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COLORED STONES AND ORGANIC MATERIALS

Unusual epigenetic malachite discs in quartz. When mineral-rich fluids become trapped in open cracks and evaporate, they can leave behind epigenetic mineral precipitates. These epigenetic mineral deposits are fairly common in gem materials, usually taking the form of brownish iron oxide staining in cracks. These deposits are often a nuisance, impairing the potential beauty of the finished gem, but occasionally they contribute vibrant colors with interesting geometric shapes, such as inclusions of pyrite trapped in cracks in quartz.

Figure 1. This 52.99 ct freeform cabochon of rock crystal quartz, reportedly from Brazil, contained several epigenetic discs of malachite. Photo by Robison McMurtry.

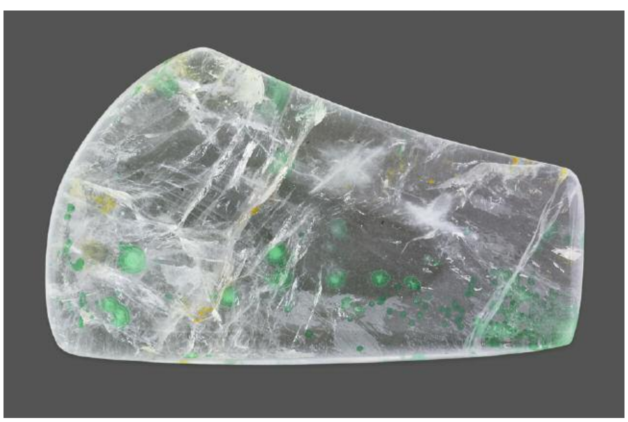

Figure 2. These radial discs of epigenetic malachite were trapped within cracks of rock crystal quartz. Photomicrograph by Nathan Renfro; shadowed illumination, field of view 2.15 mm.

An unusual freeform cabochon of rock crystal quartz (figure 1), obtained from Leonardo Silva Souto (Cosmos Gems, Teófilo Otoni, Brazil) and reportedly of Brazilian origin, was examined at GIA's Carlsbad laboratory. The 52.99 ct stone contained several interesting inclusions of malachite (figure 2). These inclusions were obviously epigenetic, as they were confined within secondary cracks in the quartz host. This planar confinement caused the inclusions to grow outward after nucleating. The flattened discs were particularly interesting due to the vibrant interference colors visible in polarized light (figure 3), as well as their uniform structure. The identification of the inclusions and the host was confirmed by Raman spectroscopy.

Malachite has been previously reported as a syngenetic inclusion in chalcedony and gypsum (e.g., E.J. Gübelin and J.I. Koivula, *Photoatlas of Inclusions in Gemstones*, ABC Edition, Zurich, 1986), but epigenetic malachite in quartz

Editors' note: Interested contributors should send information and illustrations to Justin Hunter at justin.hunter@gia.edu or GIA, The Robert Mouawad Campus, 5345 Armada Drive, Carlsbad, CA 92008.

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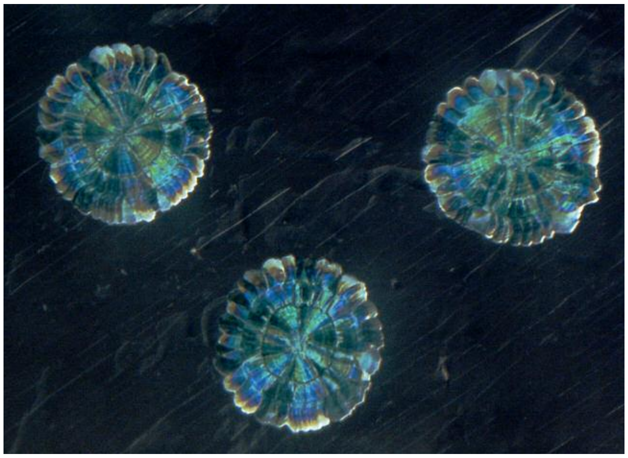

Figure 3. In polarized light, these malachite discs showed vibrant interference colors. Photomicrograph by Nathan Renfro; field of view 2.15 mm.

is rather uncommon. In this stone, the resulting vivid green color is an improvement over the otherwise ordinary, colorless quartz.

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DIAMOND

Unusual dumbbell-like inclusion in diamond. The French Gemmological Laboratory (LFG) in Paris had the opportunity to grade a 0.40 ct near-colorless round brilliant diamond with a large, unusual inclusion (figure 4). Initial inspection under the loupe showed the internal feature consisted of a thin middle "bar" and two broader appendices (figure 5). Viewed from certain angles, it appeared almost metallic. This feature, which lowered the clarity grade to SI_2, was reminiscent of the metallic dumbbell inclusions observed in HPHT synthetic diamonds.

