GEM TRADE LAB NOTES

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CUBIC ZIRCONIA, Carved to Imitate an Ancient Buddha

A transparent brownish yellow carving of Buddha (figure 1) was submitted to the East Coast laboratory, with the statement that it appeared to be a 16th-century piece. At seemingly random sites on the 30.6-mm-high carving were remnants of a yellow metal gilt. In addition, many incised areas contained a foreign material that appeared to be quite worn, implying considerable age.

Routine refractive-index testing gave only a shadowy over-the-limits refractometer reading. The bright orangy red fluorescence to both long- and short-wave ultraviolet radiation was not of much help either. The similarity in color between the carving and the high-lead-content glass hemicylinder of our refractometer prompted us to check each with a hand spectroscope. The absorption spectra were somewhat similar. When a hardness point was applied in an inconspicuous place, the statuette proved to be slightly harder than synthetic spinel (Mohs 8), thus ruling out glass (Mohs 4–6). However, the specific gravity (determined hydrostatically), which was slightly greater than 6.00, pointed toward cubic zirconia (S.G. of 5.800 ± 0.20).

Figure 1. This 64.19-ct yellow Buddha carving (30.6 mm high) is actually cubic zirconia.

Energy-dispersive X-ray fluorescence analysis (EDXRF) by GIA Research revealed the presence of zirconium, as well as hafnium and yttrium, thus confirming the identification as cubic zirconia. This is the first time we have encountered this common diamond simulant as a carved art object. *GRC*

DIAMOND
Cyclotron-Treated

Although neither cyclotron nor radium treatment is now used commercially to color diamonds, enough diamonds were subjected to these surface treatments in the past that we still see them occasionally in the laboratory. Some have features that are unlike those typically seen in diamonds treated by these methods. For instance, figure 2 shows the usual "umbrella" effect seen at the culet of a green, cyclotron-treated, round brilliant-cut diamond. Note the symmetry of the zone of color that produces the diagnostic umbrella. This contrasts with the lack of symmetry of the umbrella seen in the cyclotron-treated round diamond shown in figure 3.

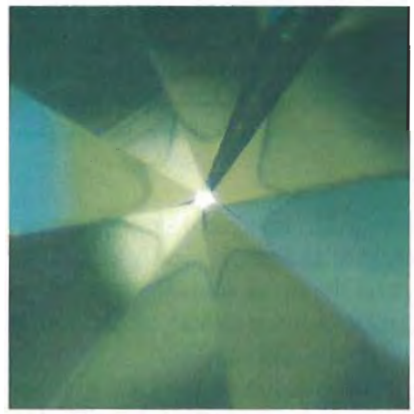

Figure 2. Typically, the umbrella effect seen around the culet of cyclotron-treated round brilliant-cut diamonds is symmetric. Magnified 36×.

An umbrella effect is indicative of the shallow penetration of the green color reaching from the culet to the girdle; typically, the zone of color is uniform around the culet. To try to explain the asymmetric color zone in the diamond in figure 3, we went back to the early literature. In light of the fact that the cyclotron was not

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

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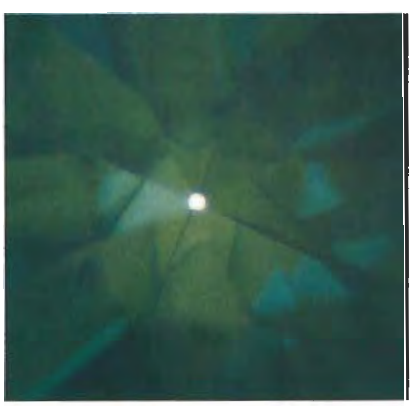
invented until 1931, and there were only three of any size in operation prior to World War II, it was enlightening to learn that the existence of cyclotron coloration of diamond was published as early as 1942, by J. M. Cork (*Physics Review*, Vol. 62: "Induced Color in Crystals by Deuteron Bombardment" [p. 80] and a "Note on Induced Diamonds" [p. 494]). By 1949, such treated stones were appearing in the gem market and the gemological literature. In "Cyclotron Treated Diamonds" (*The Gemmologist*, March 1949), A. E. Alexander observed that the green color appeared to be confined to the crown areas of one stone, an emerald cut, with the pavilion being essentially colorless (evidence of treatment from the top, which was uncommon, rather than from the culet).

