GEM TRADE LAB NOTES

DIAMOND

Heat-Damaged Filled Diamond

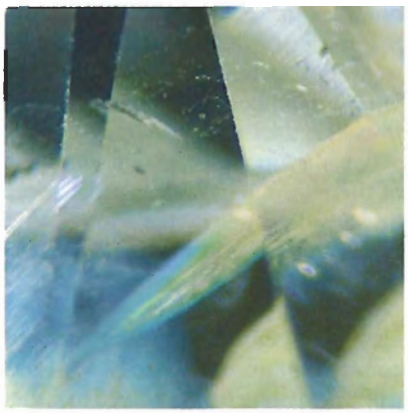
When GIA first began to study fracture-filled diamonds, we discovered that heat from a jeweler's torch during jewelry repair could damage the filling substance and make a fracture more visible (see, e.g., "The Characteristics and Identification of Filled Diamonds," *Gems & Gemology*, Summer 1989, p. 80). Because in most cases diamond jewelry can be repaired without removing the diamonds, we knew it was just a matter of time before we would begin to hear from unsuspecting jewelers who had unwittingly damaged filled diamonds by exposing them to the heat of the torch.

Therefore, we were not surprised when a jeweler recently submitted to the East Coast laboratory a 3.02-ct diamond mounted in a ring that was being repaired. Fractures that previously had been invisible could now be seen with the unaided eye and had a strong yellow cast (figure 1).

When we examined the stone, we saw that it still exhibited the "flash effect" (figure 2) characteristic of filled diamonds (again, see the Summer 1989 issue of *Gems & Gemology*, p. 72; also, the Gem News section of the Spring 1990 *Gems & Gemology*, p. 105). In general, a filled fracture will reveal an orange-to-blue or purple-to-green "flash" when examined in a direction nearly parallel to the fracture. Because our research has shown that the filling material used contains some lead, which is relatively opaque to X-rays, we took an X-radiograph of the stone to confirm that it had been filled. As we had suspected, the filled fractures did indeed appear "lighter" on the X-ray film (figure 3).

Figure 1. The filling in this 3.02-ct diamond was damaged by the heat from a jeweler's torch.

Figure 2. A faint orange-to-blue flash effect can still be seen in the damaged fracture-filled diamond shown in figure 1. Magnified 32×.



Such heat damage reinforces the need for jewelers to identify the presence of fracture filling in diamonds before they perform any jewelry repair that requires heat. If the diamonds are filled, they MUST be removed from their mountings to prevent heat damage.

DH

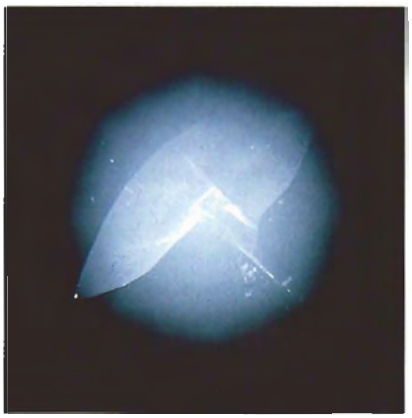


Figure 3. An X-radiograph of the diamond in figure 1 provides further proof of fracture filling, as the filling material is opaque to X-rays.

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

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Large Chameleon-Type Diamond

The color and appearance of the 22.28-ct heart-shaped brilliant-cut diamond (figure 4) recently submitted to the East Coast lab suggested that the stone might be a chameleon-type diamond. A strong greenish yellow fluorescence to long-wave ultraviolet radiation (figure 5) and a persistent yellow phosphorescence, combined with absorption bands at 415 and 425 nm, identified the stone as diamond and proved the natural origin of its color (see, e.g., Fritsch et al., "Optical properties of diamonds with an unusually high iron content," *New Diamond Science and Technology, 1991*). Furthermore, gentle heating with an alcohol lamp prompted a color change from fancy grayish yellow to nearly pure yellow with similar intensity.