Figure 4. This 0.40 ct round brilliant diamond displayed a large, unusually shaped inclusion. Photo by Aurélien Delaunay.

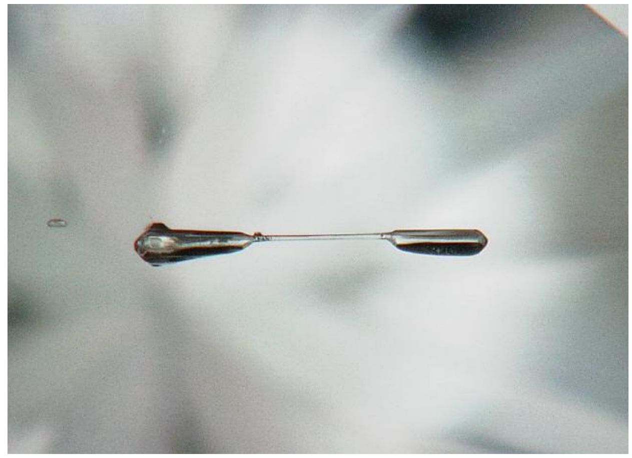
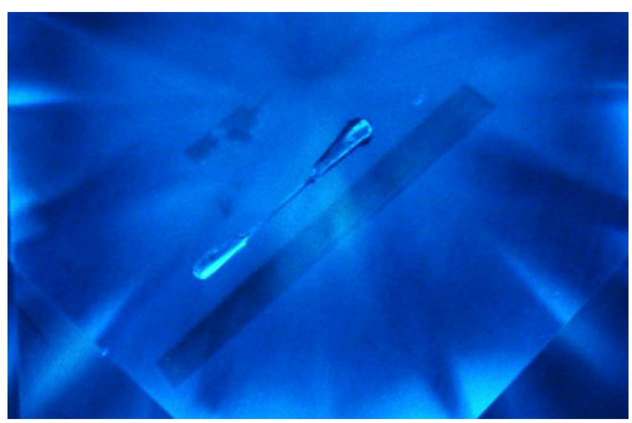


Figure 5. At first inspection, this dumbbell shape was reminiscent of the metallic flux inclusions seen in HPHT synthetic diamond. Photomicrograph by Aurélien Delaunay; magnified 120×.

DiamondView luminescence images demonstrated that the inclusion was crystallographically oriented, parallel to the edge between two octahedral faces (figure 6). Dumbbell inclusions in HPHT synthetics have a clear crystallographic orientation, often along an octahedral edge as well.

But the stone fluoresced weak blue under long-wave UV radiation and was inert in short-wave UV, a classic luminescence behavior for natural diamond and unknown in synthetic specimens. Furthermore, the infrared absorption spectrum was typical of a natural type IaAB diamond with minor hydrogen. This proved the natural origin of both the

Figure 6. DiamondView luminescence images demonstrated the diamond's natural origin, as only traces of octahedral growth were found. The inclusion is crystallographically oriented, parallel to the edge between two octahedral faces, as highlighted by the slightly more inert elongated rectangle beside it. Photomicrograph by Aurélien Delaunay, magnified approximately 60×.



stone and its inclusion. With magnification, the inclusion appeared colorless and transparent rather than metallic and opaque, though with a high optical relief. Its morphology was not cuboctahedral—as with inclusions in synthetic diamond—but rounded octahedral. The exact nature of the crystal remains unknown, as we could not obtain a Raman spectrum due to its intense luminescence, but colorless inclusions in diamonds are often forsterite.

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SYNTHETICS AND SIMULANTS

Boron carbide: A new imitation of black diamond. As black diamond has gained popularity in the past few years, so have aggregates of black synthetic moissanite and crystalline silicon (Spring 2011 Lab Notes, pp. 54–55). One specimen received by the Gem Testing Laboratory in Jaipur, a 1.04 ct black submetallic round brilliant (figure 7), appeared to be synthetic black moissanite but proved otherwise. The specimen was presented to the owner as a diamond.

Due to the prevalence of black synthetic moissanite in the marketplace and gem laboratories, we immediately checked for the typical aggregate structure under the microscope. Although a granular pattern was distinctly visible in reflected light (figure 8), it was much denser and finer than the kind usually seen in black synthetic moissanite. This raised uncertainty, so additional gemological tests were performed. The specimen had an over-the-limit RI, an SG of 2.43, and a hardness above 9 on the Mohs scale. The low SG value ruled out the possibility of synthetic moissanite (3.22), but otherwise offered no clues as to the specimen's identity.

