

GEM TRADE LAB NOTES

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Treated AMBER

Occasionally the laboratory embarks on detective work beyond the straight identification of an item. For example, a client submitted two amber cabochons to the East Coast lab, with the request that we determine why the paler of the two had faded when it was mounted and displayed in a brightly lit showcase. The client reported that the "faded" sample was from the same parcel as the darker one and was originally the same color (figure 1).

In the process of confirming that the samples were amber, we noticed an unusual orange fluorescence and the presence of a layer of minute gas bubbles in swirls just beneath the sur-

face on both pieces (see, e.g., figure 2). In the April 1986 *Journal of Gemmology*, Kenneth Scarratt reported on amber with a dark surface color that showed similar (but straighter) strings of gas bubbles; removal of part of the surface revealed that it was actually a very shallow layer over a very pale core.

The prominent stress fractures ("sun spangles") in the faded stone are undoubtedly due to exposure to heat. It is known that cloudy amber can be clarified by gradual heating—in 50° increments—to approximately 200°C (see, e.g., Kurt Nassau, *Gemstone Enhancements*, Butterworth's, 1984). It appears that, during heat treatment, the gas bubbles that cause the cloudiness migrate to the surface, the

Figure 1. These two clarified amber cabochons (each about 25 × 18 mm, 10.50 ct) were from the same lot and, originally, both the same color. The paler specimen (right) had faded over time while displayed in a brightly lit case.



Figure 2. This veil-like plane of minute bubbles was seen just beneath the surface of the faded cabochon in figure 1. Magnified 10x.

surface darkens, and the color in the center may be almost totally lost. Presumably, the darker brown surface color is due to oxidation caused by the heating. However, the finished product usually has a dull, chalky green (rather than orange) fluorescence to long-wave ultraviolet radiation.

Ken Scarratt has reported (pers. comm., 1993) that this clarification process may be carried out with the stones heated in what he calls "sump oil" (old crankcase oil). With no specimens known to be treated in this fashion available for study, however, we have not been able to investigate this possibility.

Nassau and others also state that

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

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Figure 3. When the brown "skin" was removed from one end of an amber sample from the same lot as the stones shown in figure 1, a near-colorless center was exposed.

amber may be surface colored with organic dyes, and these may fade. However, the detection of an organic dye on an organic substrate, such as amber, commonly requires other than routine gemological procedures and so was beyond the scope of this investigation.

The client subsequently provided us with several more cabochons, all of which had the swirled layer of tiny gas bubbles and fluoresced the unusual orange color noted above. The client also gave us permission to subject the specimens to any tests we felt appropriate. First, we removed a portion of the "skin" from one end of one of them, which revealed that the material underneath is almost colorless (figure 3). Next, we cut another sample in two and placed one half in

Figure 4. Originally, both portions of this 25×18 mm cabochon were dark. The section on the right faded when it was exposed to the light from a 14-watt Tensor lamp for approximately 170 hours at close range.



the dark while we left the other approximately 1.5 cm from the bulb of an illuminated 14-watt Tensor lamp for seven days. At the end of that time, the exposed piece had become markedly paler (figure 4). Our work on these two stones confirmed that the fading was probably due to exposure to intense light. GRC

had previously been detected in diamond fillings (see J. Koivula et al., "The Characteristics and Identification of Filled Diamonds," in the Summer 1989 issue of *Gems & Gemology*). X-radiography further confirmed the presence of the filling in the form of white, X-ray-opaque areas on the X-radiograph.

It is important to reiterate that, although the diamond contained

DIAMOND

Extensive, Subtle Fracture Filling in a Diamond

Preliminary examination of a 0.88-ct heart-shaped brilliant submitted to the West Coast lab revealed what appeared to be a filled diamond with extremely low relief "fingerprint" inclusions containing minute voids. Because the GIA Gem Trade Laboratory does not issue grading reports on filled diamonds, the stone was referred to the Identification and Research Department for additional testing and issuance of an identification report.

