

GEM TRADE LAB NOTES

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CERAMIC Statue

Occasionally, carvings or statues come into the lab that we suspect are created from manufactured materials, but the difficulty may come in trying to prove this. A case in point occurred last May, when the West Coast lab was asked to identify an opaque, artistically stained "carving" of a seated man with a dragon (figure 1). Several clues led us to suspect that it was fashioned from a material that was not of natural origin.

For example, the "carving" had a half-inch [approximately 1 cm] hole in its base. This would serve no obvious purpose in a rock carving, but it would enable gases to escape from a ceramic during firing. Also, certain scratches on the statue showed parallel striations that were difficult to explain unless they had occurred while the material was still soft (i.e., before firing). The object was cracked in several places, and some of the cracks were filled with a material that burned when touched with the thermal reaction tester (hot point). The dragon's snout had been replaced with what appeared to be the same filling material. The balance of the cracks were empty. None showed any of the vein-filling minerals (e.g., quartz, calcite) that might be expected in a rock.

To confirm our suspicions, we took a minute amount of powder from two spots on the statue's surface for X-ray diffraction analysis. Long exposures to the X-ray beam produced patterns that contained 15 faint lines. These were subsequently identified as matching lines in the patterns of mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$),



Figure 1. This 40-mm-high "carving" proved to be a ceramic product.

kalsilite (KAlSiO_4), and possibly gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$). We believe the patterns were faint because most of the material had become amorphous after firing, with only a small amount of mullite and kalsilite formed. Energy-dispersive X-ray fluorescence spectroscopy (EDXRF) detected Al, Si, Ca, K, and a trace of Fe, which was in agreement with these identifications.

Mullite, kalsilite, and gehlenite are rare minerals that occur in unusual geologic environments. In particular, mullite occurs only in metamorphic rocks, and kalsilite occurs only in alkali basalts; they do not occur together. However, all three minerals are known to occur in ceramics; in fact, mullite was originally discovered in firebricks and porcelains. Thus, the unusual mix of minerals conclusively proved that this statue was a fired, but unglazed ceramic.

MLJ, Dino De Ghionno, and
Patricia Maddison

Large "Knot" in CITRINE

As gemologists, we usually think of inclusions as minute internal features seen only with magnification. For most gem materials, the presence of eye-visible inclusions is considered to detract from the value. In other instances, however, eye-visible inclusions are desirable and are even included in the gemstone's name. Examples of the latter include acicular rutile crystals in rutilated quartz, tourmaline needles in tourmalinated quartz, and dendritic manganese oxide precipitates in dendritic and moss agates. Still a third category, however, is the presence of an unusual eye-visible inclusion in an otherwise transparent gem material that gives that gem a distinctive appearance.

The West Coast lab received for identification a sizable (229.09 ct, measuring $40.40 \times 28.31 \times 21.60$ mm) faceted stone that appeared to have been cut specifically to display a large included crystal in the center of its table (figure 2), with the inclusion's well-formed first-order prism termination pointing toward the pavilion. Using standard gem-testing techniques, we identified the transparent, light brownish yellow, modified rectangular step cut as natural citrine quartz.

Using similar testing methods, we determined that the large inclu-

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

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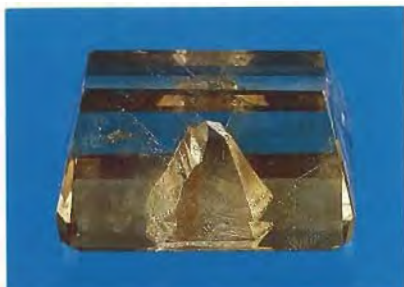


Figure 2. This 229.09-ct citrine was apparently cut to prominently display a large quartz inclusion.

sion was also quartz. Polariscope testing showed that the crystallographic orientation of the inclusion was different from that of the host. With diamonds, the term *knot* is used to describe an included diamond crystal that is oriented differently from the host diamond. By this definition, the inclusion in this citrine could be thought of as a quartz "knot." RCK and KH

DIAMOND

Americium-Treated Green Diamond

A 0.43-ct yellowish green, Old European-cut diamond submitted to the East Coast laboratory for an origin-of-color report revealed uneven color that was a challenge to interpret. The stone had two naturals at the girdle, but neither area was green. Instead, shallow, pale green blotches (which did not photograph well because they were very subtle) were visible on the table and the lower half of the pavilion. We saw them best by using diffused light with the stone immersed in methylene iodide. The visible spectrum, obtained with a Pye-Unicam SP8-400 spectrophotometer, showed a broad GR1 absorption band at 741 nm; this is typical of "surface-skin" green coloration, whether produced by natural irradiation or by laboratory treatment.

