GIA GEM TRADE LAB NOTES

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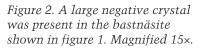
GIA Gem Trade Laboratory, East Coast

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GIA Gem Trade Laboratory, West Coast

BASTNÄSITE, A Rare Faceted Example

We seldom see rare-earth carbonates fashioned as gems, for several reasons: These minerals typically do not occur in large transparent crystals, they are usually brown, and they have low hardness. Recently, staff members in the West Coast laboratory examined an unusual 19.64 ct orangy brown oval modified brilliant (figure 1), which the client represented as the rare-earth carbonate bastnäsite. The following gemological properties were determined by senior staff gemologist Dino DeGhionno and staff gemologist Phil Owens: diaphaneity—transparent; refractive indices-1.722 and greater than 1.81 (over the limits of our standard refractometer); pleochroism—light and dark orangy brown; optic character—uniaxial positive; specific gravity (measured hydrostatically)-5.16; inert to both long- and short-wave UV radiation. These properties were consistent with those



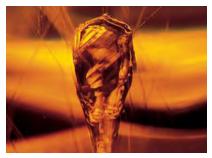




Figure 1. This 19.64 ct bastnäsite $(15.79 \times 11.38 \times 10.66 \text{ mm})$ is the first faceted example of this mineral that has been seen in the Gem Trade Laboratory.

reported for bastnäsite by W. L. Roberts et al. (*Encyclopedia of Minerals*, 2nd ed., Van Nostrand Reinhold, New York, 1990, pp. 73–74).

The handheld spectroscope revealed a "rare earth" spectrum: general absorption below 490 nm, and

Figure 3. The bastnäsite also contained numerous needle-like inclusions. Note the optical doubling of most of the inclusions. Magnified 40×.



bands at 500–525, 530, 560–585, 610, 660, and 690 nm. With magnification, we could see negative crystals (e.g., figure 2), "needles" (figure 3), and a partially healed fracture containing fluid inclusions (figure 4). As bastnäsite is quite soft (Roberts et al. list a hardness of 4 to 4¹/2), we would not expect it to be in common use as a gem material.

According to Roberts et al., the formula for bastnäsite is (Ce,La)(CO₃)F; energy-dispersive X-ray fluorescence (EDXRF) analysis revealed rare-earth elements (lanthanum, cerium, and neodymium) and yttrium, but we were not able to measure these quantitatively with our system. Raman spectroscopy revealed peaks at 164, 258, 304, 351, 394, 733, 1095 (strong), 1437, and 1738 cm⁻¹, with a good match to the bastnäsite spectrum in our reference library. An X-ray powder diffraction pattern matched the spectrum in the International Centre

Figure 4. This partially healed fracture, or "feather," in the bastnäsite showed interesting geometric patterns. Magnified 25×.



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Figure 5. This dense tangle of natural etch channels was typical of the numerous etch features observed in a 25.02 ct brownish yellow round brilliant diamond. Magnified 17×.



Figure 6. In this view of the 25.02 ct diamond, several etch channels have converged to form a rough-surfaced, undulating "cavern." Magnified 20×.



Figure 7. Note the width variations within this single etch channel, which abruptly narrows and turns back on itself, looping in tight curves. Magnified 33×.

for Diffraction Data (formerly JCPDS) reference library. The infrared spectrum included an absorption edge at about 3000 cm⁻¹ and peaks at 5828, 5070, 4660, 4464, 4292, about 4000 (strong), 3758, 3590, 3421, 3229, 3179, and 3154 cm⁻¹.

As bastnäsite is an uncommon gem material, it is difficult to say whether it might have been identified without the advanced testing methods. In this case, as all the evidence pointed to bastnäsite for this stone, we were ultimately confident of the identification.

When it was time to write up the conclusion, we had some slight problems concerning what to put on the report. In Fleischer's Glossary of Mineral Species 1999 (J. A. Mandarino, Mineralogical Record Inc., Tucson, AZ), this material is identified as "bastnäsite-(Ce)." (There are also lanthanum and yttrium end members.) However, because we were not able to obtain a quantitative chemical analysis, we could not confirm that a predominance of the rareearth sites were occupied by cerium (as the -Ce notation implies), so we left off this suffix. Also, because our report printing process does not accommodate umlauts, we substitut-

Editor's note: The initials at the end of each item identify the editor(s) or contributing editor(s) who provided that item.