Further examination with magnification using standard darkfield illumination revealed several transparent, colorless, filled fractures that contained the minute voids mentioned above and showed a very subtle orange-to-blue flash effect. The flash effect in these fractures was particularly difficult to detect because the fractures lay at very shallow angles to the surface of the diamond.

The treatment became more apparent (figure 5) when a pinpoint fiber-optic illuminator was used. This lighting technique revealed the extent of the filled breaks, which included one very large fracture beneath, and nearly parallel to, the table. The intense illumination also made the flash effects significantly more noticeable and revealed hairline fractures in the filling material. With transmitted light, the outlines of the filled areas were easier to detect when a single polarizing filter was placed between the microscope's objective and the diamond.

Qualitative chemical analysis performed by GIA Research, using energy-dispersive X-ray fluorescence (EDXRF), detected lead. This element

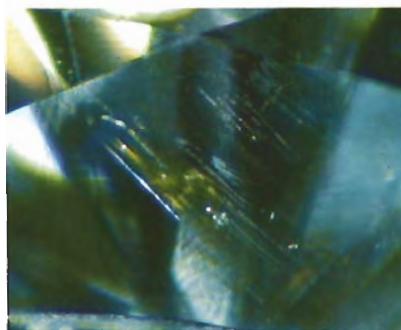


Figure 5. The orange and blue flash effects are very subtle in this extensively filled diamond. Fiber-optic illumination; magnified 40x.

numerous filled fractures, the diagnostic microscopic features were quite subtle. These might have been easily overlooked if only darkfield illumination had been used. It is therefore prudent to use additional lighting techniques, including pinpoint fiber-optic illumination and polarized light, in all cases where fracture filling is suspected.

RCK and SFM

Iridescent "Dislocation" in a Diamond

In our experience, iridescence in diamond is almost always associated with very fine fractures. It was thus a pleasant surprise to encounter the unusual internal scene shown in figure 6 in a 1.60-ct round brilliant submitted to the West Coast lab for grading. Here, iridescence revealed a thin-film separation along what appeared to be a "V"-shaped dislocation that reminded us of a "Stealth" fighter aircraft. A visual estimation of the angle

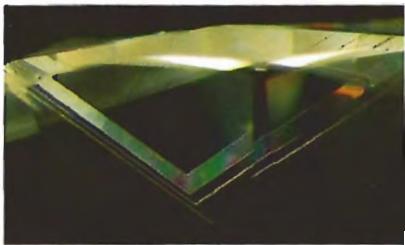


Figure 6. Iridescence can be seen in this "V"-shaped thin film in a diamond. Magnified 30x.



Figure 7. When exposed to long-wave U.V. radiation, several of the stones in this pendant exhibited the blue fluorescence typical of diamond.

led us to believe that the thin film follows a pair of possible dodecahedral planes. The iridescence, visible at some orientations, is apparently due to the narrowness of the separation.

RCK and John I. Koivula

Mounted Diamonds Mistaken for Simulants

The Fall 1991 Gem News section contained an entry on small diamonds set in pendants by means of a transparent, colorless, carbon-based polymer. It was noted that small stones so mounted can give false "simulant" readings on thermal conductivity meters. Other tests, such as magnification, were recommended in such instances.

Recently, the West Coast lab was asked to examine one of these pendants after a jeweler had told the client that the stones were imitations. Testing of one randomly selected stone revealed properties diagnostic of diamond, including typical microscopic features. We also resolved 415.5-nm absorption lines in the spectra of some of the stones, and a number of them fluoresced blue to long-wave U.V. radiation (figure 7), a behavior that is quite typical of diamond, but not of its simulants. Therefore, we concluded that the stones were indeed diamonds.

