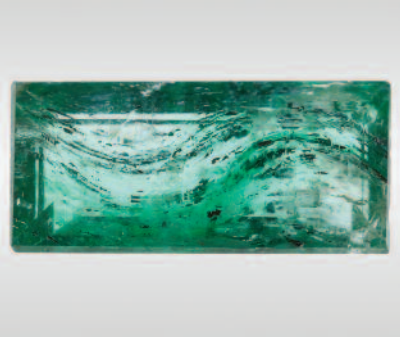
Unusual Curved Color Zoning In EMERALD

While curved color zoning is typically associated with melt-grown synthetics, particularly flame-fusion products such as synthetic corundum and synthetic spinel, an unusual natural emerald with this feature was recently examined in the Carlsbad and Bangkok laboratories. This 1.17 ct emerald was obtained for GIA's permanent reference collection (sample number 100305160993) at a street market in Kabul, Afghanistan, for its very unusual inclusion scene. The stone was represented as being of Afghan (Panjsher) origin, but the internal inclusions seemed to resemble those of emeralds collected from the Swat Valley of Pakistan. Careful chemical analysis by laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS) was performed at the GIA lab in Bangkok. The chemical data showed a fairly even composition within the sample except for chromium, which varied proportionately with the green color zoning (from approximately 300 to 9000 ppmw Cr). The chemical data of the unusual emerald were compared to those of reference standards from known deposits, particularly Panjsher and the Swat Valley, to investigate its probable origin. The data showed a close match only with the reference emeralds from the Swat Valley, making that the most likely origin.

The gemological properties showed an RI of 1.589–1.597 and a visible spectrum consistent with emerald. The stone showed no reaction to a standard 4-watt gemological UV light source. Microscopic examination revealed numerous inclusions of pyrite, carbonates, and reflective thin films oriented perpendicular to the optic axis (parallel to the length of the stone), a typical assemblage of a natural emerald. The most unusual observation about this stone was a prominent green "S" shape color zone that ran down its length (figure 13). Emerald's hexagonal structure would ordinarily lead one to expect planar or angular color zoning related to the crystal morphology. The presence of curving zones in a faceted emerald was an anomaly that needed further examination.

One possible explanation for the

Figure 13. This 1.17 ct emerald, which hosted pyrite and carbonate crystals, displayed unusual curved zoning.

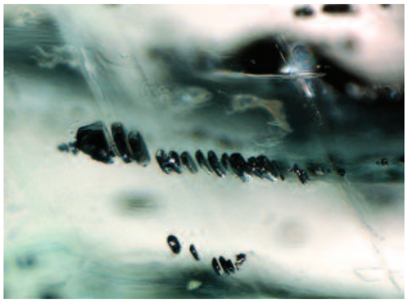


unusual zoning is that compressional geological forces caused shearing along the emerald crystal's basal plane. This was supported by the appearance of pyrite grains, which looked as if single, brittle pyrite crystals had been sheared and slightly displaced parallel to the basal plane of the beryl host (figure 14). This apparent lateral shearing movement throughout the emerald crystal could have caused the pyrite grains to separate into aligned tabular fragments offset from each other and the beryl host. Also observed between the pyrite grains were planar optical irregularities. These planes suggested localized shear zones that would have significantly higher defect concentration, giving rise to the optical nonconformity consistent with their location between the tabular pyrite inclusions. Under cross-polarized illumination, we observed dense clusters of birefringent crystals that were essentially invisible under non-polarized lighting. The low relief of these dense inclusions suggested they were beryl inclusions within the beryl host, possibly a result of partial recrystallization caused by dynamic environmental conditions.

Gemology is generally a non-destructive, observational science, which can be limiting at times. Destructive techniques could have revealed the crystallographic orientation of the pyrite grains with respect to each other and explained the stone's unusual structure. Because this was a unique sample, destructive testing was not a practical approach. While the exact cause of this unusual zoning remains unknown, it is certainly interesting and thought provoking.The discovery of such gems demonstrates that there are complex geological conditions we do not yet understand which, when observed, invite further gemological exploration.

Nathan Renfro, Vincent Pardieu, and Supharart Sangsawong

Figure 14. Arrays of tabular offset pyrite fragments were seen throughout this unusual emerald. Field of view: 0.86 mm.



Rare Faceted NEPTUNITE

An 11.78 ct faceted "black" octagonal modified step-cut stone was recently presented to the Carlsbad laboratory for identification (figure 15). Standard gemological testing revealed an RI of 1.69–1.73 and a hydrostatic SG of 3.19. Microscopic examination with a fiber-optic light source showed the stone was actually a very dark orangy red. It was heavily included with fractures and platelets, as well as randomly oriented unidentified thin needles evenly distributed throughout the gem (figure 16). These tests suggested neptunite, an identity confirmed using Raman spectroscopy.

Neptunite commonly occurs in association with, and as a guest inclusion in, the rare blue mineral benitoite (E.J. Gübelin and J.I. Koivula, *Photoatlas of Inclusions in Gemstones,* Vol. 1, ABC Edition, Zurich, 1986). Both minerals are Ti-bearing silicates. Benitoite may contain whitish inclusions of crossite (an amphibole) or natrolite (a zeolite), so it is possible that the thin needles in this neptunite are composed of one of these metamorphic minerals. The main source of neptunite is San Benito, California, where it is found along the walls of natrolite veins in blueschist (B. Laurs et al., "Benitoite from the New Idria District, San Benito County, California," Fall 1997 *G&G*, pp. 166–187). The type locality for this material is the Narsarsuk pegmatite in Greenland (O.V. Petersen and O. Johnsen, "Mineral species first described from Greenland," *Canadian Mineralogist* Special Publication No. 8, 2005, pp. 76–77). Neptunite crystals have been found in sizes up to a few inches, but they are rarely faceted because of their brittle nature and relative softness (5–6 on the Mohs scale). The few crystals that are faceted usually weigh less than 1 ct, making this large specimen of particular interest. To the best of our knowledge, this is the first faceted neptunite identified by GIA.

Amy Cooper and Tara Allen

Figure 15. This unusual 11.78 ct faceted neptunite appeared to be black but was actually a dark orangy red.

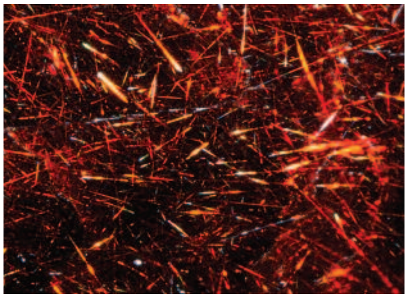


Figure 16. The orangy red neptunite contained dense needles of unknown composition. Field of view: 1.72 mm.

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