Although we have encountered chameleon diamonds with more dramatic color changes (see, e.g., this section of the Winter 1982 *Gems & Gemology*, p. 228), this stone was nearly three times larger than any previous chameleon diamond tested in the East Coast lab. *TM*

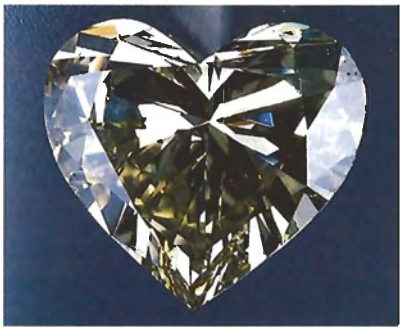


Figure 4. At 22.28 ct, this heart shape is by far the largest chameleon-type diamond ever tested at the East Coast lab.

Figure 5. The strong fluorescence to long-wave U.V. radiation (and persistent phosphorescence) of the stone in figure 4 is typical of chameleon-type diamonds.

Treated "Black" Diamond

The East Coast lab recently received a pear-shaped diamond, which appeared to be black, for an identification and origin-of-color report. The stone measured 15.20 × 10.00 × 6.30 mm (computed to be approximately 5.75 ct) and was set in a yellow- and white-metal pendant-style locket with numerous near-colorless round brilliants (figure 6). The visual characteristics and the high thermal conductivity, as measured with a GIA GEM Instruments Duotester, confirmed that the stone was diamond. The stone appeared to be opaque throughout, and microscopic examination showed a well-polished surface. Most natural-color black diamonds polish poorly because the black appearance is caused by the presence of numerous minute graphite inclusions (Kammerling et al., *Gems & Gemology*, Winter 1990, pp. 282–287).

Closer examination with a GIA GEM FiberLite with pinpoint attachments revealed that the stone was actually very dark green, a color we have never seen in nature and we know results from radiation treatment in a nuclear reactor. Stones treated in this manner can exhibit residual radioactivity. Indeed, scanning of this diamond with a hand-held Geiger counter did reveal residual radioactivity.

For a more precise measurement of the radioactivity, the pendant was forwarded to our new radiation-testing facility in the West Coast lab. The piece was placed over a high-purity germanium gamma-ray detector (see the article by C. Ashbaugh in this issue) and counted for an hour. Analysis of the gamma-ray spectrum showed measurable quantities of three radionuclides that were produced during treatment in the nuclear reactor: 3.75 nCi/g (nanocuries per gram) of europium-152 (Eu-152), 0.5 nCi/g of europium-154 (Eu-154), and 0.3 nCi/g of cobalt-60 (Co-60). Our data (the ratio of Eu-152 to Eu-154) indicate that this stone was treated relatively recently, that is, within the past few years.

Figure 6. The "black" diamond in this pendant proved to be a radioactive laboratory-irradiated dark green diamond. It was computed to be approximately 5.75 ct.

The United States Nuclear Regulatory Commission (NRC) has set legal limits for the sale or distribution of gem materials in the U.S. that contain reactor-produced radionuclides. For these three isotopes, the limits are 0.6, 0.6, and 0.5 nCi/g, respectively. At the time we measured this diamond, the total radioactivity of the stone was more than seven times these limits combined (computed as the sum of the ratios). However, this value is still only about twice the ambient radiation found in the environment (background level) for most of the United States, so, in compliance with the laws of the United States and the regulations

of the NRC, the item was returned to the client with full disclosure.

Radioactivity decreases with time. The decay rate of each radionuclide is described by its half-life, which is the time it takes for the amount of the radionuclide present (and, therefore, the amount of radioactivity) to decrease by half. Thus, after two half-lives there will be one-fourth of the original radioactivity present, after three there will be one-eighth, and so on. The half-life of Eu-152 is 13.5 years, that of Eu-154 is 8.6 years, and that of Co-60 is 5.3 years. Using these decay rates, we calculated that this diamond would reach the NRC release limits in about 36.6 years.

Ilene Reinitz and Chuck Ashbaugh

Editor's Note: GIA recently received licenses from the United States Nuclear Regulatory Commission (NRC) and the State of California to possess, test, and distribute radioactive material in conjunction with a new Radiation Testing Service being offered at the West Coast facility of the GIA Gem Trade Laboratory.