Gems & Gemology, Vol. 35, No. 2, pp. 136-141 ©1999 Gemological Institute of America ed the archaic "ae" for "ä." Therefore, our report describes this unusual gem as "bastnaesite." *MLI*

DIAMOND, with Abundant Etch Channels

This past spring, staff members at the East Coast laboratory had the opportunity to examine a 25.02 ct brownish yellow round brilliant diamond with a multitude of etch tubes. Figure 5 shows an example of these densely intertwined channels in one area of the girdle. We have previously reported on etch tubes and channels (see, e.g., Gem Trade Lab Notes, Fall 1992, pp. 193–194; Winter 1992, pp. 262–263; Summer 1994, p. 115), but this stone showed much more internal etching than any of us had ever seen in a gem diamond.

Etching—whether it forms tubes, channels, grooves, pits, or other structures—occurs along crystal defects in the diamond (Yu. L. Orlov, The Mineralogy of the Diamond, John Wiley & Sons, 1973, pp. 82, 98-99). Like other naturally colored diamonds of similar hue, this round brilliant owes its color-and its distinctive gemological properties—to a variety of crystal defects. We saw medium-to-strong lines in the desk-model spectroscope at 415 nm, 494 nm, and 503 nm. The diamond fluoresced with medium strength in a mixture of blue and yellow colors to both long- and shortwave UV radiation, and it showed a

mixture of blue and green fluorescence to strong visible light ("transmission" luminescence). These properties indicate two optical centers in this diamond—the N3 and H3—which are composed of three nitrogen atoms, and two nitrogen atoms with a neutral vacancy, respectively (A. T. Collins, "Colour centres in diamond," *Journal of Gemmology*, Vol. 18, No. 1, 1982, pp. 37–75).

The diamond also showed medium-to-strong phantom graining, with pale brown color along two sets of graining planes; such colored graining is believed to form from dislocations between octahedral planes in the diamond (again, see the A. T. Collins reference given above). However, all colored diamonds contain some crystal defects, and many show defect combinations similar to those observed in this diamond, but relatively few show any etch tubes. The presence of such extensive etching suggests that the original crystal (1) was full of dislocations and other weaknesses, and (2) had an unusually prolonged exposure to the caustic geologic fluids that can chemically oxidize diamond.

The etch features in this diamond formed in a number of interesting shapes (see, e.g., figures 5 and 6), as well as in a range of sizes (see, e.g., figure 7). They serve as a reminder of the many differences between laser drill holes and natural etch tubes or channels (see, e.g., M. L. Johnson et al., "When a drill hole isn't," *Rapaport*

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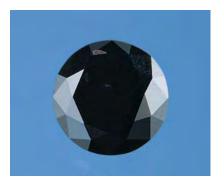


Figure 8. When this 0.29 ct black round brilliant was submitted for identification of its color origin, it was found to be a synthetic diamond.

Diamond Report, Vol. 21, No. 45, December 1998, p. 1). Although the individual tubes in this diamond were rather small in diameter, they showed angular outlines, rather than the round or oval outlines formed by laser drilling. Drill holes are usually straight, but etch tubes rarely are. Most tellingly, drill holes tend to lead to some inclusion, such as a void from which a dark crystal was removed, or a feather, which may be fracture filled. In contrast, etch tubes follow atomsize crystal defects, and thus appear to meander randomly through the diamond.

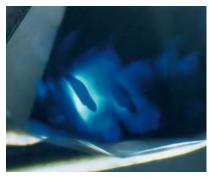


Figure 9. Magnification and fiberoptic lighting revealed both a dark blue color and a rod-shaped inclusion (typical of a metallic flux) in the synthetic diamond. Magnified 40×.

SYNTHETIC DIAMOND

Black

As the use of black diamonds in jewelry has increased, we have seen more of them at both laboratories for color-origin determination. In most black diamonds, the overall color appearance is caused by a large number of dark inclusions. We routinely examine black diamonds near the girdle with pinpoint fiber-optic illumination to look for areas of partial transparency (which are typical of natural color) or for a dark green color (which is an indicator of treatment). Such "greenish"

black color results from a high dose of radiation, usually in a nuclear reactor, and diamonds treated by this method may be radioactive (see, e.g., Summer 1992 Lab Notes, pp. 124–125).