Martin L. Ehrmann wrote one of the first good accounts of the history of cyclotron treatment, in the Summer 1950 issue of *Gems & Gemology*. Less than a year later, in the Spring 1951 issue of *Gems & Gemology*, F. H. Pough and A. A. Schulke provided clues to identifying cyclotron treatment in "The Recognition of Surface Irradiated Diamonds." This article gave additional historical background, as well as notes about the different cyclotrons used and methods of holding stones during treatment. Here we find a clue to the possible cause of the asymmetric umbrella seen, in the authors' statement that their first commercial work was with an older cyclotron at Columbia University. This unit did not allow the beam to strike the stones squarely on the culet, so the treater either had to rotate the stone 180° after the first exposure and treat it a second time, or hope for the best with a single exposure. By the time this article was printed, the site for cyclotron treatment had been moved to a much newer and more versatile cyclotron at George Washington University in St. Louis, Missouri.

Therefore, the stone with the asymmetric umbrella in figure 3 could be one treated with the old Columbia University cyclotron, if it was not simply misaligned in a more recent procedure. *GRC*

Figure 3. In this 0.52-ct cyclotron-treated round brilliant-cut diamond, the "umbrella" effect is unusual in its asymmetry. Magnified 29×.



Fancy-Color Diamond with Unusual Radiation Stains

Natural radiation stains occur on diamonds where they have been in contact with radioactive solutions or solids. On fashioned diamonds, they are most often found on naturals on the girdle, but they also appear in cavities and surface-reaching fractures; less commonly, remnants of such staining have been noted on culets and facets where very little of the skin of the rough diamond has been removed (see, e.g., Spring and Winter 1981 and Fall 1986 Lab Notes).

Recently, the West Coast lab examined a 2.37-ct fancy dark yellowish brown diamond with an unusual internal scene. This included a tapered etch tube, with a hexagonal cross-section, that ran from the pavilion area just below the girdle to the center of the stone. The etch tube had a series of distinct brown radiation stains along its length (figure 4), including one at the very bottom; this created the unusual situation of a surface-related radiation stain in the heart of the stone. Additional radiation stains were noted on the adjacent girdle area and on an upper girdle facet. Another notable inclusion was a cloud of minute white particles throughout the stone that formed a pattern reminiscent of a four-leaf clover. There was also green graining parallel to the "leaves" of this pattern, which is typical of hydrogen-rich diamonds. The absence of any of the absorption features associated with laboratory-induced radiation led us to conclude that the diamond was of natural color. *RCK*

Figure 4. A series of brown radiation stains were noted along the length of this etch tube in a 2.37-ct fancy dark yellowish brown diamond. Magnified 35×.

Unusual "Melted" Cavity in a Diamond

A 0.50-ct slightly grayish green, triangle-shaped, brilliant-cut diamond was submitted to the West Coast lab for a grading report. In addition to numerous brown radiation stains, like those commonly seen on naturals and occasionally in etch channels (as described in the preceding entry), we observed one large brown radiation stain inside a cavity on a crown facet near the faceted girdle.

When we examined the cavity with magnification, we were able to resolve the striated, step-like formation that is characteristic of etch

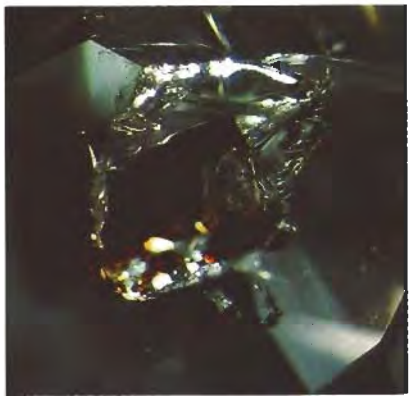


Figure 5. Note the unusual glassy, "melted" appearance on the edge of this cavity in a 0.50-ct diamond. Magnified 30×.

channels. However, we also noted a smooth surface around the edge of the cavity (figure 5) that gave it the appearance of melted glass. Although seen in the Gem Trade Laboratory only rarely, similar features in diamond were described by Yu. L. Orlov in his *Mineralogy of the Diamond* (John Wiley & Sons, New York, 1977 [transl.]). *KH*

Faceted Yellow SYNTHETIC DIAMOND

The Fall 1993 Lab Notes section described a 0.74-ct orangy yellow crystal submitted to the East Coast lab for identification. That specimen exhibited features that clearly identified it as a synthetic diamond and suggested that it might be of Russian origin.