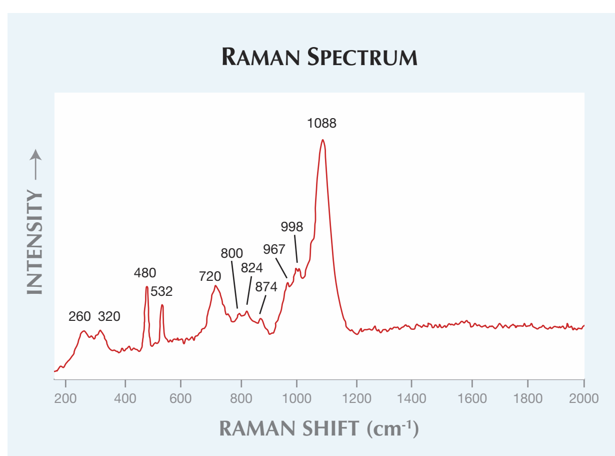
Qualitative EDXRF analysis revealed Fe, with traces of Si and K. Raman spectra taken from several points using 532 nm laser excitation showed major peaks at approximately 260, 320, 480, 532, 720, and 1088 cm^{-1}, with smaller peaks at about 800, 824, 874, 967, and 998 cm^{-1} (figure 9). This combination of features did not match anything in our database, but an extensive literature search revealed an exact match with the Raman spectra of boron carbide (V. Domnich et al., "Boron carbide: Structure, properties, and stability under stress," *Journal of the American Ceramic Society*, Vol. 94, No. 11, 2011, pp. 3605–3628, http://dx.doi.org/10.1111/j.1551-2916.2011.04865.x).

Figure 8. Under reflected light, the boron carbide specimen displayed a dense, fine granular structure typically associated with aggregates such as ceramics. Photomicrograph by Gagan Choudhary; magnified 48×.

Boron carbide is an advanced ceramic material with high hardness, low density, thermal stability, and extreme abrasion resistance, making it suitable for nuclear, military, and aerospace applications. The material is typically produced by reacting and fusing carbon with boric oxide (B_2O_3) in an electric arc furnace, followed by a sintering

Figure 7. This 1.04 ct black submetallic round brilliant, initially represented as diamond, was identified as boron carbide. Photo by Gagan Choudhary.

Figure 9. The boron carbide's Raman spectrum showed major peaks at approximately 260, 320, 480, 532, 720, and 1088 cm^{-1}.



process of hot pressing in graphite dies under a vacuum or argon atmosphere at temperatures of 1900–2200°C and pressures of 0.02–0.04 GPa for 15–45 minutes (Singhal and Singh, "Sintering of boron carbide under high pressures and temperatures," *Indian Journal of Engineering and Materials Sciences*, Vol. 13, April 2006, pp. 129–134).

Although boron carbide is common in engineering and materials science—used as scratch-resistant coating and plating in tank armor, bulletproof vests, and padlocks—this was our first encounter with it as a diamond simulant. Our literature search (by Mr. Sandeep Vijay, a staff gemologist) did not reveal any gemological uses of this ceramic product. Distinguishing boron carbide from black diamond or synthetic moissanite was straightforward on the basis of SG, but Raman spectra and reference data were needed for identification. The possibility of market penetration cannot be ruled out.

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Amber with insect-bearing filling. We recently tested an interesting strand of Islamic prayer beads resembling amber (figure 10). It consisted of 33 round yellow beads approximately 12 mm in diameter, two oval-shaped separators, and a fancy-shaped link. It was purchased as amber from a dealer at an annual festival in Dubai.

The round beads contained dark brown plant debris and a variety of insects: ants, flies, and spiders, all in good condition. The beads' surfaces also featured noticeable circular zones, and when exposed to long-wave UV radiation, they displayed a weak, blue fluorescence and distinctive chalky blue circular areas (figure 11). The beads were inert to short-wave UV radiation.

These features were similar to those of copal filled with plastic (Winter 2010 GNI, pp. 326–328). We obtained spot RI readings of 1.52 on the circular zones and 1.54 elsewhere. Next, we analyzed the strand using Raman spectroscopy with a 785 nm diode laser. The separator link and the main portion of the beads showed peaks between 1434 and 1644 cm^{-1}, consistent with amber. The circular areas' strong bands at 623, 807, 1100, and 1599 cm^{-1} were consistent with epoxy resin (figure 12).

