Fracture Filling of Emeralds

Opticon and Traditional "Oils"

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The filling of surface-reaching breaks in emeralds is a relatively common practice, for which various kinds of oils and a natural resin have historically been used. Now, however, epoxy resins are replacing the more traditional fillers such as cedarwood oil and Canada balsam. The most widely known of these epoxy resins is sold under the brand name Opticon. The results of a broad study of various fracture-filling materials found that Opticon treatment (1) was, like the traditional materials, best detected using magnification with a variety of lighting techniques; and (2) although somewhat more durable than the traditional enhancements, was still altered in the course of routine jewelry cleaning and manufacturing processes. This article also examines the filling of surface pits with epoxies, the potential effectiveness of "dyed" Opticon, and the use of Opticon to fill the fractures in gem materials other than emerald.

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Although color enhancements have played a preeminent role in gemology in recent years, clarity enhancements are rapidly gaining in prominence. The filling of fractures in emeralds has been practiced for decades. During the 1980s, we also saw the introduction of "filled" surface pits and cavities in ruby, as well as the filling of surface-reaching separations in diamond (as reviewed in Kammerling et al., 1990).

Historically, the fractures in emeralds have been filled with a variety of oils (of which cedarwood oil is perhaps the best known) and the natural resin Canada balsam (see, e.g., Ringsrud, 1983). However, recent reports in the trade press have mentioned a more sophisticated fracture-filling procedure and the greater use of epoxy resins (Themelis and Federman, 1990; Themelis, 1990). What appears to be the most popular of these epoxy resins is marketed under the brand name Opticon. Still other reports make note of additional, proprietary fracture-filling treatments for emeralds. These include one being offered in Israel by the firm (Zvi Yehuda, Ltd.) that pioneered the filling of fractures and cleavages in diamond (see, e.g., Gilbertson, 1990; Lee, 1990; Yehuda, 1990); another being offered by CRI Laboratories of Grand Rapids, Michigan ("Emerald Clarity Enhancement Offered by U.S. Treatment Laboratory," 1990; "Emerald Treatment Services," 1990; Lee, 1990); and yet another being provided by the Kiregawa Gemological Laboratory in Japan (Y. Doi, pers. comm., 1991).

With this proliferation of processes and substances, concern has developed that filled fractures may have become even more difficult to detect. In addition, there is considerable question in the trade as to the durability of the different fillers and filling processes. For example, there is the perception that Opticon offers advantages over so-called traditional fillers because the breaks can be sealed at the surface. In other cases, as with the CRI and Yehuda emerald-filling services, claims to enhanced durability

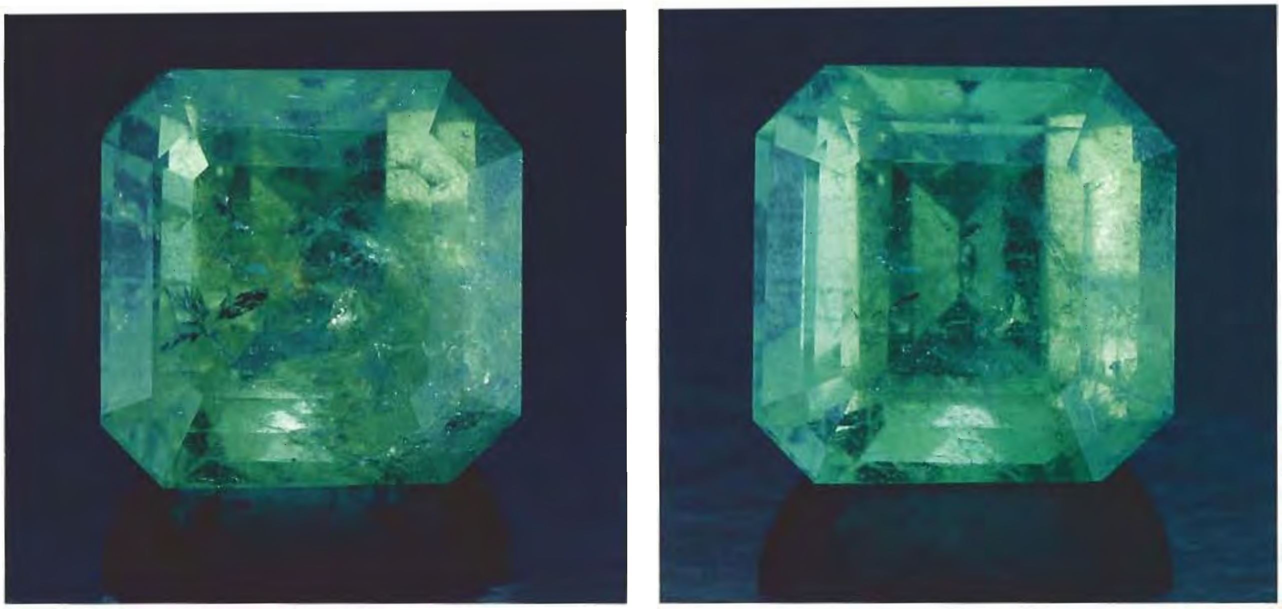


Figure 1. These two photos of a 5.74-ct emerald before (left) and after (right) Opticon treatment graphically illustrate how effective this fracture-filling treatment can be in improving apparent clarity. Photos © GIA and Tino Hammid.

have been made openly (see, e.g., Everhart, 1989; Gilbertson, 1990; Lee, 1990; Themelis and Federman, 1990; "U.S. Firm Offers New Emerald Treatment," 1990). Last, because of the various durability claims, jewelers and gemologists alike are trying to relate various features (e.g., "flash effects"; Kane, 1990) to specific processes.