More recently, a 0.34-ct square modified brilliant cut that also had an orangy yellow body color was submitted to the East Coast lab for routine examination. When exposed to an intense beam of white light, the stone luminesced a weak to moderate green. To long-wave U.V. radiation, it fluoresced a zoned, slightly chalky, moderate-to-strong orange; the zones were defined by two narrow, intersecting cross-like "arms" of greenish yellow fluorescence that extended diagonally from the girdle edges. The reaction to short-wave U.V. radiation was similar, but only slightly stronger. Most of the yellow synthetic diamonds we have examined to date have had a significantly stronger reaction to short-wave than to long-wave U.V. radiation. There was no phosphorescence to either wavelength.

Examination with a microscope revealed distinct color zoning. Darker zones set off a lighter yellow central funnel-shaped "core," with these two zones sharply delineated by a very thin rim of an even darker yellow. Table-down, the stone revealed some near-colorless zones near the center and at the corners. Magnification also showed a cloud of reflective pinpoint inclusions throughout the stone, with the heaviest concentration just outside the central, lightest-colored zone. Although we saw drag lines on the table from where these pinpoints broke the surface, we saw no surface graining. We also noted one small crystal and several elongated black inclusions, mostly located at the corners. Remnants of the original crystal surface on two opposing corners indicated that the stone had been cut for maximum size.

We noted weak anomalous birefringence when we examined the stone between crossed polarizers. The stone was attracted by, but did not attach to, a pocket magnet. Infrared spectroscopy showed the diamond to be a mixture of types Ib and IaA.

We subsequently received for examination two crystals and two cut stones, all brownish yellow to brown, that were reportedly obtained from Chatham Created Gems, Inc. Their characteristics indicate that they are from the same source as the synthetic stones described above and the known Russian synthetic diamonds reported in the article by Shigley et al. in this issue.

RCK, TM, and Emmanuel Fritsch

EMERALD, with Large Filled Etch Channels

The GIA Gem Trade Laboratory routinely identifies emeralds that show evidence of clarity enhancement, their surface-reaching fractures filled with transparent, essentially colorless substances. Because these filled fractures are usually very thin, any gas bubbles they contain are generally flat, appearing as highly reflective areas when examined with oblique illumination.

Less frequently, we examine emeralds in which one or more large, cross-sectional areas have been filled. In one such stone—an 8.02-ct round mixed cut—submitted for identification to the West Coast lab, magnification revealed three large, and several smaller, etch channels that had been filled and apparently sealed at the surface. However, they were not completely sealed, as the heat from the microscope's darkfield lamp caused a small amount of the filler to "sweat" out of the larger channels. That the filler below the hardened surface was still a fluid was further confirmed by the presence of large, spherical gas bubbles (see, e.g., figure 6), which moved when the stone was rocked on the microscope stage. These observations, along with the presence of orange "flash effects" from the filled fractures, indicated that the stone had been treated with a synthetic resin and an attempt had been made to polymerize the surface. *RCK*

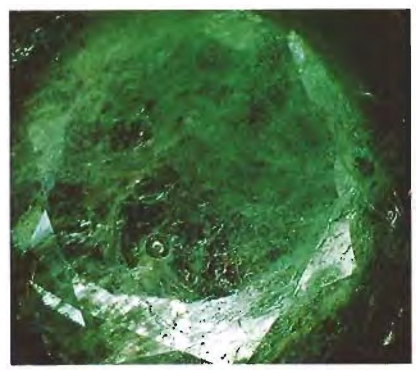


Figure 6. Note the large gas bubble in the filling material that was used to clarity enhance this 8.02-ct emerald. Magnified 9×.

GARNETS, Fracture Filled

Although much has been written about fracture filling in diamonds and emeralds, the lab periodically sees other stones that have been similarly treated. Just in the past year or so, these have included amethyst, aquamarine, chrysoberyl, sapphire, spinel, tanzanite, and tourmaline.