Microscopic examination with immersion in water indicated that all the round beads had been cored and filled (figure 13). The beads were identified as amber filled with colorless to light yellow plastic to imitate insect-bearing amber.

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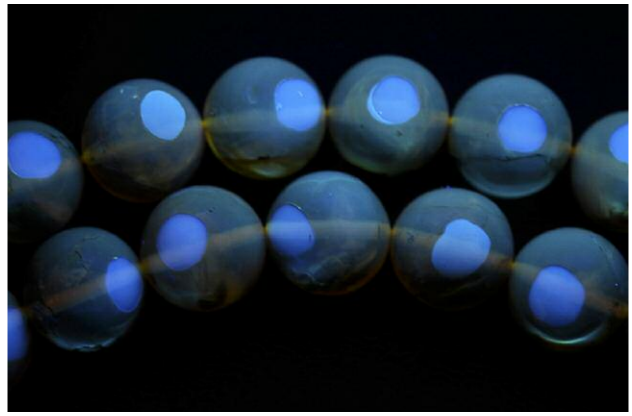
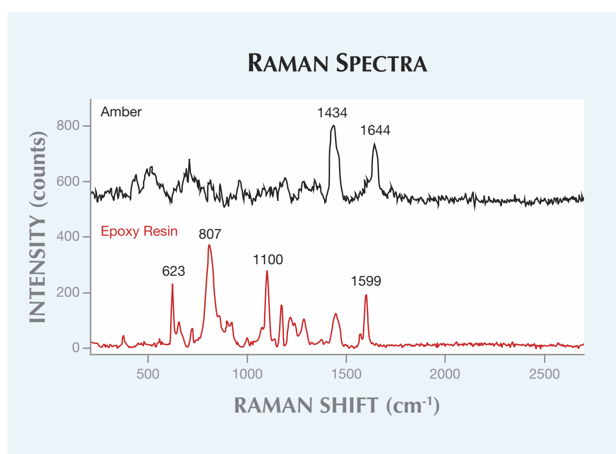


Figure 11. When exposed to long-wave UV radiation, these beads displayed weak blue fluorescence and distinct, chalky blue circular areas. Photo by Sutas Singbamroong.

Figure 10. The beads in this strand of prayer beads measure approximately 12 mm in diameter. Photo by Sutas Singbamroong.

Figure 12. Raman spectra collected from the main portion of the beads were consistent with amber, whereas the circular areas indicated an epoxy resin.



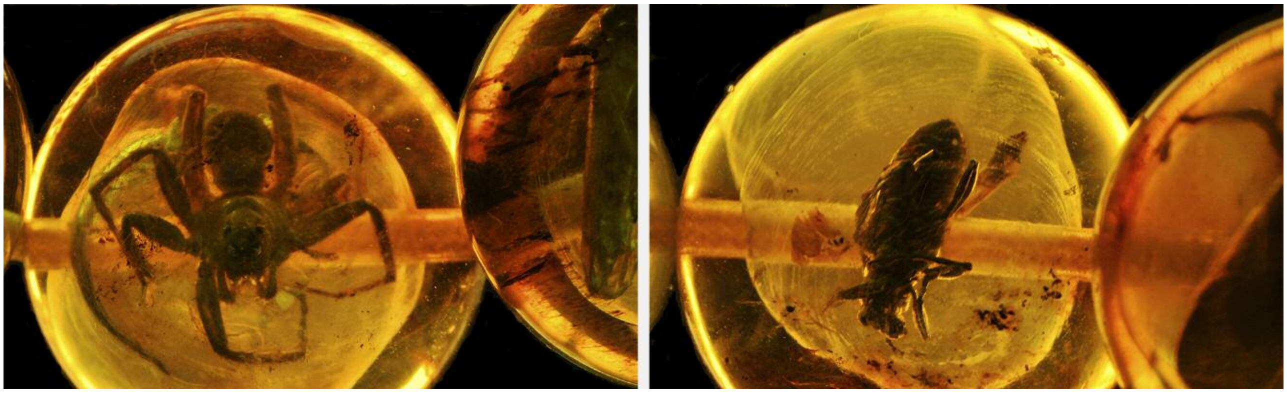


Figure 13. Microscopic observation with immersion in water clearly indicated that these amber beads had been cored and filled with a colorless to light yellow plastic containing insects. Photomicrographs by Sutas Singbamroong; magnified approximately 10×.