However, the pattern of green spots on polished facets of this stone indicated treatment with radioactive salts. Such treatment usually leaves some residual radioactivity in the

diamond, but this stone showed no radioactivity above background levels when checked with a handheld Geiger counter. To determine if there was radioactivity below the detection limits of this type of meter, we sent the stone to the West Coast laboratory for radiation testing. A three-hour scan for gamma rays revealed a small, but significant, peak from americium-241 (^{241}Am), an artificial radionuclide, thus proving that the color of this diamond was due to treatment. The amount of radioactivity present corresponded to 0.09 nCi/g of ^{241}Am . (Analysis of another americium-treated diamond was described by C. E. Ashbaugh III in the article "Gamma-Ray Spectroscopy to Measure Radioactivity in Gemstones," *Gems & Gemology*, Summer 1992, pp. 104–111.)

Possession and sale of ^{241}Am , which has a half-life of 432.7 years, is regulated in the U.S. by the Nuclear Regulatory Commission. Because ^{241}Am is readily oxidized, can be inhaled, and is also an alpha emitter (alpha particles transmit their energy to human tissue with high efficiency), the NRC limit for permissible ^{241}Am exposure is very low. This diamond, although too low in radioactivity to register on a Geiger counter, may not be legally sold or distributed in the United States until the 50th century (see the letter by W. W. Hanneman regarding such regulations, on page 80 of the Summer 1993 *Gems & Gemology*.)

The abraded facet junctions on the crown, together with its old-fashioned cut, indicated that this stone was faceted long before americium was first made in the late 1940s or early 1950s. The reader is cautioned that a diamond may be treated at any time to alter its color, so indications that a fashioned diamond is old do not guarantee that the color is of natural origin. IR and MLJ

Unusual Triangular Inclusion in a Diamond

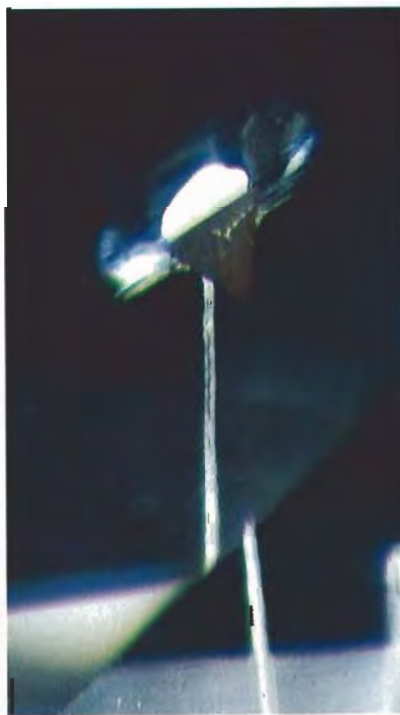
A 3.31-ct marquise-shaped brilliant-cut diamond submitted to the West Coast laboratory for full grading

revealed an unusual inclusion. In addition to a white "cloud" that had been reached by a laser drill hole (in itself not unusual), there was a curious dark, flat, triangular inclusion that had no connection to either the white cloud, the laser drill hole, or the surface of the stone (figure 3).

Just as interesting as the inclusion itself (which was presumably graphite) was the question of what caused it. Three possibilities came to mind:

1. Triangular surface features (trigons) occur as shallow, flat-bottomed depressions or shallow pyramidal pits. Perhaps this is a growth phantom, a trigon that was filled with the dark material before diamond growth continued around it.
2. Stacking faults in diamond can cause flat triangular (or truncated triangular) patches of hexagonal-

Figure 3. This triangular inclusion in a diamond appears to be filled with graphite. The laser drill hole seen here actually goes to the cloud behind the inclusion, not to the inclusion itself. Magnified 45 $\times$.



structure diamond (lonsdaleite), which is usually only detected by X-ray tomography (see, e.g., J. E. Field, ed., *Properties of Natural and Synthetic Diamonds*, Academic Press, London, 1992, pp. 217 and 218). This may have been an inclusion of lonsdaleite that, because it is less stable than (isometric) diamond, later reverted to graphite.

