G&G Micro-World

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Man-Made "Inclusion"?

Two important internal features in gemstones are inclusions and fissures. Inclusions are an important source of information in the gemological evaluation process. They can often provide indications about geographic origin (see, e.g., S. Saeseaw et al., "Three-phase inclusions in emerald and their impact on origin determination," Summer 2014 *G&G*, pp. 114–132); growth conditions (e.g., A. Cheilletz et al., "Time-pressure and temperature constraints on the formation of Colombian emeralds: An ^{40}Ar/^{39}Ar laser microprobe and fluid inclusion study," *Economic Geology*, Vol. 89, No. 2, 1994, pp. 361–380; J.G. Toloza et al., "Similarities and differences between fluid inclusions hosted by Colombian emeralds," *Special Issue on the 15th IAGOD Symposium*, 2018, pp. 166–167); natural or synthetic origin (e.g., N.D. Renfro et al., "Chart: Inclusions in natural, synthetic, and treated emerald," Winter 2016 *G&G*, pp. 402–403); and whether the stone has been treated to improve clarity.

Fissures are openings in the stones, and those "empty" spaces affect clarity in a negative way. Therefore, gemstones are treated to fill those gaps with different substances. Fillers can generate physical phenomena that resemble natural inclusions and may be misleading to the untrained eye.

Emeralds are most commonly enhanced with fissure filling. Fillers in emerald are identified by looking at the flash of color (blue, violet, orange, and yellowish) shown when the transmitted light of the microscope hits the inclusions or with the use of FTIR or Raman spectroscopy.

Recently the authors identified a man-made "inclusion" in a 1.15 ct cushion-cut Colombian emerald (figure 1) measuring 8.2 × 7.3 × 3.1 mm. It displayed a typical yellow flash most commonly seen in emeralds filled with liquid resin, which was later corroborated by FTIR spectroscopy. The "inclusion" was barely visible and almost transparent under regular microscope light sources (transmitted and reflected). Therefore, a 365 nm UV light was employed to check whether the foreign object fluoresced, and in fact it did (figure 2).

In the authors' opinion, the fiber could only have come from one of three sources: a cotton handkerchief, a microfiber jewelry cloth, or the net used during the immersion

Figure 1. Fiber inside a cavity filled with liquid resin in a cushion-cut emerald measuring 8.2 × 7.3 × 3.1 mm. Photomicrograph by Holman Alvarado.

About the banner: This rock crystal quartz from Brazil contains a band of bright red hematite flakes. Photomicrograph by Nathan Renfro; field of view 7.22 mm. Courtesy of the John Koivula Inclusion Collection.

Gems & Gemology, Vol. 56, No. 3 pp. 426–435.

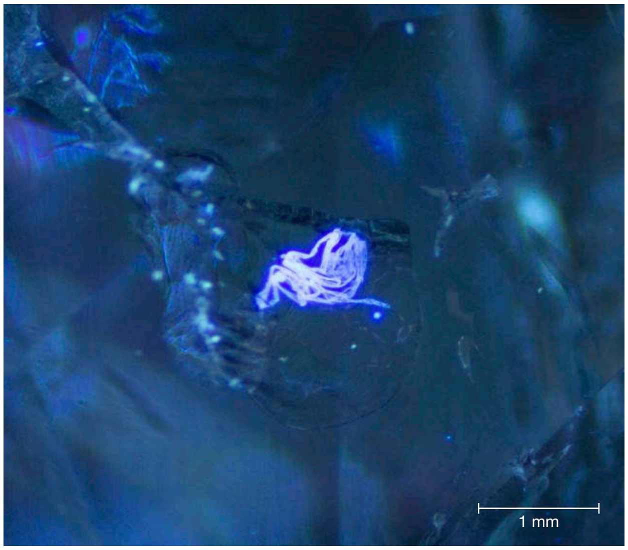
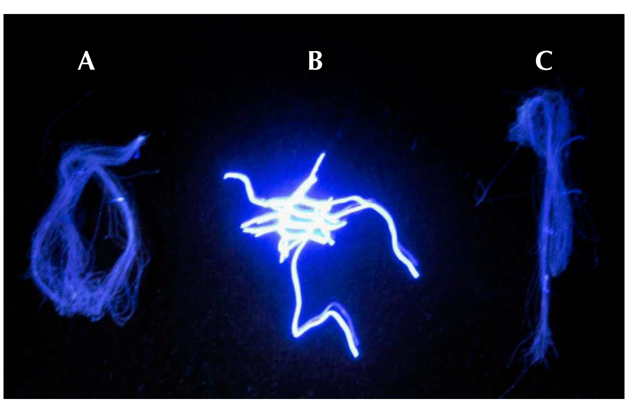


Figure 2. Fiber seen inside the cavity under 365 nm UV light at 50× magnification. Photomicrograph by Luis Gabriel Angarita.

of the stones in the enhancement container of the oiling machine. The first two would imply that a piece of fiber got stuck to the surface of the stone before the enhancement procedure took place, which is highly unlikely. The third source seemed the most plausible. In order to test that hypothesis, three strains of fibers were collected and compared using Raman spectroscopy with a Horiba HR Evolution with the laser operating at 532 nm. Although the spectra showed peaks in the 500 to 1500 cm^{-1} range, they were not specific for either of the fibers. Using the Olympus BX 41 microscope at 1000×, the diameter of the fiber was measured, and there was consistency with the diameter of the fiber collected from the net, about 50 µm. A 365 nm UV light source was also used to differentiate among them. A clear difference was evident between the nylon fiber from the net and the other two fibers (figure 3). This second test corroborated the authors' hypothesis.