Twice in about a week this past summer, we found evidence of clarity enhancement in garnets submitted to the West Coast lab for identification. In the first instance, two hessonites contained low-relief fillings that had some flattened gas bubbles. In the second, a pyrope-almandine had a filled break that exhibited very low relief (figure 7). We did not see dispersive flash effects in either case. As is sometimes required with filled breaks, we confirmed the treatment by carefully holding the tip of a thermal reaction tester ("hot point") just above the area where the fractures reached the surface, causing movement within the filling material. (This test should only be performed with extreme caution, as some gem materials are very susceptible to heat damage.)

Readers are encouraged to check all gems for such clarity enhancement. Those with surface-reaching breaks that appear to have lower-than-normal relief are most suspect.

RCK and SFM

Figure 7. This low-relief filled fracture is in a 17.93-ct pyrope-almandine garnet. Magnified 10×.

Iridescent ORTHOAMPHIBOLE, "Nuummite"

After reading the article describing a new iridescent orthoamphibole from Greenland (*Gems & Gemology*, Spring 1987, pp. 36–42), we wondered how long it would be before examples of this material came through the laboratory. In September 1993, a client brought an attractive necklace of 18.00 × 13.00 mm flattened oval beads (figure 8) into the East Coast lab for examination, saying that the customer to whom they belonged was unsure of the name of the material. She did know that it was from Greenland and that the name sounded like "neumit" or "newmite." We recalled the 1987 article and the fact that the material was being sold under the trade name "Nuummite" (because the deposits were found within about 50 km of the city of Nuuk). The gemological properties listed in the article (average R.I. of 1.64–1.66, average S.G. of 3.24), along with the appearance of the polished stones, made identification fairly easy. We also determined that the beads were attracted to a small magnet, a property not mentioned in the 1987 article.

GRC

Figure 8. The beads (approximately 13 × 18 mm) in this necklace were identified as the iridescent orthoamphibole known in the trade as "Nuummite."

ORTHOPYROXENE, a Carved Mask

Although most of the items submitted to the laboratory are faceted or cabochon cut for jewelry use, we occasionally receive larger items to examine, including carvings. Recently, the opaque, mottled brown-and-white mask of a male face shown in figure 9 was submitted to the West Coast lab for identification. As is often the case with such items, there were limitations on the gemological testing that could be done. The size of the piece, roughly 30.2 × 25.8 × 9.0 cm, prevented determination of specific gravity by normal methods, and the condition of the surface made it impossible to obtain a clear R.I. reading.

Therefore, we performed X-ray powder diffraction analyses on small scrapings taken from three locations on the mask's base. Although the three patterns differed slightly from one another, they all closely resembled those of the orthopyroxenes enstatite and hypersthene. Because none of the patterns was an exact match for any one mineral—and, as the testing indicated, there was some variation in the structure from one area to another—the life-size mask was identified as a rock consisting of one or more minerals in the orthopyroxene group and possibly additional unidentified minerals. Petrographic testing, which is destructive, would be necessary to characterize the material fully.

RCK

Some PEARL Observations

A multi-strand pearl necklace submitted to the East Coast lab was found to be composed of both natural and cultured pearls, a situation that frequently calls for many X-radiographs and tedious examination of every pearl. This necklace was no exception. The surfaces of nearly one-third of the natural pearls on one strand, which consisted mostly of

Figure 9. This life-size carved mask (30.2 × 25.8 × 9.0 cm) is a rock consisting of one or more orthopyroxenes and possibly additional minerals.

natural pearls, had a reticulated appearance that we had never before encountered (figure 10). Many of these pearls were noticeably yellow. Most of the other strands, nine in all, had from one to five natural pearls with the same strange appearance.

At first we thought this unusual surface might be a form of the "hammered" appearance seen often on natural pearls but only very rarely on cultured pearls. However, a typical hammered effect appears to have been done with a flat hammer head (see figure 11), whereas the pearls in question appeared to have been hammered with a ball peen. Since there were several of these "peened" pearls, and almost all were yellower than the bulk of the other pearls, they may all originally have been strung on the same necklace, with some sort of damage responsible for the unusual appearance.

Figure 10. The surface of this 6.5-mm natural pearl has an unusual pattern, as if it had been hammered with a ball peen. Magnified 14×.