3. It may have been an inclusion of some other shape on which the faces altered to a negative-crystal macle, although the sharp triangular points make this unlikely. However, trigons have been seen in crystal inclusions in diamond (E. J. Gübelin and J. I. Koivula, *Photoatlas of Inclusions in Gemstones*, ABC Edition, Zurich, 1986, p. 95), and in diamond (Gem Trade Lab Notes, *Gems & Gemology*, Spring 1990, p. 95).

Because the triangular shape does not appear to be equilateral, the reverted-lonsdaleite explanation is probably the most plausible.

MLJ

Figure 4. This 47.31-ct cut-cornered, faceted triangle is a rock composed primarily of white grossular garnet and green diopside.



GROSSULAR-DIOPSIDE Rock

Various rough and cut samples of a handsome, mottled white-and-green rock (represented to be massive grossular garnet) were recently seen in the West Coast GIA Gem Trade Laboratory. All polished pieces of this material, like that in figure 4, show variations in luster. A spot refractive index of 1.74 was obtained on one piece.

Four separate materials could be seen through the microscope: high-luster, opaque round green grains; a lower-luster, semi-translucent white material that in most cases made up the bulk of the rock; a high-luster, translucent gray-to-brown material (usually occurring as veins); and a low-luster, semi-translucent white vein material. X-ray powder diffraction analyses proved these to be, respectively: diopside, grossular garnet, idocrase, and clinocllore (a member of the chlorite group). White grossular was the most common mineral by volume in most of the samples we examined, although at least one massive piece was predominantly idocrase; we also saw a brownish yellow-green semi-transparent idocrase cabochon.

The combination of grossular with diopside and idocrase is typical for calcium-rich veins (called rodingites) that are found in metamorphosed ocean sediments. This may be the origin of this material, and some of the rough we examined shows slickensides (striations caused by slippage along a fault plane).

Miners of this material informed us that it comes from northern Washington State. Core drilling indicates that the grossular-rich layer in the deposit is as much as 35 feet (about 11 m) thick. Blocks weighing up to almost 50 kg have been cut on site from this layer.

MLJ

Iron-Rich HORNBLENDE

A necklace of highly polished, 10-mm opaque black beads (figure 5) was purchased in Tucson, Arizona, as black jadeite that had been mined in that state. Our client was skepti-



Figure 5. Although these 10-mm opaque black beads were purchased as jadeite from Arizona, extensive testing revealed that they are actually ferrohornblende.

cal because of the appearance (e.g., quality of the polish) of the beads and the rarity of black jadeite (most commercial black jade is nephrite).

Although the gem name *jade* is properly used for either jadeite or nephrite, they are two wholly unrelated minerals that sometimes occur in similar colors. Jadeite is a single mineral within the pyroxene group, while nephrite is a solid-solution series between two closely related minerals of the amphibole group. What these jades have in common is toughness, but even this property is arrived at in different ways. Jadeite grains grow into interlocking aggregates, while nephrite forms compact, fibrous, matted aggregates.

Initial testing in the East Coast laboratory of one bead removed from the necklace gave results that could be mistaken for jadeite. The "spot" refractive index reading appeared to be midway between 1.60 and 1.70; the specific gravity was found to be 3.36. Still, the evenness of the polish just did not match the rather dimpled or uneven surface usually seen

on jadeite. Although most minerals in both the pyroxene and amphibole groups have R.I.'s between 1.6 and 1.7, have S.G.'s between 3.0 and 3.9, and occur in the same range of colors as jade, no other mineral in either group exhibits jade's toughness. To identify this material conclusively, we sent the bead to the West Coast laboratory for X-ray diffraction analysis.

The results indicated *ferrohornblende*, an iron-rich member of the amphibole group. Being amphibole, this material is certainly not jadeite; being ferrohornblende rather than nephrite, it cannot properly be called jade at all. As the amphibole group contains 60 separately recognized mineral species, finding a more exact designation than ferrohornblende most probably would require (destructive) petrographic testing. Although a well-known and pleasing name may aid the marketing of an opaque black material, gem names become devalued when they are misapplied.