Phosphorescence in synthetic sapphire. A 4.89 ct colorless oval mixed-cut specimen was submitted as a sapphire to the Gem Testing Laboratory at the Indian Gemological Institute in Delhi for identification (figure 14). Standard gemological examination gave an RI of 1.760–1.768, with a uniaxial negative optic sign and a birefringence of 0.008, as well as a hydrostatic SG of 4.00. These properties were consistent with sapphire. While this specimen was eventually proven to be synthetic, its unusual characteristics—a lack of curved growth banding visible in the DTC DiamondView and very rare phosphorescence—called for instrumentation generally used in diamond identification.

Magnification showed what appeared to be long needles near the girdle, but closer examination revealed they were long cracks (figure 15). Rather than the Plato lines generally found in light-colored flame-fusion synthetic sapphires, the sample displayed two sharp planes under crossed polarizers. These planes, which had a noticeable separation in between, were visible in twinning lamellae position. Small stress knots near the table surface—but no physical inclusions—were also visible under crossed polarizers (figure 16). Such cracks, which are generally found on the surface of synthetic sapphire, may be due to stress during crystal growth.

The sample was inert to long-wave UV radiation but showed a chalky blue fluorescence in short-wave UV, a common feature of synthetic sapphires. It also produced 30 seconds of phosphorescence—the first time this contributor has observed phosphorescence in sapphire, natural or synthetic. Examination in the DiamondView also showed an uncommon blue phosphorescence (figure 17), but it did not show curved growth banding—a fluorescence pattern normally seen in synthetic sapphire grown by the flame-fusion process.

Identification could only be made on the basis of short-wave UV transmission analysis using the SSEF Diamond Spotter. When the instrument was switched on, short-wave UV radiation transmitted through the sample and the green dot glowed. The specimen's transparency under the Diamond Spotter's short-wave emission at 254 nm proved its synthetic origin, as natural sapphire does not transmit short-wave UV below 288 nm (S. Elen and E. Fritsch, "The

Figure 14. This 4.89 ct colorless synthetic sapphire was presented as a sapphire. Photo by Meenakshi Chauhan.

Figure 15. These fine, linear cracks near the girdle resemble long needles. Photomicrograph by Meenakshi Chauhan; magnified 40×.

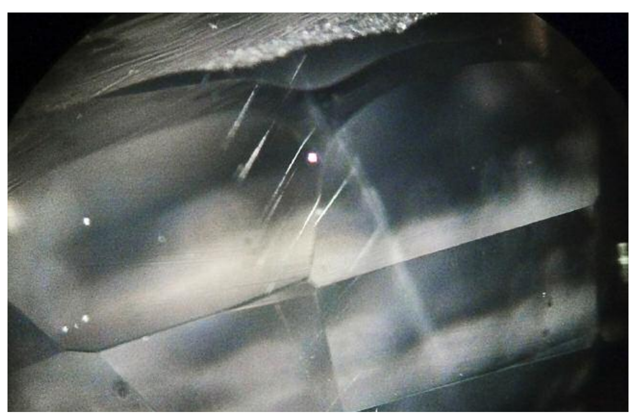


Figure 16. These small stress points, visible on the table between crossed polarizers, were confined to a small space. Photomicrograph by Meenakshi Chauhan; magnified 30×.

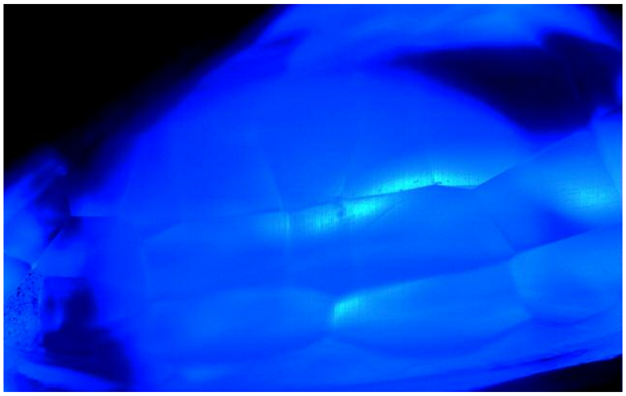


Figure 17. The colorless sample showed blue phosphorescence in the DiamondView, evidence of its synthetic origin. Image by Meenakshi Chauhan.

separation of natural from synthetic colorless sapphire," Spring 1999 *G&G*, pp. 30–41, http://dx.doi.org/10.5741/GEMS.35.1.30).

Despite the confirmation of synthetic origin, no conclusion could be made regarding the synthesis process used. Consistent with specimens grown by the Czochralski pulling method, there were no curved color bands, inclusions, or Plato lines. Conversely, the specimen's chalky blue fluorescence and twinning are indicative of flame-fusion growth. These findings present the possibility of either (1) a synthesis process not yet explored by the gemological community or (2) growth by a known process, but with near perfection. While the DiamondView and Diamond Spotter were developed to analyze diamonds, they can also play a vital role in identifying other gemstones.