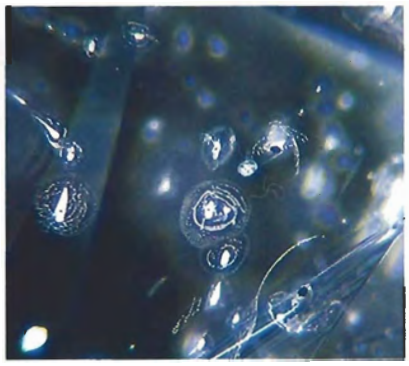
EMERALD Imitation: YAG

Green YAG (yttrium aluminum garnet) is sometimes used as an emerald imitation. Although it is usually relatively easy to identify, a local dealer, suspicious about a reportedly Colombian emerald he had been offered, was confused by inclusions that he thought resembled the "*jardin*" seen in natural emeralds. To be on the safe side, he sent the 5.56-ct oval mixed cut to the East Coast lab for identification.

The GTL examination revealed that the stone had a refractive index over the limits of the refractometer (emerald typically ranges from 1.57 to 1.58), was singly refractive (emerald is doubly refractive and uniaxial), and had a high specific gravity, 4.55 (compared to about 2.72 for emerald). Finally, examination with the microscope at high magnification revealed that the inclusions that the dealer thought were

Figure 7. High magnification (126×) revealed that what was believed to be "jardin" in an emerald is actually a combination of bubbles and flux in a green YAG.

Figure 8. Inclusions in this near-colorless YAG are similar to those shown in the YAG in figure 7. Magnified 27×.



"*jardin*" were actually a myriad of gas bubbles intermixed with a white flux-like material of unknown composition (figure 7).

All of the properties obtained were typical of YAG. On checking the GTL reference collection, we found a 3.95-ct near-colorless round-brilliant-cut YAG with similar inclusions (figure 8).

DH

GLASS Imitations of Various Gems

From time to time, GTL almost simultaneously receives items from different sources that turn out to be the same material. Lately, we in the East Coast lab have seen several different types of glass imitations. The following describes three such identifications.

The samples shown in figure 9 resemble some of the many thousands of "crystals" that have been used in jewelry and sold in the crystal-healing market with claims that they will cure almost anything. However, closer examination showed stretched gas bubbles which, together with the specific gravity of 2.52 and R.I. of 1.52 (singly refractive), proved they were glass.

Another item was a turn-of-the-century ring that some dealers believed was set with a piece of fine Persian turquoise (figure 10). Identification of this material as glass was not easy, since we could not take a good refractive index because the surface was pitted. Higher magnification revealed that these "pits" were actually vitreous conchoidal fractures, which would indicate glass or at least eliminate turquoise as a possibility. With the aid of a strong fiber-optic light source, we observed

Figure 9. These two glass imitations (note the stretched gas bubbles in the larger one) were sold as "healing crystals." The larger piece measures 34.16 × 10.10 × 9.27 mm.

Figure 10. Some dealers thought that the 9 × 6 mm glass cabochon in this ring was fine Persian turquoise.

translucent internal flow lines; these, together with the vitreous luster on the conchoidal surface fractures, proved that the material was glass.

The third example illustrates a situation that is of concern to many dealers. A number of stones in what looks like, and was thought to be, a parcel of amethyst or synthetic amethyst (figure 11) turned out to be glass. Not only are they a good color match, but they have a refractive index of about 1.55, which overlaps the R.I. range for amethyst (1.544–1.553). So, taking a hasty single refractive index, without checking for birefringence, could well lead to the wrong conclusion.

One good "quick" test on a parcel of such stones is to expose them to short-wave U.V. radiation. As illustrated in figure 12, the glass fluoresces a chalky blue, while the amethyst (whether synthetic or natural) is inert. Note that this "quick" test should only be used as an indicator that something is amiss, not as an identification.

Nicholas DelRe

PEARLS

Black, from Baja California

As we reported in the Spring 1991 issue of *Gems & Gemology* (p. 42), the Gulf of California appears once again to have become a major source of natural black pearls. Even before this earlier report, the editors were shown some shells of the pearl-producing oyster *Pinctada mazatlanica*, measuring approximately 13 × 14 cm, that had been fished off the east coast of Baja California. One shell showed a light brown 3/4 blister pearl that was approximately 11.5 mm in diameter.

At the 1991 Tucson Gem & Mineral Show, we were shown a beautiful purplish brown button pearl (figure 13), approximately 15 mm in diameter and 10 mm deep, that had recently been found off La Paz, Baja California. Subsequent X-ray examination in our West Coast lab confirmed that the pearl was indeed natural. The pearl also fluoresced a strong red to long-wave U.V. radiation, a characteristic of natural-color black pearls from the La Paz area.