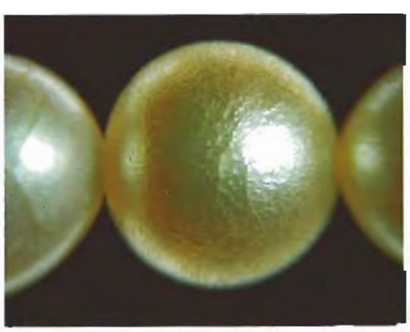
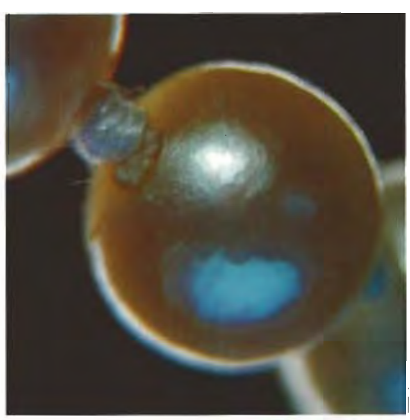


Figure 11. The "hammered" appearance typically found on natural pearls appears to have been done with a flat hammer head. Magnified 17×.



One pearl in the multi-strand necklace had some areas of "normal" hammering (again, see figure 11). In addition, the drill hole was plugged with a small wooden dowel. Such dowels are seen in older natural pearls that have enlarged drill holes, probably due to skin acid on the string.

Our heightened awareness of pearl surfaces led us to note still another surface anomaly, this time on several pearls in a cultured pearl necklace. Here, the surface appeared reticulated, but in a somewhat parallel pattern. As with the dimpled black cultured pearls described in the Summer 1993 Gem Trade Lab Notes (pp. 127–128), these unusual surfaces gave the pearls an added element of charm. GRC

SYNTHETIC RUBY, Striae Resolution Technique

The West Coast lab was asked to identify two specimens that had been represented to our client as natural ruby rough from Vietnam. The larger (25.71 ct) of these was relatively easy to identify, inasmuch as curved striae were clearly visible through a "window" that had been polished on one surface.

The second specimen (18.45 ct) presented more of a challenge, since the entire piece exhibited a rough, abraded appearance, with no polished surface. Examination with the microscope—using darkfield illumination (both with and without immersion), brightfield, and shadowing—failed to resolve any growth features. Using a method that is common among European gemologists, we next immersed the specimen in methylene iodide and then examined it with brightfield illumination. Curved striae were easily resolved (figure 12).

Further experimentation with this microscope technique showed that it was often quite helpful in

Figure 12. The curved striae in this 18.45-ct synthetic ruby, which was misrepresented as natural Vietnamese rough, became readily apparent when the stone was examined using combined brightfield illumination and immersion. Magnified 10×.

resolving growth features in melt synthetics. In one test, we examined 10 boule fragments of synthetic ruby and synthetic pink sapphire using a variety of lighting techniques. The brightfield/immersion combination enabled us to see curved striae in six of the 10 specimens; using any other technique, we saw curved striae in only one specimen. Furthermore, the addition of a transparent green plastic filter (for color contrast) between the microscope objectives and the stone increased the relief of the striae. This is comparable to the technique of using a blue filter to resolve striae in yellow-to-orange synthetic corundum. We have since used the brightfield/immersion combination to examine Czochralski-pulled synthetic alexandrites, with some promising results.

Dino DeGhionno and RCK

SAPPHIRE
A Difficult Identification

In the routine identification of blue sapphires, our experience has been that the larger the stone is, the easier the identification will be, with tiny calibre-cut set stones often the most time consuming. Therefore, we were surprised at the problems we encountered identifying a 25+ ct cushion-shaped mixed-cut sapphire that was set in a ring. Initially confident that magnification would quickly establish whether the stone was natural or synthetic, we became concerned when we could not detect any inclusions or color banding. The setting precluded a satisfactory Plato test, which we attempted after exposure to short-wave U.V. radiation revealed a chalky green fluorescence in what appeared to be synthesis-associated curved bands (figure 13). In our experience, however, very large synthetic blue sapphires are almost always watery in appearance, with color stronger in one area than another. This stone was uniformly colored, with no watery appearance. The possibilities were that it was either an unusual synthetic or an early heat-treated natural stone. To no avail, we checked with the hand spectroscope for an iron line, which when present in heat-treated blue sapphires is usually weak. We finally asked the client to remove the stone from the ring.

Figure 13. The fluorescent bands—actually straight parallel growth planes—in this 25+ ct natural sapphire could easily be misconstrued to be curved.