Figure 3. Three fibers under 365 nm UV light. A: Cotton handkerchief used to remove oil from the surface of the stone. B: Net fiber in which emeralds are placed for the enhancement process. C: Jewelry cleaning cloth also used to remove oil from the surface. Photomicrograph by Javier Toloza.



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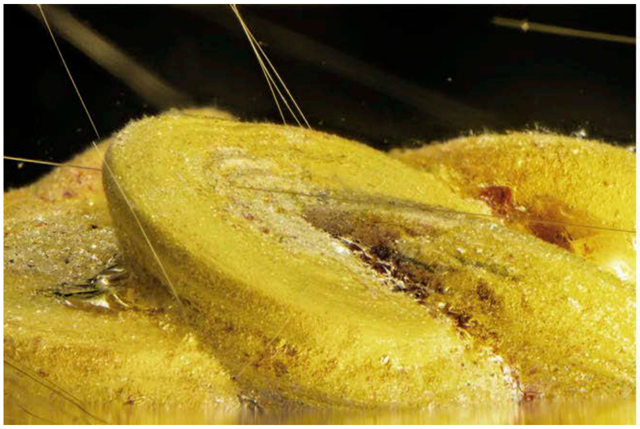
Manufactured Inclusions in Gem Materials

Inclusions in gems have gained popularity as social media has exposed collectors to a wide range of gem materials with interesting inclusions. As a result, there has been an increase in artificial inclusions in natural rock crystal quartz as predicted by E. Skalwold (Summer 2016 Micro-World, pp. 201–202). Recently, the authors had the opportunity to examine several unique gems with manufactured inclusions. Microscopic examination revealed that the main methods for manufacturing inclusions were carving, assembling, dyeing, three-dimensional internal laser engraving, or a combination of these methods.

A 47 ct rutilated quartz cabochon exhibits an eye-catching yellow and red floral-shaped inclusion produced by creative carving and filling with a colored composite material (figure 4). Microscopic examination revealed cir-

Figure 4. This 47 ct cabochon was creatively carved from the back, and the hollow cavity was then filled with a yellow and red composite material to highlight the floral inclusion. Photo by Robert Weldon; courtesy of Mike Bowers.



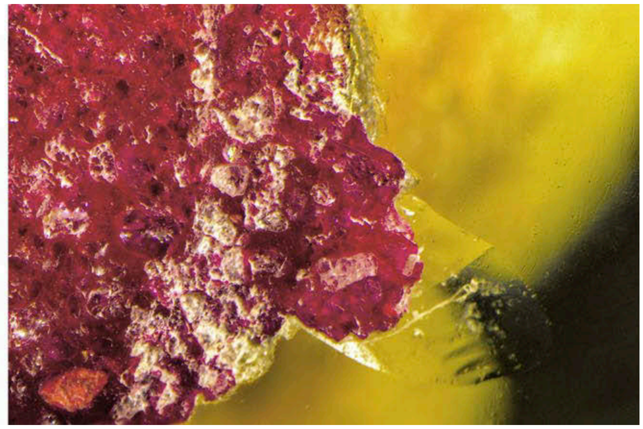


Figure 5. Tool marks from a rotary abrasive burr are clearly visible in this manufactured floral-shaped inclusion (left; field of view 9.72 mm). The cavity was subsequently filled with a yellow and red composite material consisting of sand grains and a colored binder or resin material (right; field of view 4.26 mm). Photomicrographs by Nathan Renfro; courtesy of Mike Bowers.

cular marks from a rotary abrasive tool used to create the intricate cavity (figure 5, left). The cavity was subsequently filled with a yellow and red composite material of fine sand grains and a colored binder or resin (figure 5, right).

Another creatively manufactured inclusion in a quartz gem can be seen in a 226 ct round tablet containing a large fracture (figure 6). The fracture was filled with colored orange and yellow resin, which resembles natural iron oxide epigenetic staining sometimes seen in rock crystal quartz. A quick examination in the microscope revealed incomplete filling and trapped gas bubbles in the colored resin (figure 7), making the separation between this manufactured inclusion and its natural counterpart quite easy.

Creative dyeing also produced the manufactured inclusion in a 109 ct quartz with a completely enclosed green

Figure 6. This 226 ct round tablet of quartz contains an orange and yellow resin-filled fracture that convincingly imitates natural iron oxide mineral staining in quartz. Photo by Robert Weldon; courtesy of Mike Bowers.