We were, therefore, somewhat surprised when the strong light revealed a blue color in a 0.29 ct "black" round brilliant (figure 8) that was examined last winter in the East Coast lab. The strong electrical conductivity of the sample (100:135 to 120:135) indicated that the blue color was caused by boron impurities (see J. M. King et al., "Characterizing natural-color type IIb blue diamonds," Gems & Gemology, Vol. 34, No. 4, 1998, pp. 246-268). The piece was inert to both long- and short-wave UV radiation. It also was heavily included, with only a few relatively transparent areas. One of those areas contained a dark, rod-shaped inclusion (figure 9), which is a common morphology for flux inclusions in synthetic diamond. This prompted us to test the sample for magnetic attraction by suspending it from a thread and bringing a powerful magnet near it. The sample's strong attraction to the magnet provided another indication that it was synthetic.

To confirm this identification and further characterize this unusual specimen, we examined the sample with a De Beers DiamondViewTM (see, e.g., C. M. Welbourn et al., "De Beers natural versus synthetic diamond verification instruments," Gems & Gemology, Vol. 32, No. 3, 1996, pp. 156-169). We hoped that the greater intensity of this source would stimulate visible fluorescence. Although the reaction was weak and severely interrupted by the many inclusions (which did not fluoresce), we observed a fluorescence pattern that was hexagonal (i.e., reflecting the faces of an octahedron modified by a cube; cubic growth is characteristic of synthetic diamonds), rather than the concentric rectangular pattern typical of natural diamonds. The sample luminesced more strongly to X-rays, revealing a blue, angular pattern typical of synthetic growth.

Figure 10. These two synthetic diamonds (0.59 and 0.63 ct) illustrate the range of color, from orangy yellow to greenish yellow, that was displayed by a group of 18 synthetic diamonds submitted to the laboratory at one time.



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Group of 18 Synthetic Diamonds Submitted at One Time

A regular client submitted a group of 18 round-brilliant and square-modified-brilliant "diamonds" to the East Coast lab for identification and colororigin determination. They ranged from 0.10 to 0.71 ct and exhibited saturated colors that varied from orangy yellow to greenish yellow, as represented by the two samples in figure 10. Standard gemological testing quickly revealed, however, that all 18 samples were synthetic diamonds. All were purchased in Russia, where they were represented as natural, so the client was surprised to hear the results.

Six of the samples contained inclusions that were larger than pinpoints, in the rod- and droplet-shaped morphologies that are typically seen in synthetic diamond. These six also showed obvious attraction to a strong magnet, while the other samples had a very weak or no attraction. We performed EDXRF analyses on two samples, one with large inclusions and one without; both revealed Fe and Ni, the principal elements in the flux that is used to facilitate synthetic diamond growth. All 18 samples exhibited some unevenness of body color, but many showed weak color zoning in which the pattern was difficult to discern. However, their reaction to UV radiation was vividly diagnostic: All showed a yellowish green cross pattern. This pattern was most clearly seen through the crown, but it was strong enough in some samples to be evident through the pavilion as well. Although some natural-color and especially treated-color diamonds fluoresce vellowish green, the cross pattern is diagnostic of synthetic origin (see, e.g., J. E. Shigley et al., "A chart for the separation of natural and synthetic diamonds," Gems & Gemology, Vol. 31, No. 4, 1995, pp. 256-264).

The fluorescence ranged from moderate to strong for long-wave UV radiation and from weak to strong for short-wave UV. This general trend of a stronger reaction to long-wave than short-wave UV is unlike that seen in



Figure 11. The strong pleochroism of this cobalt-doped synthetic forsterite makes it a convincing tanzanite imitation. The largest cushion mixed cut weighs 6.15 ct and measures $12.29 \times 9.85 \times 7.06$ mm. Courtesy of Tom Chatham.

most of the earlier yellow synthetics GIA described (J. E. Shigley et al., "The gemological properties of Russian gem-quality synthetic yellow diamonds," Gems & Gemology, Vol. 24, No. 4, 1993, pp. 228-248). It suggests that some of the nitrogen impurities were aggregated at high temperature after growth, forming a type Ia component (A. T. Collins and M. Stanley, "Absorption and luminescence studies of synthetic diamond in which the nitrogen has been aggregated," Journal of Physics D: Applied Physics, Vol. 18, 1985, pp. 2537–2545.). Unfortunately, these synthetic diamonds were not available long enough for us to perform the infrared spectroscopy necessary to confirm the diamond type.