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Unusual faceted massive fuchsite. French gem dealer Patrick de Koenigswarter (MinerK) recently showed us some interesting material he purchased in 2011 from an Ethiopian rough dealer. At the time of purchase, the green rough (figure 18) was presented to him as emerald from a new find near the Kenticha pegmatite zone in Ethiopia, well known for tantalite and superb amazonite crystals. However, beryl was ruled out due to the material's waxy luster and low hardness of 3 or less on the Mohs scale—it was easily scratched with a copper coin.

Routine gemological testing was done on the four samples, which consisted of three rough pieces (17–138 ct) and a 2.56 ct emerald cut. The faceted stone (figure 18, bottom right) always remained lit under crossed polarizers, indicating a polycrystalline structure. The color was unevenly distributed, from light green to intense green, sometimes close to an emerald color. The refractive index, though difficult to measure, was around 1.60. The specific gravity values were 2.80 for two of the pieces and 2.90 for the others, a difference possibly due to the presence of mineral inclusions. All four pieces were inert under both long- and short-wave UV, and red to pink under a Chelsea filter. Additional analyses were required to properly identify this material.

Qualitative and quantitative analyses were performed with a JEOL-5800LV scanning electron microscope (SEM) equipped with a PGT (Princeton Gamma Tech) energy dispersive IMIX-PTS detector. The following elements were found (in atomic %): Si = 16.1%, Al = 17.0%, K = 4.0%, and Na = 1.3%; O was calculated as 61.0%, plus traces of Ba (0.3%), Fe, and Cr. These last two elements were clearly detected on the spectrum but in concentrations too low to be measured (below 0.1%). This composition is consistent with muscovite, a member of the mica family. We ob-

Figure 18. This massive Cr-muscovite, reportedly from southern Ethiopia, is better known in the trade as fuchsite. The longest rough piece measures 53 mm, and the faceted stone weighs 2.56 ct. The material was difficult to polish because of its low hardness, 3 or less on the Mohs scale. Photo by Thierry Pradat.

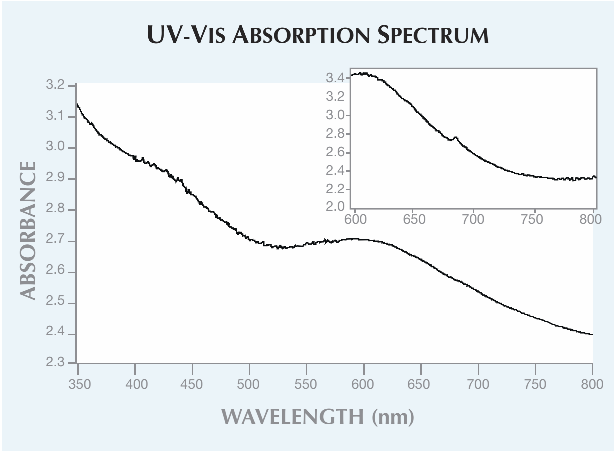

Figure 19. The UV-visible spectrum of the Ethiopian fuchsite shows a large absorption band around 600 nm, a continuum of increasing absorption toward the UV, and a weak, sharp peak at 682 nm. These features are consistent with green color produced by trivalent chromium.

Figure 20. This 444 g dark violetish blue carving of Lord Mahaveera, submitted as sapphire, was identified as corundum coated with a blue polymer. Photo by Gagan Choudhary.

served platy brown polyhedral inclusions, identified by EDS and Raman as rutile due to telltale broad bands around 611 and 443 cm^{-1}.

We also performed UV-visible absorption spectrometry using a 1 nm sampling and spectral bandwidth in the 350–800 nm range. The spectrum showed a large band centered at about 600 nm and a continuum of increasing absorption toward the UV region (figure 19). These features generated two transmission windows, one in the green around 530 nm and the other in the red to infrared. This explains both the green bodycolor and the red appearance in the Chelsea filter. The continuum is caused by the scattering of light at grain boundaries in this polycrystalline aggregate. We also observed a weak but sharp peak at 682 nm, which we attributed to "chromium lines" of the trivalent chromium (figure 19, inset).

Chemical and spectroscopic results identified Cr^{3+} as the main cause of the green color. Hence, this material was fuchsite, the chromian variety of muscovite. To the best of our knowledge, this is the first time such a massive, translucent polycrystalline fuchsite has been faceted. The gem's low hardness makes it unsuitable for jewelry, but it is adequate for collectors.