KH

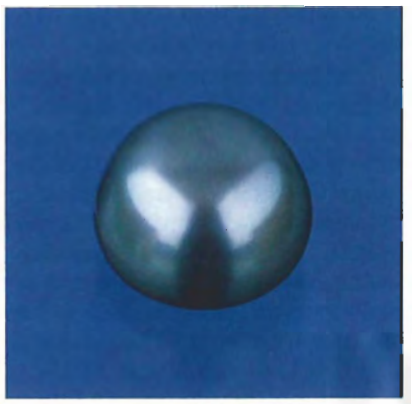


Figure 13. This natural-color natural black pearl (15 mm in diameter × 10 mm deep) was recently found off the coast of Baja California.

Treated Black Mabes

Shortly after our note on treated white assembled blister pearls (mabes) appeared in the Fall 1991 issue of *Gems & Gemology* (p. 177), the West Coast laboratory examined yet another type of enhanced assembled blister pearl. This assemblage had the usual white mother-of-pearl base, but the top was a dark purplish brown nacre that showed very high luster and orient. Figure 14

Figure 11. Note the fine color match of the natural amethysts and glass imitations in this parcel. The R.I.s are also closely matched, at 1.55.

Figure 12. In this parcel, the glass imitations fluoresce chalky blue—while the amethysts are inert—to short-wave U.V. radiation.

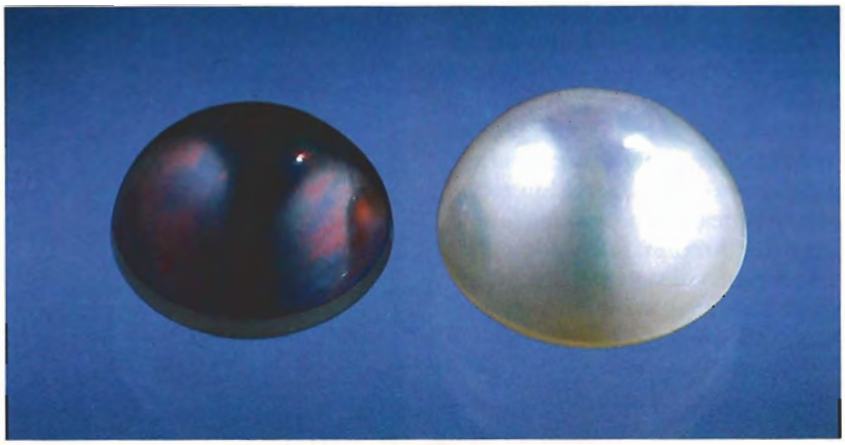


Figure 14. The color of the 13-mm black mabe assembled blister pearl on the left was produced by dye. It is shown here with a white mabe.

shows one of these "black" mabes (here, approximately 13 mm in diameter) next to a typical white mabe.

Closer examination of the black mabe assembled blister pearl revealed a slightly uneven color distribution in the nacre layer; with strong overhead illumination and high magnification, irregular darker brown areas became visible (figure 15). This splotchy color appearance suggested enhancement. A cotton swab soaked in 2% dilute nitric acid solution was stained when gently rubbed over the surface—proving that the nacre top had been dyed. In addition, this mabe pearl fluoresced a dull reddish orange to long-wave U.V. radiation, unlike the brownish red to red fluorescence usually seen in natural-color black pearls. We have since seen a number of such treated black mabes—reportedly manufactured in Japan—in the laboratory.

The GIA Research Department analyzed the nacre layer by energy dispersive X-ray fluorescence (EDXRF) to determine the chemical component that could have been used to treat this type of assembled blister pearl. In addition to silver, small amounts of bromine were detected. *KH*

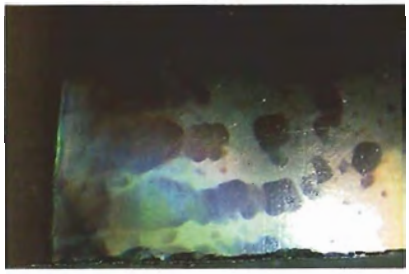


Figure 15. Irregular areas of color suggested that this black mabe assembled blister pearl had been dyed. Magnified 5×.