Figure 14. When the stone in figure 13 was removed from its mounting, immersion revealed the angular nature of the color zones.

What we saw with the stone loose was diagnostic. Formerly hidden by the setting, but now faintly visible, was a small patch of altered silk. What had appeared under short-wave to be curved fluorescent bands now, with immersion, could be seen to be normal parallel growth planes in a natural sapphire (figure 14).

GRC

Diffusion Treatment Obscured by Mounting

Recently, the West Coast lab received for identification a 1.14-ct transparent blue, oval mixed cut set in a woman's yellow metal ring. Gemological testing revealed properties consistent with corundum. Although the nature of the mounting severely limited microscopic examination, we did resolve diffused straight color banding and broken "silk" through an exposed area near the culet. All of these observations pointed to an identification of natural sapphire that had been heat treated.

However, because of the possibility of diffusion treatment, we routinely examine all rubies and sapphires with immersion. With the stone in question, immersion revealed a few facets, one row up from the culet, that appeared to lack color. Again, because of the nature of the mounting, this could have been an optical anomaly. We advised the client accordingly, and he agreed to remove the stone from the ring so we could examine it further.

As can be seen in figure 15, the sapphire was, in fact, diffusion treated. This should serve as an excellent

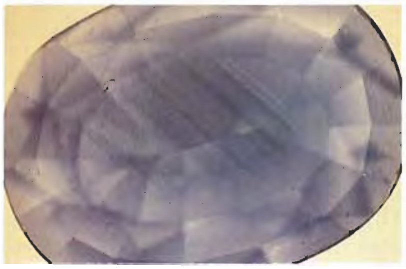


Figure 15. The darker color evident at the facet junctions, and uneven coloration of the different facets, only became apparent when this 1.14-ct diffusion-treated sapphire was removed from its mounting. Magnified 18×.

Figure 16. Prominent, slightly curved parallel graining is evident in this 15.45-ct green YAG. Magnified 17×.

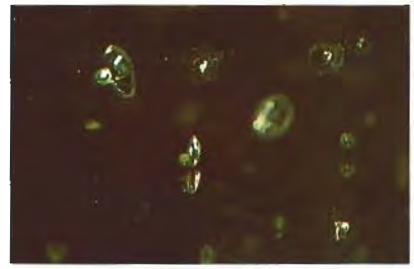


Figure 17. These included crystals, with surrounding disk-like fractures, are unusual in YAG. Magnified 38×.

reminder that the presence of features consistent with high-temperature treatment does not rule out diffusion treatment, as this enhancement technique also employs heating to a high temperature.

RCK and Dino DeGhionno

YAG with Unusual Inclusions

Yttrium aluminum garnet (YAG) debuted in the gem trade in colorless form as a diamond simulant, but it is available today in many colors, including violet, dark blue, greenish blue, green, and yellow. Until recently, most of the YAGs we saw were essentially inclusion-free. Lately, however, we have seen a greater number of included specimens. One such example was a 15.45-ct dark green rectangular emerald cut (13.91 × 12.08 × 7.70 mm) examined in the West Coast lab. Gemological testing identified the specimen as YAG; EDXRF analysis further confirmed both the identity and the presence of chromium as the principal coloring agent. Magnification revealed an unusual inclusion scene: elongated gas bubbles sheathed in fine layers of blue coloration; distinct, slightly curved parallel graining (figure 16); and scattered small, unidentified crystals, each surrounded by stress fractures. The last (figure 17) were reminiscent of the discoid fractures noted in heat-treated corundums.

The colored YAG that first entered the gem industry was most likely material rejected for optical (e.g., laser) applications because of structural defects (for more information, see K. Nassau's *Gems Made by Man*, Chilton Book Co., Radnor, PA, 1980). Now, however, YAG is also produced specifically for gem use. Perhaps lower purity tolerances in the "gem" production process account for the included materials we have been seeing. Or this may be material currently produced in Russia by a floating zone technique referred to as "horizontal crystallization," which we have observed to be fairly heavily included.

RCK and SFM

PHOTO CREDITS

Nicholas DelRe supplied the photos used in figures 1–3, 8, 10, 11, and 13–15. Photomicrographs in figures 4 and 5 are by John I. Koivula. The photos used in figures 6 and 9 were taken by Maha DeMaggio. Shane McClure provided figures 7, 12, 16, and 17.