RCK and Cheryl Wentzell

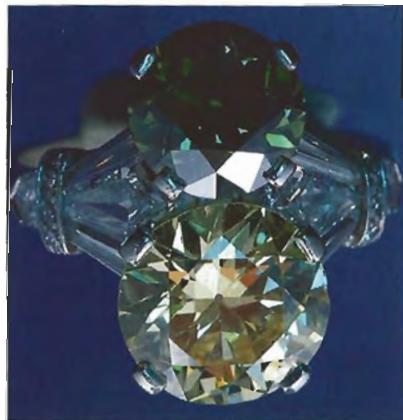
Treated Green Diamond

Before beginning some alterations on the diamond-set white-metal ring shown in figure 8, a local jeweler sub-

mitted the piece to the East Coast laboratory for a report on the origin of color of the green stone. The jeweler's client had indicated that the ring was purchased from a prominent New York City jewelry firm in the 1930s and had been in the family ever since.

Treatments that alter the color of gem diamonds have been known since 1906, and treatment with radioactive compounds became commercial in 1915. Other methods of radiation treatment did not become common until after 1946. However,

Figure 8. The green diamond in this ring measures approximately 8.5–8.6 mm in diameter by 5.3 mm deep and owes its color to a surface treatment with a radioactive compound.

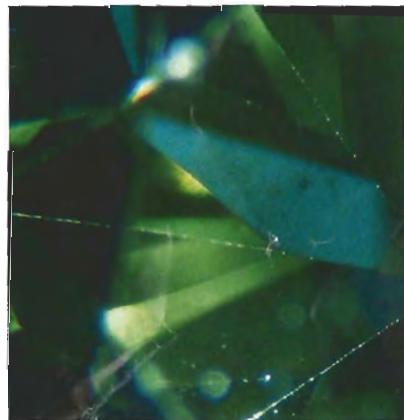


examination of this green diamond with a binocular microscope revealed numerous brown spots and patches on the polished surfaces, especially on the pavilion (figure 9). A distribution of green spots and patches frequently results from surface treatment with a radioactive compound, a process that leaves the diamond itself radioactive. When checked with a hand-held survey meter, the stone exhibited significant radioactivity, with a maximum reading of 42 mR/hour, thus confirming our suspicion of treated color.

Subsequent radionuclide testing at the West Coast lab revealed the characteristic gamma-ray signature of lead-210 (Pb-210) in this green diamond. We know that if lead-210 is present, its radioactive daughter nuclides, bismuth-210 (Bi-210) and polonium-210 (Po-210), must also be present; however, only Pb-210 releases enough gamma rays during decay to be measured quantitatively in our laboratory.

Pb-210 and Bi-210 both decay by emission of beta particles, and Po-210 decays by emission of alpha particles. The penetration of alpha and beta particles is very shallow in diamond—only about 0.01 mm and 1

Figure 9. The patchy brown radiation stains on the pavilion of the green diamond shown in figure 8, seen here through the table, were probably caused by heating after the radiation treatment. Magnified 21x.



mm, respectively. The surface stains are one result of this shallow penetration; another is the fact that most of the green color is confined to a layer at, or just below, the surface of the stone. The fact that the surface stains appear brown here, rather than green, indicates that the stone was heated to 550°–600°C at some point after it was irradiated.

These radionuclides occur naturally as part of the uranium-238 (U-238) decay chain. A quantity of Pb-210 and Bi-210, when separated from radium (or uranium), has been known traditionally in the nuclear industry as a "Radium D+E source"; it was once employed as a high-energy beta radiation source for instrument calibration but is rarely, if ever, now used.

Pb-210 is subject to regulation whenever it is separated chemically from uranium ore by artificial means. In the case of this green diamond, the 75 nanocuries of Pb-210 measured in this 2.34-ct (0.468 gram) stone greatly exceeded the U.S. Nuclear Regulatory Commission's exempt concentration limit of 0.001 nanocuries per gram. However, because it was below the exempt quantity limit of 100 nanocuries, we returned it to our client with full disclosure as to what radionuclides are present and in what quantities. Inasmuch as the half-life of Pb-210 is 22.3 years, this treated green diamond would not be legal to sell, or trade, in the United States until around the year 2192. The report also gave the surface dose rate—that is, the amount of radiation someone wearing the ring would receive. In the absence of other exposure, this ring could be worn 357 hours a year without exceeding the U.S. federal recommendation for radiation exposure to the hands for the general public.