GRC, MLJ, and IR

Figure 6. The jadeite jade beads of this necklace have such high diaphaneity that the stringing material is clearly visible through even the largest (9-mm) bead.



JADEITE JADE

Exceptionally Translucent Necklace

One pleasure of working in the laboratory is seeing gem materials that are exceptional examples of their species or variety. This past spring, the West Coast lab was asked to identify a double strand of 117 intensely colored green beads (figure 6), which ranged from about 9.06 to 3.77 mm in diameter. Standard gemological testing proved them to be jadeite jade, visible-light spectroscopy revealed that they were of natural color, and infrared spectroscopy showed that they were not polymer impregnated.

The fineness of the aggregate and the resulting high degree of diaphaneity were such that individual strands of the stringing cord could be clearly seen through even the largest bead.

RCK

Jadeite Jade with Impregnating Substance in Drill Hole

Bleached and polymer-impregnated jadeite jade is one of the most difficult detection challenges to confront the gem industry in the 1990s. As explained in the Fall 1992 *Gems & Gemology* article by E. Fritsch et al. (pp. 176–187), infrared spectroscopy is one of the few advanced testing methods that can detect this treatment in all cases. Although the instrumentation needed for such testing is not readily available to most gemologists, the above-referenced article did point out some tests that can be performed with standard gemological equipment. In particular, examination of the piece with magnification may reveal concentrations of the impregnating substance in irregularities, such as fractures or cavities, on otherwise smooth surfaces, or in recessed areas on carved jadeite that has been so treated.

The West Coast laboratory recently found another place where such concentrations might occur, when we examined a graduated necklace of mottled green and white beads that ranged from approximate-



Figure 7. Residue of the polymer material with which this jadeite bead was impregnated is evident in its drill hole. Magnified 28×.

ly 9.50 to 5.90 mm in diameter. Standard gemological testing identified the material as jadeite jade, and the spectra seen with a desk-model spectroscope confirmed that the green color was natural. However, microscopic examination revealed the presence of a colorless polymer layer in the drill holes of most of the beads (figure 7). Infrared spectroscopy confirmed that the material had been polymer impregnated.

Although the polymer in the drill holes provided very useful evidence that the beads had been polymer impregnated, such visual proof is—in our experience—very unusual. Only once before did we see similar evidence in jadeite beads that were later determined by infrared testing to be impregnated.

RCK

PEARLS

"Banded" Natural Pearl, of Natural Color

A very unusual pearl ring was examined last summer in the West Coast laboratory. Handcrafted of 18k matte gold, the ring was custom designed to highlight a rather distinctive pearl, which measured about 11 × 13 mm. The pearl was mounted such that it could be rotated to show on top either solid black (as in figure 8) or the distinctive banding (seen on the side in figure 8).

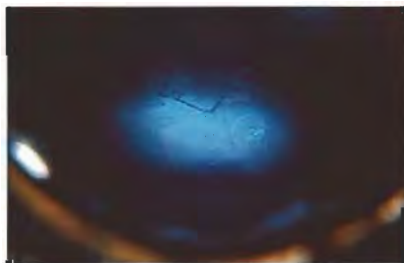
Our client asked us to determine if the barrel-shaped pearl, which was reportedly from the Gulf of Cali-



Figure 8. Different aspects of this unusual natural black pearl (11 × 13 mm) can be viewed by rotating it in its setting.

fornia, might be an assemblage because of the peculiar "banded" appearance. Visual examination showed no indication of assemblage. The "banding" consisted of nacreous layers that were alternately light and dark gray. The top and bottom layers appeared almost black with a very high luster and strong overtones of green and blue. When exposed to long-wave ultraviolet radiation, the dark areas fluoresced a very strong red, similar to the fluorescence seen in other black pearls from the Gulf of

Figure 9. The decrepitation of the nacre and resulting iridescence seen here often occurs on the surface of pearls that have been stained with a metallic compound. Magnified 18×.



California. The X-radiograph showed a large dark central core, proving that the pearl was indeed natural and not cut from the shell or an assemblage. We cannot imagine what might have caused such an unusual growth pattern in this pearl. KH

Cultured Pearl, Treated Color

The treated-color black cultured pearl seen in the East Coast lab and discussed here was one of a strand of 30, which were graduated in diameter from 16.35 to 11.45 mm. On the developed X-ray film, a white ring was seen around the nucleus of the pearl, proving that the pearl had been treated with a metallic compound (which is opaque to X-rays). EDXRF analysis, which revealed a silver signature typical of the metallic compounds often used to stain pearls, confirmed the treatment.