Figure 7. A fracture in this rock crystal quartz has been filled with orange and yellow resin to give the appearance of natural iron oxide staining. Trapped gas bubbles make this manufactured inclusion readily identifiable. Photomicrograph by Nathan Renfro; field of view 8.94 mm. Courtesy of Mike Bowers.

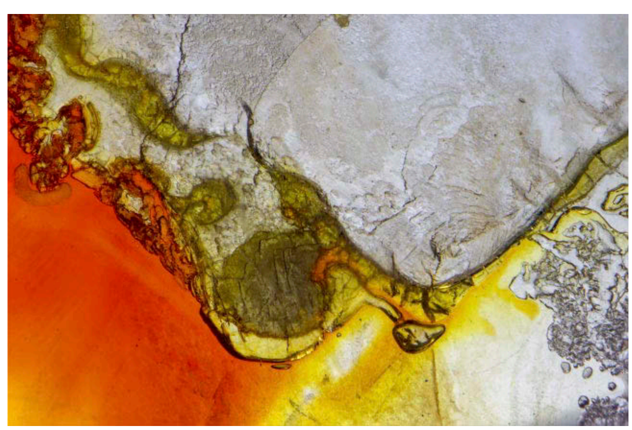


Figure 8. This 109 ct polished block of quartz has been assembled from two halves that contain numerous fractures filled with green resin. They have been glued together so that the moss-like inclusion is completely enclosed in the colorless quartz. Photo by Robert Weldon; courtesy of Mike Bowers.

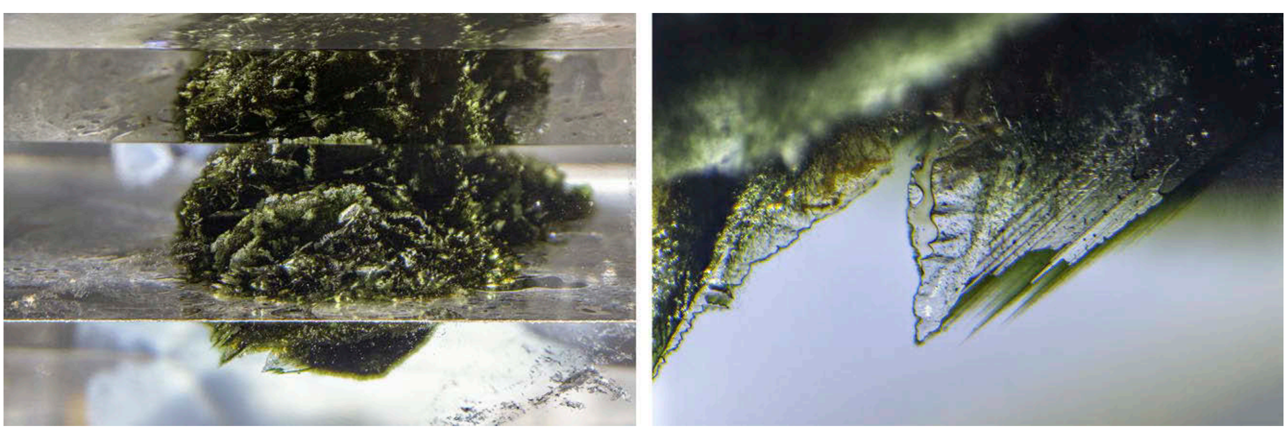
Figure 10. This 724 ct rock crystal quartz contains two manufactured inclusions induced by a 3D subsurface laser-engraving process. Photo by Robert Weldon; courtesy of Mike Bowers.

moss-like inclusion (figure 8). This stone consists of two pieces of rock crystal quartz, each with a centralized network of fine fractures that may have been artificially induced by laser, judging from their unnatural irregular pattern and striated appearance. The fracture network in each piece was subsequently filled with a dark green resin to give the appearance of a moss-like inclusion. The two halves were then glued together with colorless cement, completely enclosing the green moss-like inclusions in water clear rock crystal quartz. This left a somewhat obvious assembly plane (figure 9, left) when examined with the microscope, as well as trapped gas bubbles in the green resin-filled areas (figure 9, right).

The fourth example of manufactured inclusions recently examined is a 724 ct quartz crystal with a polished face that contains two white stellate inclusions consisting of numerous radial arms surrounding a spherical core structure (figure 10). Closer examination revealed a carefully layered series of micro-fractures consistent with 3D subsurface laser engraving (figure 11). This is by far the most technologically advanced example of a manufactured inclusion in a gem material examined by author NR.

While these four examples of manufactured inclusions may not be quite as sought after as gems with natural inclusions, they certainly can be appreciated for the efforts

Figure 9. This moss-like inclusion results from a fracture network in two pieces of rock crystal quartz filled with green resin and glued together to encapsulate the inclusion. The assembly plane of the two quartz halves is clearly visible on the side of the stone (left; field of view 18.80 mm). The green resin contained trapped gas bubbles, and the fractures displayed an unnatural striated appearance suggesting they were artificially induced (right; field of view 2.59 mm). Photomicrographs by Nathan Renfro; courtesy of Mike Bowers.