Ilene Reinitz and
Charles E. Ashbaugh

EMERALD

An Ancient Miniature Carving

On occasion, common gem materials provide challenging identifications due to their surface condition or the

form in which they are fashioned (e.g., carvings). The miniature bust in figure 10 (22.1 × 14.1 mm; 25.36 ct) was submitted to the East Coast lab with an important provenance: It was reported to depict Roman Emperor Nero at approximately 11–13 years of age (49–51 A.D.) and to have been carved during that era.

Because the carving is so intricate, we could not establish the refractive index. Using the hydrostatic method, we determined that the specific gravity was 2.70. Although not strong, the spectrum revealed the chromium absorption typical of emerald. The carving also showed a weak pink color—a reaction seen in natural emeralds from a variety of sources—when examined with the Chelsea color filter. The diaphaneity was semitransparent to translucent and, using fiber-optic illumination, we saw numerous fluid inclusions as well as several small crystals that we could not conclusively identify (although we believe that at least one,

which had a cleaved section that reached the surface on the side of the "head," is biotite mica). Most of the numerous surface-reaching fissures had yellowish brown staining, probably residue of iron compounds. No evidence of "oiling" was present. From the properties, we concluded that the specimen is natural emerald.

If the provenance of the sculpture is genuine, the emerald is most likely from Egypt. According to John Sinkankas in *Emerald and Other Beryls* (Chilton Book Co., Radnor, PA, 1981), the Egyptian mines were worked extensively during the Graeco-Roman period and beyond, from roughly 330 B.C. to about 1237 A.D. In 1991, Robert C. Kammerling, of the GIA Gem Trade Laboratory, visited a number of the ancient emerald mines in Egypt and obtained samples locally. He reports that the material he obtained is very similar in color and transparency to this carving and shows the same turbidity of fluid inclusions, stained fractures, and biotite-mica crystals (see the article by Jennings et al. in this issue of *Gems & Gemology*). TM

Figure 10. This 22.1-mm-high emerald carving is purported to be of Roman Emperor Nero and to date from approximately the first century A.D.



EUCLASE, Colored by Chromium

Color is the first clue in the identification of a gemstone, and normally suggests a number of possibilities. These can then be narrowed down on the basis of further testing. If the gem falls outside its usual color range, however, the final identity can be rather surprising. This was the case when the East Coast lab identified the 1.85-ct, vividly colored, greenish blue, square-emerald-cut stone shown in figure 11 as euclase. Gem-quality euclase is usually colorless, pale blue to green (from Brazil), or sometimes very dark blue (from Zimbabwe).

Although the refractive indices and birefringence of this sample were consistent with published values for euclase, the specific gravity (at 3.14) was slightly higher than the usual range of 3.00 to 3.12. In addition, the

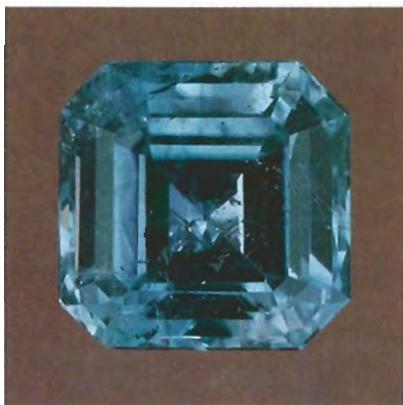


Figure 11. This unusual 1.85-ct greenish blue euclase was found to be colored by chromium.

trichroic colors of purple, blue-green, and colorless were different from the bluish gray, light blue, and colorless usually seen in the pale blue material. Unlike the pale material, which shows no reaction to the Chelsea color filter, this stone appeared red. Like the pale material, however, this stone was inert to both wavelengths of ultraviolet radiation. The stone displayed absorption lines in the hand spectroscope, with a doublet centered at about 460 nm and a sharp line at about 680 nm, as previously reported by B. W. Anderson (*The Gemmologist*, Vol. 24, No. 283, 1955, p. 31).