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Benjamin Rondeau
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TREATMENTS

Corundum carving coated with colored polymer. Coating, one of the traditional forms of enhancement, is still widely used on a number of gem materials. This contributor alone has reported on recent examples (e.g., Spring 2011 GNI, pp. 71–72; Summer 2012 GNI, pp. 154–155). The latest is a dark violetish blue carving of the early Jain spiritual leader Lord Mahaveera, submitted as sapphire (figure 20), that turned out

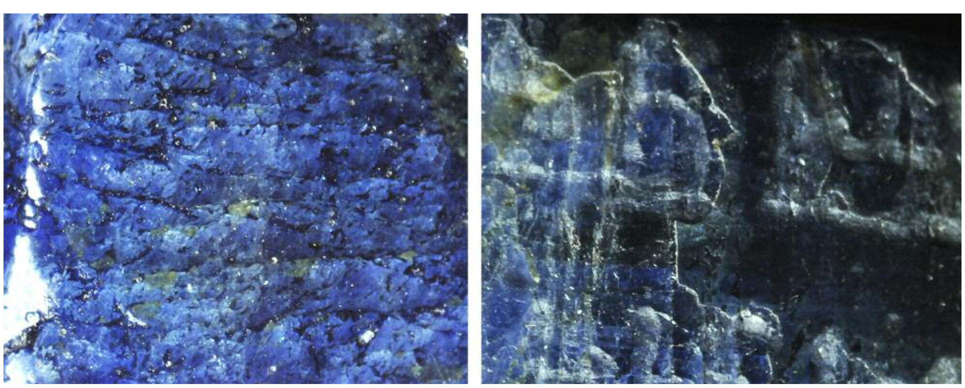

Figure 21. Color concentrations within cavities, large fractures, and twinning planes (left) confirmed the presence of a dye. The chipped layer of blue polymer (right) indicates a coating; this polymer layer was easily indented by a metal pin. Photomicrographs by Gagan Choudhary; magnified approximately 20×.

Figure 22. These fire opals, a 14.49 ct cabochon (left) and a 1.09 ct cushion cut (right), are shown prior to immersion in plain water at room temperature. Note the natural-looking color. Photos by Larry Tai-An Lai.

to be corundum coated with a blue colored polymer.

The 444 g opaque carving measured 10.00 × 8.00 × 4.50 cm. Although it had the heft of sapphire, its dull, vitreous to waxy luster (again, see figure 20) raised doubts. Examination from different directions using a fiber-optic light revealed substantial color variation in different parts of the carving; some areas appeared pale blue, others dark violetish blue. This patchy, concentrated color variation is associated with dyes. Parallel whitish crisscross lines were also observed, consistent with the twinning planes associated with corundum. Further microscopic observation revealed color concentrations within cavities, large fractures, and twinning planes, confirming the material had been dyed (figure 21, left). Finer fractures appeared whitish, however. When the carving was rotated, some areas also displayed a chipped bluish layer on the surface (figure 21, right), an attribute typically found in coated materials. This layer was easily indented with a metal pin, suggesting the presence of a polymer. In addition to these features, the carving possessed a granular texture in many places, as commonly seen in rocks. These grains were also identified as corundum on the basis of the crisscross pattern of their twinning planes, which is consistent with corundum.

Under UV light (long- and short-wave), the carving displayed patchy white and chalky green fluorescence, mainly on the surface. A desk-model spectroscope revealed weak absorptions at 540, 610, and 640 nm, confirmed with UV-Vis-NIR spectroscopy. Such bands are associated with cobalt-based dyes. RI and SG could not be measured because of the piece's large size, though visual features were sufficient to identify the carving as corundum.

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Water immersion revealing dye in fire opal. Opal is often treated to enhance its play-of-color or improve its durability. Dye or impregnation methods in particular are easily applied to opals and other porous amorphous materials. A recent article on treated hydrophane opal (N. Renfro and S. McClure, "Dyed purple hydrophane opal," Winter 2011 *G&G*, pp. 260–270) noted that we could expect to see more of this material. Two years later, this appears to be the case.