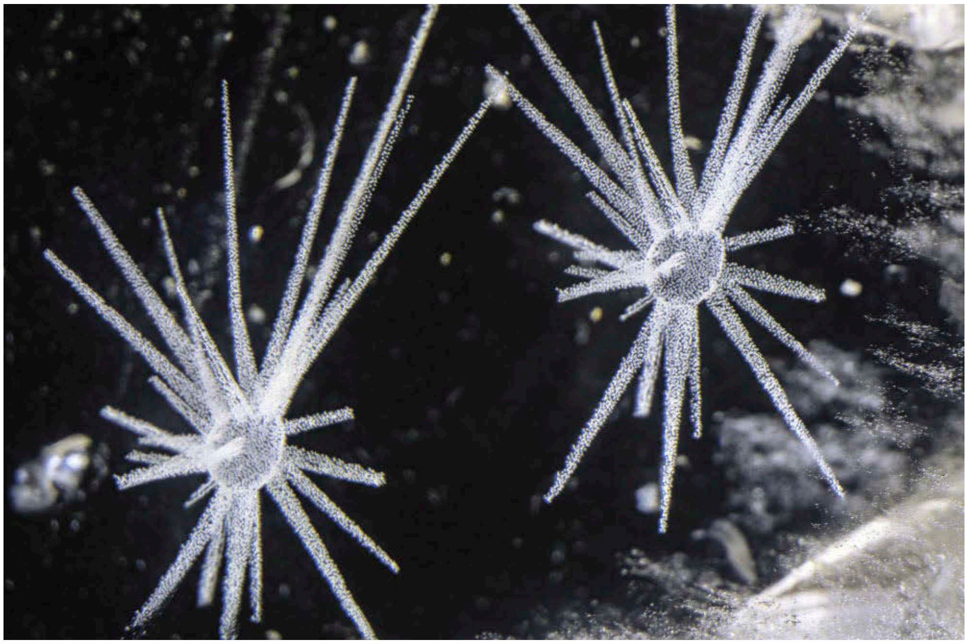


Figure 11. These stellate inclusions result from 3D subsurface laser engraving, which creates light scattering microfractures in a controlled pattern. These represent the latest advancement in manufactured inclusions within gem materials. Photomicrograph by Nathan Renfro; field of view 23.50 mm. Courtesy of Mike Bowers.

and techniques employed by the manufacturers. Obviously, collectors of gems that feature inclusions should be aware that manufactured inclusions such as those described here exist in the trade. While some manufactured inclusions may be intended purely as an artistic enhancement, others may be produced with the intention to deceive the consumer, and caution should be used if a manufactured origin is suspected.

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Interesting "Egg" in Rock Crystal Quartz With Rutile

The micro-world of gemology is fascinating and often mimics scenery, landscapes, or, in this case, food. Recently the authors examined a natural rock crystal quartz that showed needles piercing a yellow and white circular inclusion (figure 12). The inclusion mimicked a fried egg and was seen during microscopic examination using fiber-optic lighting. The "egg" was actually an example of epigenetic residue surrounding a rutile needle trapped in a crack

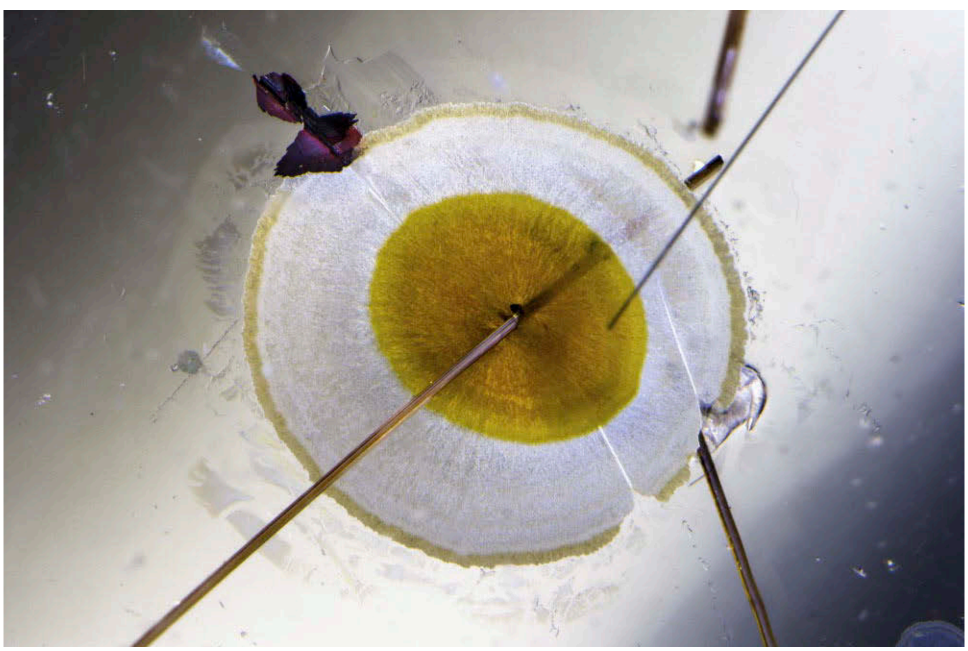

Figure 12. Rutile needles extending outward from epigenetic growth in rock crystal quartz. Photomicrograph by Nathan Renfro; field of view 8.81 mm. Courtesy of Mike Bowers.