Because we thought that artificial irradiation was one possible cause of the unusual color, we sent the stone to our West Coast radiation-testing facility. However, no residual radioactivity was detected, thereby eliminating the possibility of recent irradiation in a nuclear reactor, and diminishing the possibility of high-energy electron irradiation.

The origin of color in dark blue euclase from the Miami area of Zimbabwe was assigned to the Fe^{2+} - Fe^{3+} charge transfer by S. M. Mattson and G. R. Rossman (*Physics and Chemistry of Minerals*, Vol. 14, 1987, pp. 94-99). Euclase colored by this process exhibits a broad peak at 670 nm, and peaks at 860 and 1250 nm. E. Gübelin (*Gems & Gemology*, Winter 1978-79, pp. 104-110) attributed the

various other hues (light blue, green, deep yellowish green, and greenish blue) of euclase to various states of iron oxidation, although Anderson had attributed blue in euclase to chromium on the basis of gemological spectroscopy ("The spectroscope and its application to gemology," Parts 10 to 17, *The Gemmologist*, Vol. 23, Nos. 275-282, 1954-55).

Qualitative energy-dispersive X-ray fluorescence spectrometry performed at GIA Research showed this stone to contain significant amounts of chromium and vanadium, but only trace amounts of iron and titanium. An ultraviolet-visible absorption spectrum of this dark greenish blue euclase (recorded in a random orientation—the top graph in figure 12) shows two broad bands centered at approximately 405 and 605 nm, and weak bands at about 468, 653, 685, 705, and 755 nm. The bands at 468 and 685 correspond to those estimated at 460 and 680 with the handheld spectroscope. The general shape of the spectrum and the position of the weak bands suggest absorption caused by Cr^{3+} . The lower three graphs in figure 12 show the polarized

absorption spectra of a very light bluish green euclase from Villa Roca, in Minas Gerais, Brazil, that was colored by Cr^{3+} . These spectra were provided for comparison purposes by Dr. George R. Rossman of the California Institute of Technology, Pasadena, California. The similarity of the absorption features is striking, and the spectrum of the euclase submitted to us can easily be interpreted as being a combination of the three polarized absorption spectra of the comparison stone. It is uncertain whether or not vanadium contributes to the absorption.

We concluded that the unusual, highly saturated greenish blue color of this 1.85-ct stone is natural and due to chromium, rather than iron as was previously established for other dark blue euclases. The pleochroism of chromian euclase explains why its faceup color varies from greenish blue to bluish green (and possibly purple), depending on the crystallographic orientation of the rough relative to the gem's table.

TM, Emmanuel Fritsch,
Meredith Mercer, and
Ilene Reinitz

Figure 12. The U.V.-visible absorption spectrum at the top is of the 1.85-ct dark greenish blue euclase in figure 11, taken in a random orientation. The three spectra below it are of a very light bluish green euclase, known to be colored by chromium, taken in the three crystallographic directions.