SYNTHETIC FORSTERITE, A New Tanzanite Imitation

At the April American Gem Society Conclave in New Orleans, Tom Chatham of Chatham Created Gemstones, San Francisco, California, showed contributing editor MLJ four fashioned samples and two rough pieces (figure 11) of a material grown in Russia that had been represented to him as synthetic tanzanite. As this was clearly a "pulled" product, and tanzanite is a variety of the hydrous mineral zoisite, we were skeptical of this claim and agreed to study the material further.

The four fashioned samples included a 6.15 ct cushion mixed cut, a 3.27 ct oval modified brilliant, and two (2.81 and 2.10 ct) modified triangular mixed cuts. The two pieces of rough weighed 5.75 ct and 3.50 ct; portions of these samples showed a smooth curved surface, resembling part of a boule. The fashioned samples had the following properties: color-violet; optic character-biaxial positive; pleochroism—strong, in blue and purplish pink (figure 12); (Chelsea) color-filter reaction—none; refractive indices—1.635 to 1.637, 1.650 (beta), and 1.670 to 1.671; birefringence—0.034 to 0.035; specific gravity (measured hydrostatically)-3.23 to 3.24. The samples fluoresced a moderately chalky, but very weak, orangy yellow to long-wave UV radiation, and a weak greenish yellow to short-wave UV; at both wavelengths, the color was evenly distributed. When viewed with a handheld spectroscope, they showed a 460-470 nm

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Figure 12. The two pleochroic colors displayed by the synthetic forsterite, as seen here with polarized light, were blue and purplish pink. Magnified $10\times$.

band, a 490 nm band, diffuse lines at 510 and 520 nm, and a 570–580 nm band. With magnification, we saw indistinct white inclusions, pinpoint inclusions (possibly gas bubbles), tiny "needles," and stringers (figure 13).

On the basis of the refractive indices and strong pleochroism, in particular, we suspected that the material was synthetic forsterite, Mg₂SiO₄. An EDXRF analysis performed by GTL research associate Sam Muhlmeister on the 6.15 ct cushion mixed cut confirmed this identification. EDXRF revealed major amounts of magnesium and silicon, and traces of cobalt, vanadium, and iron, with the cobalt much more prominent than the other trace elements. A Raman spectrum showed strong orientation effects (i.e., spectra

Figure 13. The 6.15 ct synthetic forsterite contained indistinct white inclusions that resemble dust particles, as well as pinpoints, tiny needles, and stringers. Magnified 11×.



taken from different angular positions had large variations in peak height), with the strongest peaks at 857 and 825 cm⁻¹ and others at 965, 919, 607, 591, 547, 435, 307, and 227 (±2) cm⁻¹. An infrared spectrum showed a cutoff at 2000 cm⁻¹, and small peaks at 2497, 2623, and 2985 cm⁻¹; there was no evidence of any significant water present in the sample.

Synthetic forsterite has been described before (see, e.g., K. Nassau, "Synthetic forsterite and synthetic peridot," Gems & Gemology, Vol. 30, No. 2, 1994, pp. 102-108); and in an October 1996 personal communication, Dr. Henry Hänni of the SSEF Swiss Gemmological Institute called our attention to Czochralski-pulled synthetic forsterite that had been grown by Solix in Minsk, Russia. In both of these instances, chromium was the only dopant. Dr. Hänni mentioned colorless, blue-green, and "olive green" samples, and Dr. Nassau reported that there was also pink and purple material, in which the chromium could be in various oxidation states. The material we examined, however, contained no chromium, but rather was colored by cobalt (with a possible contribution from vanadium).

A comprehensive article by L. Kiefert and S. Schmidt ("Some tanzanite imitations," *Gems & Gemology*, Vol. 32, No. 4, 1996, pp. 270–276) listed manufactured glass, YAG, and synthetic corundum as tanzanite simulants. Since then, we have

also seen treated-color blue beryl represented as tanzanite (Winter 1997 Lab Notes, p. 293). The pronounced pleochroism of synthetic forsterite makes it visually far more convincing as a tanzanite simulant than any we have seen before, although it can be easily distinguished from tanzanite by its refractive indices (1.635–1.670 versus 1.69–1.70 for tanzanite).

MLJ and SFM

PEARL,

Update on Non-nacreous Pearls

In the Winter 1998 Lab Notes section (p. 288), we reported on several banded, light pink and white, non-nacreous "pearls" with a sheen effect. Subsequently, the West Coast lab received samples of the shells (figure 14) that had hosted some of these concretions. Although it was previously thought that the host might be an oyster, we could readily see that the appearance was more like a scallop, of the type commonly known in some areas of the North American Pacific Coast as "lion's paw shells." The characteristic physiognomy, with seven coarsely nodular ribs and two uneven "ears," allowed us to identify the genus as Chlamys nodosus.