Visual examination with magnification also provided indications of treatment. Specifically, as illustrated in figure 9, we noted the damage to the nacre layers caused by the treatment process. The iridescence seen in this figure is a side effect of this breakdown of the surface. Another indicator, while atypical, relates to the peculiar hammered or "dimpled" surface seen on many natural pearls and on some thickly nacred cultured pearls (see, e.g., the natural-color cultured pearl in figure 15, p. 128, of the Summer 1993 Lab Notes section). Typically, when a pearl is treated with some type of silver salts, the entire surface absorbs some of the

Figure 10. The coloring agent did not affect the center of this dimple on the treated-color black cultured pearl shown in figure 9. Magnified 30×.



Figure 11. This 16.40-ct sapphire appears to be blue throughout.

dye; we have observed that, in general, even such dimpled areas take on the artificial color when treated. In this treated-color cultured pearl, we saw a white area in the center of the dimple (figure 10), where the stain apparently did not penetrate.

TM

SAPPHIRE, Natural Color with Unusual Fluorescence

When staff at the GIA Gem Trade Laboratory first encountered heat-treated blue sapphires in the late 1970s, many of the stones we examined fluoresced a weak, dull green (often in patches) to short-wave ultraviolet radiation, similar to the fluorescence seen in synthetic blue sapphire. Today we see far fewer heat-treated sapphires with this green fluorescence. This may be due to advances in the techniques and equipment used in heat treatment. Alternatively, it could be because most of the early treated sapphires appeared to be of the Sri Lanka geuda (milky white) type, whereas heat-treated stones seen more recently are from many different localities.

A 16.40-ct emerald-cut stone that we examined in the East Coast lab (figure 11) appeared a nice, relatively even color when viewed face up. When we looked at it from the side, however, we saw that it was predominantly colorless, with only a thin, dark layer of blue at the table (figure 12). Although some areas fluoresced orange to long-wave UV, as is



Figure 12. A side view of the stone in figure 11 reveals that the color is confined to a thin layer under the table. Magnified 10 $\times$.

common in blue sapphires from Sri Lanka, part of the table fluoresced a chalky green (figure 13) to short-wave UV. This led to our initial belief that the stone may have been heat treated.

When we examined the stone with magnification, however, we saw a number of what appeared to be pristine included crystals (figure 14); none showed the stress fractures that would be expected in a heat-treated stone. We then cooled the stone cryogenically and looked at it again under the microscope: Most of the "crystals" were actually negative crystals with two-phase inclusions (see, e.g., figure 15). As the stone warmed in the heat generated by the light of the microscope, the gas bubbles disappeared, indicating that the

Figure 13. The sapphire in figures 11 and 12 displays a chalky green fluorescence that has been considered indicative of heat treatment.

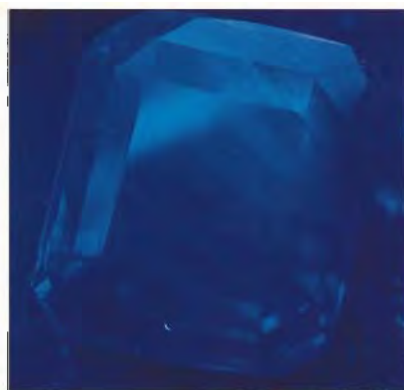


Figure 14. Seen magnified 63 $\times$ at normal room temperature, these "crystal" inclusions in the blue sapphire shown in figures 11–13 lack stress fractures, which is key evidence that the stone had not been heat treated.

two phases were liquid and gaseous carbon dioxide. Because these inclusions formed under high pressure and were still under great pressure within the stone, they would have literally blown up if heated to as little as 270°C (see, e.g., J. I. Koivula's article "Carbon Dioxide Fluid Inclusions as Proof of Natural-Colored Corundum," *Gems & Gemology*, Fall 1986, pp. 152–155). Thus, the presence of these inclusions proved that this stone was never heated anywhere near the 1500°C necessary to produce the blue color artificially. Yet we have no explanation for the chalky green fluorescence.