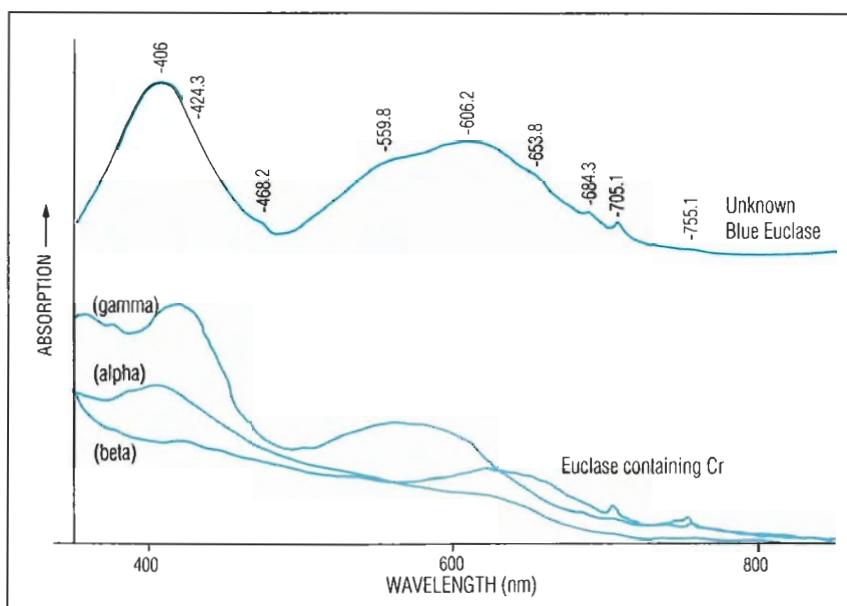




Figure 13. This suite of jewelry, set with diamonds and cabochons of odontolite, turquoise, and at least one glass imitation, is in a style typical of the mid-19th century. The center brooch measures approximately 6 × 6 cm.

ODONTOLITE

One of the joys for a gemologist is the occasional encounter with a gem material that he or she has read about in textbooks, but has rarely seen. Therefore, it was quite rewarding to be able to identify, with some assistance from GIA Research, the material known as odontolite, or "bone turquoise," among the cabochons in the suite shown in figure 13. Although this writer had seen one some 40 years ago, while studying rare gem materials in museum collections, he had never seen it set in jewelry. The suite of silver jewelry in figure 13, which also contained rose-cut diamonds as well as at least one glass imitation and a number of turquoise cabochons, appears to be in a style that was popular in the mid-19th century.

As the name "bone turquoise"

suggests, odontolite is basically a fossilized organic material—usually the tusks of mammoths or mastodons. The organic material (ivory) is largely replaced by minerals—carbonates, phosphates, or both. Generally, it is green, colored by inclusions of the mineral vivianite, but these cabochons were distinctly blue.

Although the R.I. of 1.61 matched that of turquoise, several other features suggested that some of these samples were not turquoise: their translucency, the parallel banded structure (figure 14), and the lack of a turquoise spectrum in the hand spectroscope.

To determine whether a carbonate was present, with the client's permission we placed a drop of dilute hydrochloric acid on an inconspicuous spot of a sample. With magnification,



Figure 14. This odontolite cabochon shows the parallel banding frequently seen in this material. Magnified 17×.

we observed a weak effervescence consistent with a carbonate. This was confirmed by the infrared absorption bands located between 2500 and 3000 cm^{-1} , detected with a Nicolet 510 FTIR spectrometer. Additional absorption bands between 1000 and 1600 cm^{-1} suggested that the replacement minerals also included phosphates.

For a short time in the mid-1800s, odontolite was mined commercially in the Department of Gers, in southern France. This might well be the source of the material in these pieces, since their style is consistent with that period. Max Bauer, in his book *Precious Stones* (Charles E. Tuttle Co., Vermont, 1969, reprint of 1904 translation), mentions that when the material in France was mined, it was an unattractive gray-blue color, which became a fine blue with heat treatment. GRC

PEARLS

Natural-Color Black Cultured Pearl with an Unusual Surface

Ideally, the surfaces of both natural and cultured pearls should be smooth and free of blemishes. Although sometimes, to the unaided eye, these ideal conditions appear to be met, usually there are at least some surface blemishes.

The East Coast laboratory recently examined a strand of round, natural-color, black cultured pearls that averaged approximately 13 mm in diameter. All had blemishes of one sort or other, but on some the entire surface was covered with regularly arranged, "dimpled" pits that gave the appearance of a golf ball. The cen-



Figure 15. The dimpled surface on this 13-mm black cultured pearl is very unusual.

ter pearl in figure 15 is an excellent example of this effect, which differs from the "hammered" appearance of some natural saltwater pearls.