The Lai Tai-An Gem Lab in Taipei received two fire opals from a local dealer for identification: a 14.49 ct oval cabochon (23.80 × 15.27 × 9.12 mm; figure 22, left) and a 1.09 ct cushion cut (7.67 × 7.64 × 4.67 mm; figure 22, right),

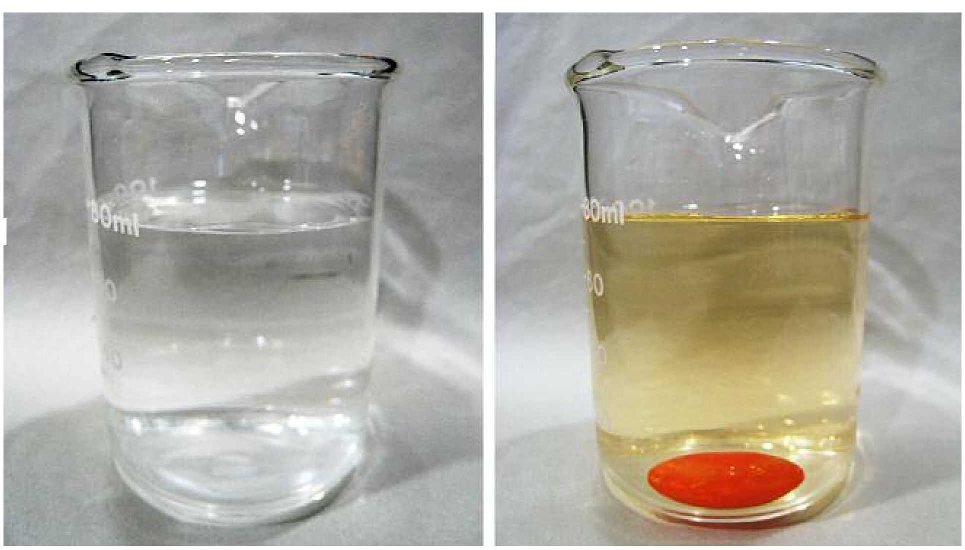

Figure 23. The clear water in this glass beaker (left) turned orange after soaking the fire opal cabochon overnight at room temperature (right). The obvious water discoloration proves the sample was dyed. Photos by Larry Tai-An Lai.

Figure 24. After removal from the water, this cabochon exhibited an obvious loss of color, indicating dye treatment. Photo by Larry Tai-An Lai.

both with pronounced play-of-color. Both gave RI readings of about 1.45. Observation with a gemological microscope and analysis with an FTIR/Raman spectrometer revealed the structure expected for natural opals. Orange color concentrations around some surface pits were also observed, indicating treatment.

But the most obvious clue was the effect caused by immersing the cabochon in water at room temperature overnight (figure 23). The water took on an orange tint, proving that the dye applied to these stones was water soluble, unlike the dye examined by Renfro and McClure (2011). The cabochon itself showed considerably less color after it was removed from the water (figure 24). This simple result reinforces the need for caution when buying fire opals, even ones with a natural-looking color. The change in color would come as a shock to any client unfortunate enough to purchase this dyed material without suitable disclosure.

Larry Tai-An Lai
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CONFERENCE REPORTS

June 2013 Hong Kong Jewellery & Gem Fair. The former British colony of Hong Kong, now a Special Administrative Region of the People's Republic of China, is one of the world's top ten importers and exporters. Almost half of Hong Kong's trade is with mainland China, which makes it a very attractive site for global gem and jewelry trade shows. The fact that it is a free trade port with no tariffs and minimal licensing also draws suppliers and buyers. Alongside Las Vegas and Basel, Switzerland, Hong Kong's gem and jewelry shows rank among the world's most important in terms of size, trading, and industry trendsetting.

Three main Hong Kong shows are held every year, in March, June, and September. The September show is the largest, with two venues needed to accommodate all the exhibitors. The June and September events are organized by UBM, a global communications and marketing firm. This year's June show, the 26th annual Hong Kong Jewellery & Gem Fair, was held at the Hong Kong Convention and Exhibition Center (HKCEC), concurrently with Asia's Fashion Jewellery and Accessories Fair. It featured several themed pavilions, including diamonds, colored stones, jadeite, pearls, fine jewelry, antique jewelry, design, and manufacturing tools.

Approximately 40,000 buyers attended the June show, a figure that typically reaches 60,000 for the September event. While the buyers represented a truly international mix, the percentage from China has consistently increased. As the country's wealth continues to soar, the emergence of a consumer economy has led to strong gains in gemstone and jewelry purchases. As Chinese consumers become more sophisticated, they are demanding higher-quality diamonds, gemstones, and jewelry.

Western designs and brands are also popular in China, which has created opportunities for international suppliers, including diamond dealers and gold jewelry manufacturers. China is the world's largest buyer of gold. With its free trade policies, geographic location, and tradition as a gateway to China, Hong Kong is poised to remain Asia's largest trading hub for fine jewelry, as well as a top destination for international suppliers and buyers.

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