Figure 14. These shells, measuring 6 cm across, are from Chlamys nodosus, a scallop that has produced non-nacreous "pearls" with color banding and a sheen effect (see inset, 14 × 12 mm).



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SYNTHETIC SAPPHIRE

Orangy Pink "Padparadscha"

A narrow range of pinkish orange to orangy pink sapphires are commonly referred to by the trade term *padparadscha*. Although such sapphires have long been highly prized by collectors, the GIA Gem Trade Laboratory does not issue a report using this trade description because of the subjectivity of the term (see R. Crowningshield, "Padparadscha: What's in a name?" *Gems & Gemology*, Vol. 19, No. 1, 1983, pp. 30–36).

A client recently submitted the approximately 9 ct slightly orangy pink cushion mixed cut shown in figure 15 for identification and color description. The client was disappointed that we could not verify if the stone qualified for the term *padparadscha*, but agreed to an identification report that would give an appropriate color description.

The R.I.'s of 1.76–1.77, uniaxial optic character, and absorption spectrum (three lines in the red due to Cr, and a doublet at 460–470 nm) verified that the stone was indeed corundum. In most cases, we can easily determine whether a fancy-colored sapphire is natural or synthetic by the internal features (e.g., the presence of straight or curved color banding). However, this attractive stone was

Figure 15. This approximately 9 ct orangy pink cushion mixed cut, which might be called "padparadscha" by the trade, was identified as a synthetic sapphire.



virtually flawless except for a tiny feather near the girdle. Immersion in methylene iodide did not reveal any other internal features; specifically, we could not resolve any color zoning, either straight or curved. The sample fluoresced an equally strong red to both long- and short-wave UV radiation, which also provided no indication as to its origin.

However, EDXRF chemical analysis of the stone showed a significant amount of Ni. in addition to Fe and Cr. Nickel is not found in natural sapphire, but it has been reported in some hydrothermally grown synthetic corundum from Russia (see, e.g., V. Thomas et al., "Tairus hydrothermal synthetic sapphires doped with nickel and chromium," Gems & Gemology, Vol. 33, No. 3, 1997, pp. 188-202). Therefore, we concluded that this sample was a synthetic sapphire, although whether it represents Russian hydrothermal growth remains uncertain. KH

Early Blue Synthetic Sapphire?

The white metal ring shown in figure 16 is characteristic of jewelry made in the early 1900s. As we have mentioned previously in this section, although it is possible to replicate virtually any style of jewelry, there are often certain features that suggest that a piece is indeed an "original" and was made at the time the style and design indicate. The ring shown here was one such example: Its overall style and the wear, specifically the "burnishing" of the metal around the stones, suggest that the stones had been set in the ring for some time. (A "burnish" implies that the metal has been rubbed over time; that is, it has a polished glossiness that is also somewhat dark from the incorporation of foreign substances. The effect, like that of seasoning a cast-iron skillet, cannot readily be imitated.) Even the synthetic sapphire in the center is consistent with material available in the early part of this century.

Synthetic blue sapphire was first



Figure 16. This ring appears to have been manufactured in the early part of this century. The Verneuil synthetic sapphire in the center shows eye-visible concentric circles of color banding.

produced by Verneuil in about 1910 (see, e.g., K. Nassau, *Gems Made by Man*, Chilton Book Co., Radnor, PA, 1980). Consequently, the large cabochon in this ring may well date from that time, as its gemological properties were typical for a Verneuil synthetic.

The noteworthy feature of this cabochon was the eye-visible curved growth banding that was present as concentric circles. Normally, synthetic corundum boules grown by the flame-fusion method are split lengthwise in preparation for fashioning. Once the boule is split, the curved growth bands-or striae-are visible only as a group of nested curves, and not as full circles. One possible explanation for the circular growth banding in this cabochon is that it was cut from a small "button" boule that was not split after growth (see, e.g., D. Elwell, Man-Made Gemstones, John Wiley & Sons, New York, 1979).

TM and GRC

PHOTO CREDITS

Vincent Cracco took photos 5, 6, and 7. Maha DeMaggio photographed figures 1, 8, 10, 11, 14, and 15. John King provided figure 16. John Koivula was the photographer for figures 2, 3, and 4. Shane McOlure photographed figures 9, 12, and 13.

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