"Geneva [Synthetic] RUBY"

The Fall 1991 issue of *Gems & Gemology* (p. 178) has a photo of a pendant containing diamonds and early synthetic rubies with a sprinkling of natural rubies. Because of the rudimentary equipment used in their manufacture (as early as 1884), the first synthetic ruby boules produced were under considerable strain. Consequently, attempts to cut sizable stones usually met with failure. The small stones in the pendant described earlier were consistent in size with most of those we had seen thus far at GTL.

Recently, however, the East Coast laboratory had the pleasure of identifying relatively large examples of these early synthetics, which were commonly called "Geneva Rubies" in the trade. These synthetic rubies were mounted in a yellow- and white-gold ring, with rose-cut diamonds (figure 16), that appears to be in the "turn of the century" style. The center stone measured approximately 7.00 × 6.00 × 3.00 mm. The weight-estimation formula suggests that the stone weighs more than 1 ct. In figure 17, the tightly curved growth lines and black impurities characteristic of these early synthetics are readily apparent. We have rarely seen this material in stones this large, and to find nine—all without strain cracks—in one piece is unexpected. *GRC*

SAPPHIRE, Durability of Heat-Treated Stones

The East Coast lab received a 3-ct sapphire, set in a modern ring, for a damage report. The client had complained about the worn facet junctions (see figure 18), which resembled those commonly seen on heat-treated zircons, and wondered about the cause. Since all damage reports include an identification report, we first determined that this stone was diffusion treated—a fact that came as a complete surprise to

Figure 16. This beautiful period ring contains nine "Geneva rubies" (early flame-fusion synthetics). The center stone is over 1 ct; none of the stones shows strain cracks.

Figure 17. Sharply curved striae and dark inclusions, seen here at 19×, prove that the synthetic rubies shown in figure 16 are of early manufacture.

our client. Unfortunately, because the color is confined to a thin surface layer, the client probably could not even repolish the stone to eliminate the abraded facet junctions.

A possible explanation for the worn facet junctions was given to us more than 10 years ago, by sapphire dealers in Australia who reported that heat treatment could make the stones brittle. Such stones would suffer more than usual "paper wear" unless each one was wrapped separately. However, one informant reported, once the parcels of wrapped stones were sent to Bangkok for marketing, they were unwrapped and mixed with other stones.

If the durability of a sapphire is indeed impaired after successful heat treatment, the question arises as to how much more it would be impaired if the sapphire were to undergo repeated exposures to high heat, as is commonly the case for those stones that do not respond to heat treatment (because they lack the necessary iron and titanium) and are submitted to diffusion treatment only after several unsuccessful attempts at heating. In addition, the "deep" diffusion treatment to which many thousands of sapphires have been subjected in recent years reportedly requires multiple heatings (for further information on diffusion-treated sapphires, see the article by Kane et al. in the Summer 1990 *Gems & Gemology*, pp. 115–133).

GRC

Identifying Curved Striae in Yellow SYNTHETIC SAPPHIRE

Curved striae are usually difficult, if not impossible, to see in yellow or orange flame-fusion synthetic sapphires. They are more difficult to see in stones with a pale body color than in the darker synthetic rubies and blue sapphires.

If it is suspected that a yellow or orange stone is a flame-fusion synthetic, it is often helpful to use a filter of complementary color when looking for curved striae. With orange to yellow sapphires, a blue plastic or glass filter placed between the stone and the microscope light source may make the curved striae easier to see (see, e.g., the article by R. Hughes in *Journal of Gemmology*, Vol. 21, No. 1, 1988, pp. 23–25; also, *Gems & Gemology*, Winter 1990, p. 299).

East Coast lab staff were, therefore, surprised when the curved striae in a yellow flame-fusion synthetic sapphire they had been asked to identify were readily visible under normal observation even without magnification (figure 19)!

DH

Figure 18. The abraded facet junctions on this approximately 3-ct diffusion-treated sapphire probably could not be repolished without removing part of the shallow color layer.

Figure 19. Curved striae are easily visible without magnification in this 15.50 x 10.75 x 6.15 mm flame-fusion synthetic yellow sapphire.

PHOTO CREDITS

Figures 1, 2, 4, 5, 7-12, and 16-19 were supplied by Nicholas DelRe. The photos used in figures 6 and 13 were taken by Shane McClure. The X-radiograph in figure 3 was taken by Bob Crowningshield. Figure 14 is © GIA and Tino Hammid. Figure 15 is by John I. Koivula.