within the host crystal. Here the rutile, a mineral composed primarily of titanium dioxide (TiO_2), is a syngenetic inclusion, having formed at the same time as the host quartz crystal, and the "egg" formed its circular shape after the rutile and quartz growth stopped. The epigenetic residue in this stone results from secondary fluids that enter surface-reaching fractures (see descriptions and images of inclusions in quartz in E.J. Gübelin and J.I. Koivula, *Photoatlas of Inclusions in Gemstones*, Vol. 2, 2005, Opinio Publishers, Basel, Switzerland, p. 541). Although quartz is relatively common as a host material, it is often an excellent source for unique mineral inclusions and, with this particular crystal, a fun novelty inclusion. Epigenetic residues in gems and crystals not only provide for interesting inclusion scenes, but they can also help determine growth phases and possible treatments.

Amy Cooper and Nathan Renfro
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Blue Inclusion in Rock Crystal Quartz

An interesting tuft of light blue needles was recently observed in a polished modified cube of rock crystal quartz (figure 13). The needles nucleated on a nearly invisible quartz crystal with the tuft tapering toward the opposite end (figure 14). When the tuft was examined looking parallel to the optic axis of the host quartz using cross-polarized light, the small included quartz crystal surrounded by the blue needles stood out in high relief due to the crystallographic misalignment with the host quartz. It was also interesting to note that the included quartz was perched at the apex of a twinned sector of the host quartz (figure 15). While the needles were located too deep within the

Figure 13. This 118.16 ct polished modified cube of rock crystal quartz contains an interesting tuft of blue needles, which may be tourmaline. Photo by Robert Weldon; courtesy of Mike Bowers.

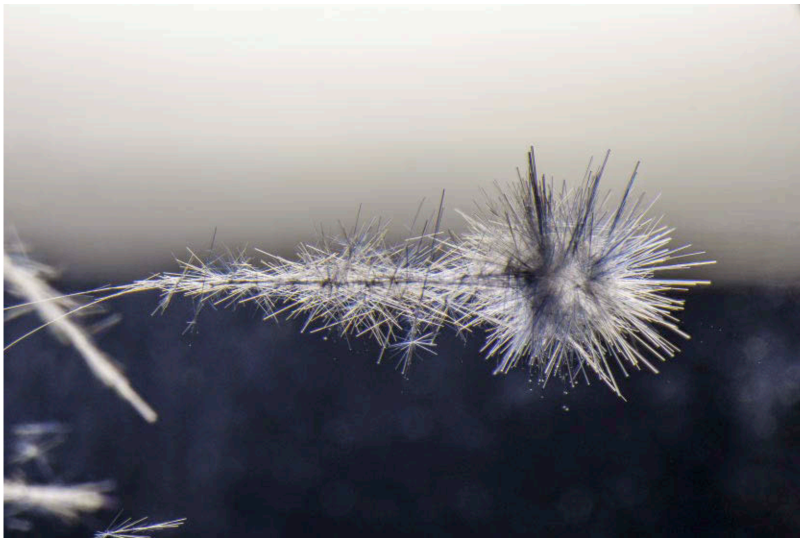
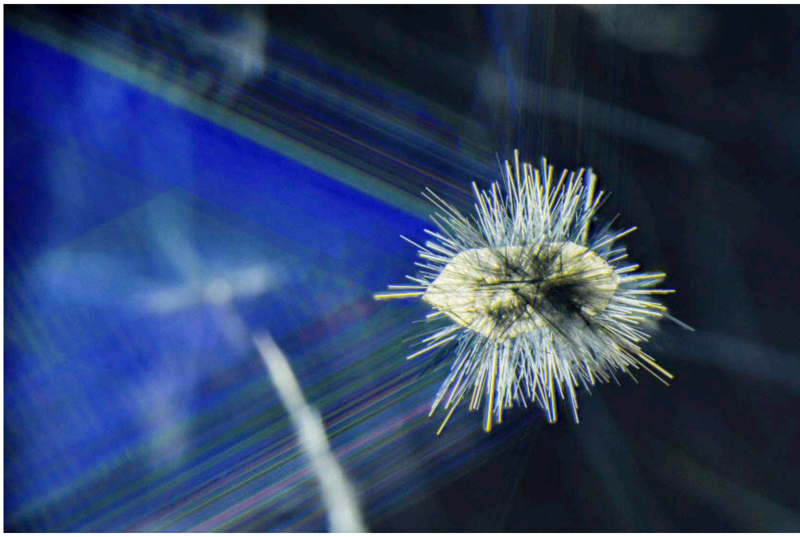


Figure 14. This tuft of blue needles has nucleated on a quartz crystal and tapers toward the end opposite the core quartz crystal. Although too deep to be identified by Raman spectroscopy, the needles resemble light blue needles of tourmaline. Photomicrograph by Nathan Renfro; field of view 10.07 mm. Courtesy of Mike Bowers.