Examination with magnification revealed the presence of very tiny bumps at the bottoms of these pits (see figure 16). Staff members at the lab do not recall seeing anything similar on other cultured pearls, and have no idea what the cause might be.

GRC

Pearl Care

Wearers of pearl necklaces have been advised for generations to wipe their necklaces with a soft cloth after each wear and avoid cleaning them with liquids, because the capillary attraction of the string may draw substances contaminated by skin acids into the drill holes and cause them to enlarge. This is particularly important for natural pearls, where the drill holes are much finer than in cultured pearls. With both types of pearls, the pearl stringer should wash them thoroughly at the time of restringing and run a length of clean thread through the drill holes to dry them out.

Although customers are com-

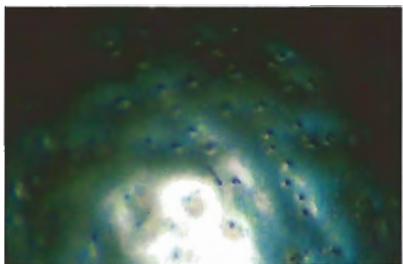


Figure 16. Tiny bumps are seen in the depressions on the pearl in figure 15. Magnified 32x.

monly told that pearls are relatively soft and are damaged by contact with skin acids, instruction in the care of pearls in jewelry other than necklaces has generally been neglected. Since most other pearls in jewelry do not come into direct contact with the skin, instruction for their care usually amounts to gentle warning about their softness and the effects of acids.

Stronger additional caution may be warranted, however. Figure 17 shows the "remains" of a 7-mm half-drilled cultured pearl from a stud earring that was worn constantly. When the pearl was unmounted, it was evident that the portion of the pearl protected from skin acids by the pearl cup now projects out from the area above it, which has been worn away so much that the nucleus is exposed and beginning to wear, too. Wearers of pearl stud earrings should be advised to wash them regularly with mild soap and water, especially if



Figure 17. Note the relationship of the eroded area on this 7-mm cultured pearl to the area protected by the earring cup.

they are worn night and day, as was the case with this earring. GRC

Pearl Half

The GIA Gem Trade Laboratory usually identifies natural and cultured pearls by the structural characteristics as seen on an X-radiograph. It is rare that we get a direct view of the interior of the pearls we are asked to identify. While examining a 6-mm round pearl that graced a diamond clasp (figure 18), the West Coast laboratory staff was surprised to find that the pearl had actually been cut in half before mounting. Figure 19 shows the back of the clasp and the exposed cross-section of the pearl, which clearly reveals its internal structure. Around a conchiolin-rich dark core, numerous darker concentric conchiolin layers have been deposited, beautifully illustrating the characteristic structure of a natural pearl. KH

Figure 18. Half of a 6-mm natural pearl has been set in this diamond clasp.



Figure 19. The back of the half pearl in figure 18 shows perfectly the concentric nacreous layers that form from the center outward in a natural pearl.

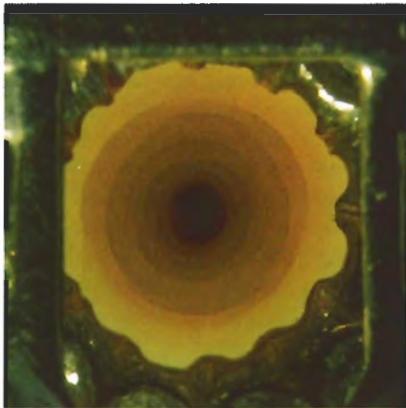


PHOTO CREDITS

Nicholas DelRe took the pictures in figures 1-4, 8, 9, and 13-17. Maha DeMaggio furnished figure 7. John I. Koivula made the photomicrograph for figure 6 and the photographs for figures 18 and 19. Shane F. McClure is responsible for the photos in figures 5 and 11. Figure 10 is compliments of Christie's. Emmanuel Fritsch provided the top spectrum in figure 12.