GRC

SYNTHETIC SAPPHIRE, Color-Zoned

Most flame-fusion synthetic corundums identified by the GIA Gem Trade Laboratory are rather evenly colored. In general, the overall color in those that display curved striae—for example, synthetic ruby and vanadium-doped color-change synthetic sapphire—is very uniform. Those that exhibit curved color banding (e.g., synthetic blue sapphire and synthetic sapphire in the yellow-to-orange color range) typically have

a uniform distribution of the color bands throughout the piece.

A 7.03-ct, modified-oval mixed cut examined at the West Coast lab was an exception to the above. The gemological properties were consistent with corundum. Face up, it appeared to be a uniform yellowish orange. However, when examined from the side, most of the body color—all but a small orange area along the keel line—appeared pink. Only by using a blue color-contrast filter with diffuse transmitted light could we see that the zone along the keel consisted of very weak, diffused, curved orange bands. This curved banding, as well as scattered clouds of minute gas bubbles, proved that it was a synthetic sapphire. RCK

SERPENTINE and FORSTERITE Rock

Although most gem materials consist essentially of one mineral species or gem variety, there are important exceptions among the ornamental gems. Examples include lapis lazuli (a rock consisting of lazurite, calcite, and pyrite), unakite (an epidote-rich granite), and chrome zoisite with ruby inclusions.

Still another example was provided by the opaque, mottled brown-and-white carving of a horse (figure 16) submitted to the West Coast lab.

Figure 15. When the same sapphire was cooled cryogenically and reexamined at 63 $\times$ magnification, it became clear that the "crystals" are actually negative crystals with two-phase CO₂ inclusions.





Figure 16. This 46-mm-long carving was identified as a rock consisting of serpentine and forsterite.

Standard testing revealed an indistinct spot R.I. of 1.55 and an S.G. of 2.45. There was no apparent difference in texture between the brown and white areas.

Subsequently, we took three X-ray diffraction patterns. Two patterns (one each in the brown and white regions) were identified as a mixture of forsterite and serpentine. The third pattern, in the white area, was identified as pure forsterite.

Although alteration can occur between serpentine and forsterite in either direction (see the section on olivine in Deer, Howie, and Zussman's *Introduction to the Rock-Forming Minerals* [Longman Group, London, 1974, pp. 1-8]), the color pattern more closely resembled serpentine altering to forsterite by dehydra-

tion. However, the question of which was the original material could only be answered conclusively with destructive testing. On the basis of the X-ray diffraction analyses, we concluded that this material is a rock consisting of serpentine and forsterite (a member of the olivine group).

RCK and MLJ

Detecting Impregnated TURQUOISE

A great many substances have been used to treat turquoise, including waxes, oils, and plastics. Furthermore, the detection of impregnation in turquoise can sometimes be difficult, often requiring advanced testing techniques. See, for example, related entries in the Fall 1984 and Summer

1986 Gem Trade Lab Notes, and the Spring 1987, Summer 1992, and Summer 1993 Gem News sections.

The West Coast lab last spring had to perform such testing on a semi-translucent to opaque piece of partially polished blue rough that contained significant amounts of a brown and white matrix. Standard gemological testing proved that the blue gem material was turquoise. However, microscopic examination revealed a colorless, transparent substance on much of the stone's surface. Using a commonly employed technique (see, e.g., Lind et al., "The Identification of Turquoise by Infrared Spectroscopy and X-ray Powder Diffraction," *Gems & Gemology*, Fall 1983, pp. 164-168), we submitted a small scraping of the coating to infrared spectroscopy. Comparison with reference spectra showed that the colorless substance was a polymer. The same procedure was then performed on a potassium bromide pellet containing scrapings taken from areas on the turquoise where we did not see any evidence of a polymer. Infrared spectroscopy revealed the polymer in these areas, too. On the basis of these results, we identified the stone as impregnated natural turquoise.

RCK

PHOTO CREDITS

Nicholas DelRe supplied the pictures used in figures 5 and 9-15. Figures 1, 4, and 16 were taken by Maha DeMaggio. The photomicrograph in figure 3 is by John I. Koivula. Shane F. McClure provided figures 2, 6, and 7. Figure 8 is © GIA and Tino Hammid