Figure 15. When the host quartz is examined looking parallel to the optic axis, the core quartz crystal stands out in high relief and is interestingly located at the apex of a twinned sector of the host. Photomicrograph by Nathan Renfro; field of view 9.32 mm. Courtesy of Mike Bowers.



quartz to conclusively identify with Raman spectroscopy, their appearance (in the author's experience) suggests they may be pale blue tourmaline. This beautiful inclusion in rock crystal quartz is a striking example of a microfeature in a gem cut to showcase an inclusion.

Nathan Renfro

Tree in Rock Crystal Quartz

The unusual quartz cabochon shown in figure 16 displays visually appealing dendritic inclusions that resemble trees. This inclusion scene is an excellent example of pareidolia, the tendency to assign familiar shapes to abstract forms (see Winter 2007 Lab Notes, pp. 363–364). Combined with a unique illumination technique, the inclusions are reminiscent of trees with green grass and a blue sky (figure 17).

The dendritic "tree" patterns are metal sulfide inclusions, as evidenced by their brassy yellow metallic color and opaque nature. When viewed with diffused fiber-optic lighting, the metal sulfide inclusions show a crisp foil-like texture near a partially healed internal fracture. This is likely where the metal sulfides entered the quartz host. They were deposited as an epigenetic residue in the fracture near the base of the tree, which was later healed, leaving behind the metal sulfides and numerous minute fluid inclusions where the fracture once was.

Adding green and blue filters behind the stone, known as modified Rheinberg illumination (Fall 2015, pp. 328–229), helped complete the appearance of a micro-landscape contained in the rock crystal quartz. This lighting technique is characterized by using contrasting color filters to bring striking color contrast to scenes viewed with a microscope (N.D. Renfro, "Digital photomicrography for gemologists," Summer 2015 *G&G*, pp. 144–159). This unique quartz gem can be appreciated for the beautiful inclusion scene it contains.

Amy Cooper and Nathan Renfro

Figure 16. This 80.60 ct rock crystal quartz cabochon contained an interesting tree-like metal sulfide inclusion. Photo by Robert Weldon.

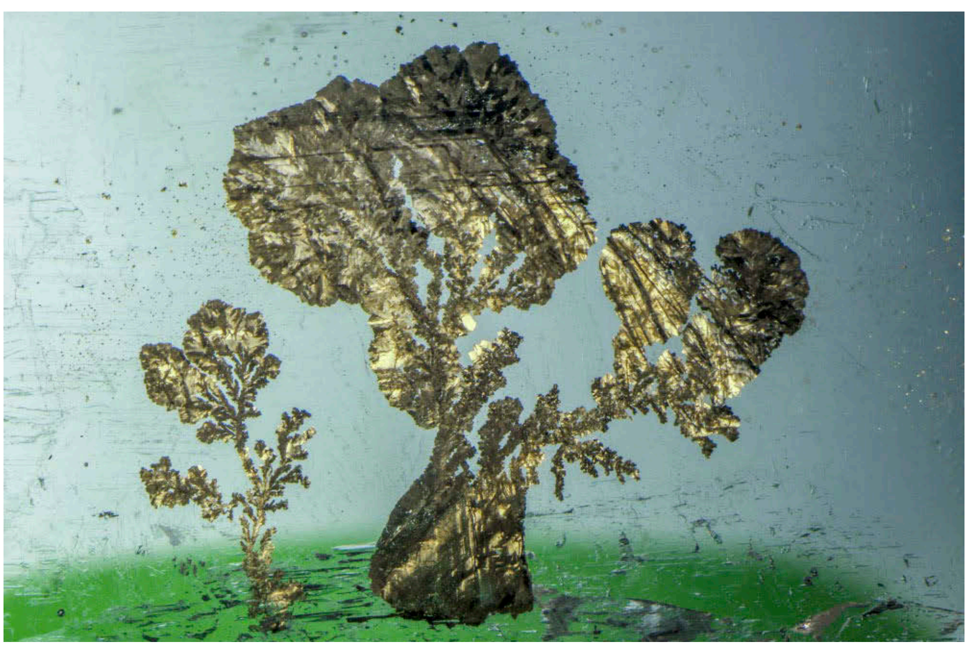


Figure 17. Epigenetic metal sulfides are trapped in a partially healed fracture of rock crystal quartz. Modified Rheinberg illumination provides artificial blue and green color contrast reminiscent of grass and a sky. Photomicrograph by Nathan Renfro; field of view 20.14 mm. Courtesy of Mike Bowers.

Figure 18. Left: When the pinkish purple sapphire was viewed under darkfield illumination, the only inclusions visible underneath the table were zircon crystals and rutile needles. Right: Examining the same area with fiber-optic light revealed unknown dendritic inclusions. Photomicrographs by Ezgi Kiyak; field of view 2.90 mm.

Unknown Dendritic Inclusions in Sapphire

The authors recently examined a 4.03 ct light pinkish purple unheated sapphire. When viewed under darkfield illumination, the inclusion scene at first seemed fairly vacant and occupied only by sparse zircon crystals and rutile needles (figure 18, left). However, microscopic observation with fiber-optic light revealed large, delicate dendritic inclusions underneath the table (figure 18, right) that resembled a fern frond. The dendritic inclusions seemed to be flat and parallel to each other and extended the entire length of the table. Small round platelets were associated with the "branches" of the inclusions.

Further examination with a polariscope and conoscope helped prove that the dendritic inclusions were oriented parallel to the basal plane of the host corundum. A distinct uniaxial interference pattern was observed when viewing these inclusions from a perpendicular angle. The thinness and relative distance to the surface of the stone made it difficult to identify the inclusions with Raman spectroscopy. This is the first example of such dendritic inclusions in sapphire the authors have observed. However, a similar inclusion has been observed previously in a heat-treated blue sapphire (see Spring 2007 Lab Notes, pp. 54–55).

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Spinel on Sapphire

Recently the authors examined a 4.8 cm tall pale blue sapphire crystal (figure 19) that featured numerous purple spinel crystals up to 8 mm in size on the surface (figure 20), a rather uncommon association. Steve Dubyk of Albuquerque, New Mexico, had acquired this specimen and doubted the accuracy of the accompanying label, indicating aquamarine from Tres Pozos in Baja California, Mexico. Raman spectroscopy confirmed Mr. Dubyk's suspicion that the specimen was in fact corundum and identified the associated purple crystals as spinel. LA-ICP-MS chemical analysis suggested that the specimen was from Sri Lanka based on the trace elements present, which indicated an average of 130 ppma iron (Fe), 38 ppma titanium (Ti), and

Figure 19. This 4.8 cm pale blue sapphire crystal contained numerous purple spinel crystals on the surface. Photo by Angelica Sanchez; courtesy of Steve Dubyk.

Figure 20. Purple spinel crystals were intergrown on the outermost layer of the sapphire crystal, which appears to be from Sri Lanka based on trace element chemistry. Photomicrograph by Nathan Renfro; field of view 13.42 mm.

37 ppma magnesium (Mg). The trace element chemistry is also notable, as the magnesium will preferentially charge compensate the titanium, leaving very little excess titanium to pair with iron to produce a blue color. The chemistry measurements were consistent with the very pale blue color observed in this stone. The chemistry of the spinel was also interesting in that its reasonably saturated purple color would indicate it was at least partially caused by chromium. However, the spinel was inert to long-wave UV light, suggesting no chromium was present. LA-ICP-MS testing confirmed the absence of chromium but showed relatively high iron (10,566 ppma) and some cobalt (2.75 ppma average) which together are likely responsible for the purple color (A. Palke and Z. Sun, "What is cobalt spinel? Unraveling the causes of blue color in blue spinel," Fall 2018 *G&G*, p. 262). While spinel is uncommon in sapphire, a spinel inclusion has been previously reported in a Sri Lankan yellow sapphire (Winter 2015 Micro-World, p. 444). This sapphire is one of the more unusual examples the authors have encountered.

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Staurolite in a Mozambique Ruby

Rubies from Mozambique have a well-known inclusion suite that can consist of particle clouds, planes of platelets, and negative crystals as well as sulfite and amphibole crystals. These inclusions and their trace element chemistry make the geographical origin of Mozambique rubies less difficult to decipher. Recently, a 1.11 ct unheated ruby was examined by the author for identification. Internally there were scattered silk with planes of thin films as well as negative crystals. These inclusions plus trace element chemistry collected by laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS) analyses confirmed the stone to be from Mozambique.

Mozambique rubies have a consistent suite of inclusions, and noting something out of the ordinary is quite uncommon. Alongside the negative crystals were clusters of a transparent orangy red crystal that the author had no prior knowledge of seeing in a Mozambique ruby (figure 21). Raman spectroscopy identified the unknown crystals

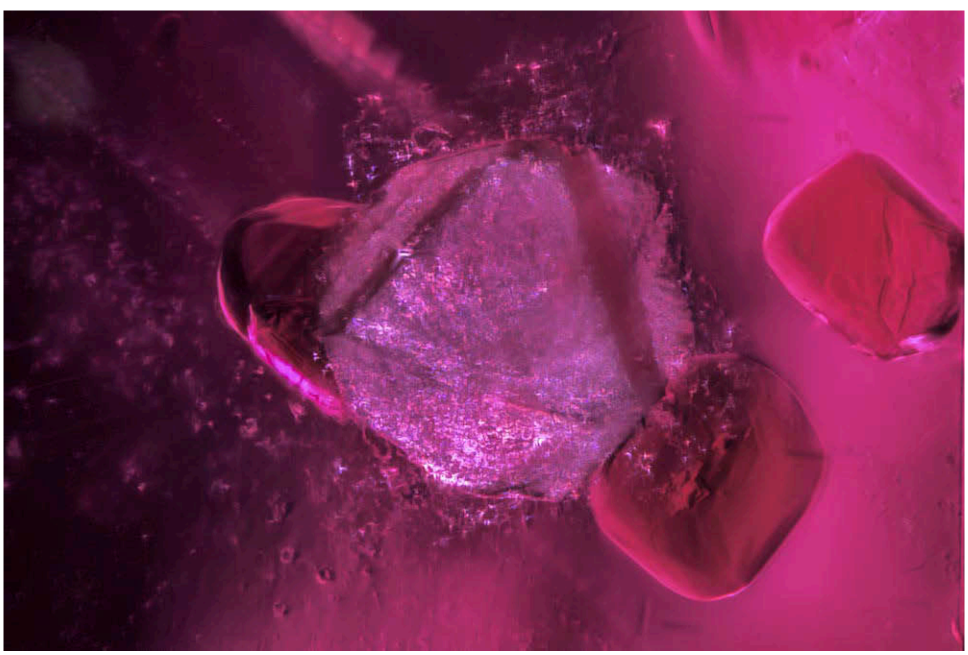

Figure 21. Orangy red staurolite crystals surround a negative crystal in a Mozambique ruby. Planes of thin films can be seen behind them. Photomicrograph by Nicole Ahline; field of view 1.42 mm.

as staurolite, a nesosilicate mineral known to occur in Mozambique. In a recent issue of *G&G*, staurolite was examined and documented for the first time as an inclusion in corundum, specifically in a Madagascar ruby (Spring 2020 Micro-World, pp. 144–145). The author believes that this staurolite is the first of its kind documented in a Mozambique ruby. Inclusions such as these will forever keep gemologists on the lookout for the next unknown.

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Quarterly Crystal: Ferrocolumbite in Topaz

The author recently acquired a 239.25 ct transparent well-formed topaz crystal from the collection of Leon M. Agee. The crystal from the Shigar Valley in Pakistan, shown in figure 22, has a flat base formed by a cleavage plane. The termination appears to be a pyramid form. The crystal plays host to two prominent opaque black inclusions and one smaller similar-appearing inclusion that is close to the surface of the host (figure 23). The two larger inclusions were too deep in the topaz to analyze. However, the small inclusion was near the surface of one of the prism faces and could be reached by laser Raman microspectrometry. Testing showed a very close match to ferrocolumbite (columbite-Fe). Because of the resemblance between the smaller inclusion and the two larger ones, we concluded that the two larger inclusions were also ferrocolumbite. As an interesting aside, these inclusions are reminiscent of star cruisers in the Star Wars anthology.

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Figure 22. This 239.25 ct topaz crystal hosts some interesting inclusions of ferrocolumbite. Photo by Diego Sanchez.

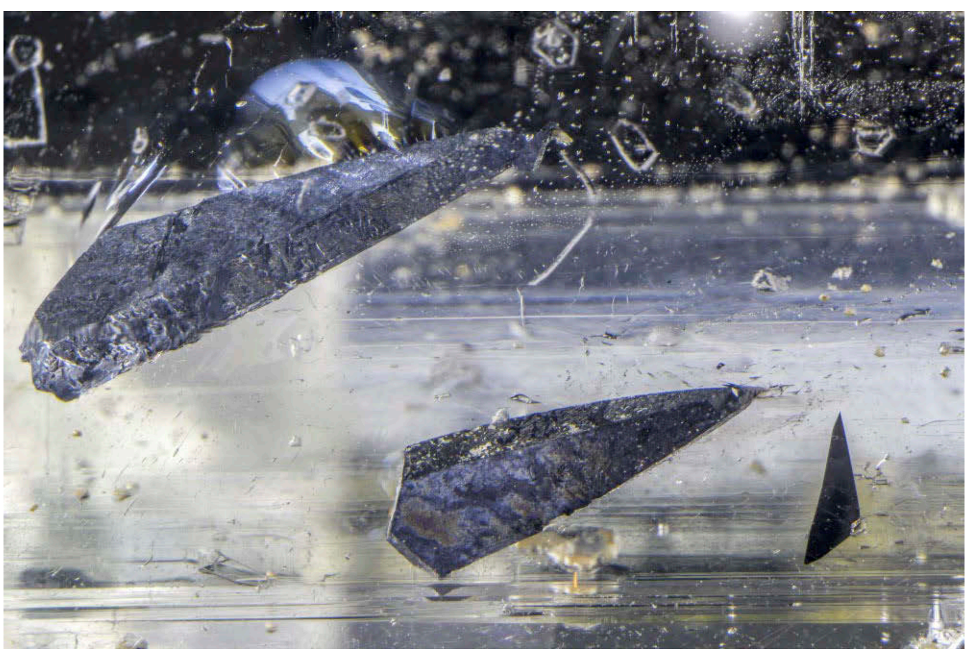

Figure 23. Laser Raman microspectrometry identified the smallest of these inclusions as ferrocolumbite. Photomicrograph by Nathan Renfro; field of view 17.62 mm.