This article, the first of two parts on the fracture filling of emeralds, will focus on stones treated with Opticon (figure 1) as they compare to those filled with the more "traditional" cedarwood oil and Canada balsam. Following descriptions of a commercial Opticon treatment procedure as practiced in Brazil and the identifying features of Opticon-treated stones, we will examine the relative durability of the filling materials when subjected to standard jewelry cleaning and manufacturing procedures. The second article in this series will describe the identifying features and durability of the Yehuda, CRI, and Kiregawa treatments.

THE OPTICON TREATMENT PROCESS

Opticon Resin No. 224, an epoxy resin marketed by Hughes Associates of Excelsior, Minnesota, is widely available commercially. The Opticon (plus hardening agent) kits used for this study were purchased at two Los Angeles–area lapidary supply shops (figure 2).

A number of dealers have told the authors that Opticon treatment is used on the majority of emeralds mined at Santa Terezinha in Goiás, Brazil (see, e.g., Koivula and Kammerling, 1989). Recently, one of the authors (PM) visited Stone World in Teófilo Otoni, Minas Gerais, Brazil, a firm that purchases and cuts rough emeralds (predominantly from Santa Terezinha) and then markets them in Brazil, the U.S., and Europe. According to Mr. Sérgio Martins, president of Stone World, production averages 5,000–6,000 fashioned stones per month, approximately a third of which are relatively large and good quality. Most stones are in the 0.5- to 2.0-ct range, with about 10% in the 2.0- to 5.0-ct range.

After cutting, Stone World treats virtually all their emeralds with Opticon (figure 3). First, the

Figure 2. Opticon treatment kits are available at lapidary shops throughout the U.S. Photo by Robert Weldon.

Figure 3. Treatment at Stone World, in Teófilo Otoni, Brazil, begins immediately after cutting. The stones are first cleaned in weak hydrochloric acid, rinsed in water, and—as shown here—examined to make sure that any polishing compound or residual fillers (used on the rough) have been removed. Photo courtesy of Stone World.

fashioned stones are cleaned, that is, they are soaked in dilute hydrochloric acid and rinsed in water. Mr. Martins indicated that the acid used is quite weak and that some treaters simply use lemon juice. The stones are cleaned not only to remove polishing compound but also to remove any residue of Opticon that the miners may have applied to the rough.

Next, the cleaned stones are immersed in Opticon in small (50 ml), heat-resistant Pyrex beakers. As many as 200 small stones may be placed in a single beaker. The beakers are then placed (uncovered) in a small oven and brought to a low temperature that is maintained for 24 hours. Considerable experimentation was done by Stone World to find optimal temperatures; they did not reveal those temperatures for proprietary reasons. However, one dealer familiar with Opticon suggests 95°C (203°F; J. Crescenzi, pers. comm., 1991). This gentle heating reduces the viscosity of the filler and thus helps it penetrate the fractures.

After the stones are removed from the oven, they are left (still immersed in Opticon) to cool to room temperature. At this point, the emeralds are removed from the beakers and the hardening agent (or a mixture of Opticon and hardener; figure 4) is wiped across the entire surface of each stone. The hardener is left on for 10 minutes; this "sets" the filler near the surface of the fractures, sealing the remainder of the still-liquid Opticon inside. According to Mr. Martins, the excess hardening agent must be wiped off at about 10 minutes; at 15 minutes, it is completely set and will require repolishing to remove. Thus, no more than 25–30 stones can be treated at one time with the hardener. After the hardening treatment, the stones are washed with a mild solution of water and baby shampoo and rinsed in water.

The stones are then carefully examined. Mr. Martins noted that approximately 15% must be recut at this point to repair surface damage that occurred during treatment. (Note: Although Mr. Martins did not elaborate on this damage, we observed minor chipping on the edges of some stones during our experimentation with Opticon treatment. This may be caused by the expansion of pre-existing fractures during heating.) After these stones are recut, they are cleaned again and then retreated with Opticon.

The above describes only one method used to treat emeralds with Opticon; there appear to be countless variations. For example, Gemlab Inc. of Clearwater, Florida, reportedly uses vacuum pumps to evacuate the fractures prior to filling, high pressure to force the filler into the fractures, and a radio-frequency thermal-wave transmitter to heat the filler during the filling process (Themelis and Federman, 1990).

Figure 4. After Opticon treatment at Stone World, the emeralds are coated with a mixture of Opticon and hardener—here, being prepared—to seal the surface-reaching breaks. Photo courtesy of Stone World.

BOX A: USE OF GREEN-DYED OPTICON

Not all of the "oiling" of emeralds is carried out with essentially colorless filling substances. It is well known that green oils and dyes are also used, primarily on pale, low-quality beryls that might not be accepted as emeralds in their untreated state (see, e.g., Fryer, 1981; Nassau, 1984). From the standpoint of disclosure, such enhancement is generally considered dyeing rather than fracture filling.

Colored Opticon is now being used on emeralds as well. Themelis (1990) noted that any of the filling agents he described (which include Opticon) could be mixed with green organic dyes. Stone World has also experimented with the use of green-dyed Opticon. According to Stone World's Luiz Martins (pers. comm., 1991), however, there was a higher incidence of breakage during the treatment process when the colored Opticon was used—for which no explanation was offered—and they use only untinted Opticon in their commercial treatment. Dealers at the February 1991 Tucson show reported that colored Opticon was being used by some emerald treaters in Brazil (Koivula and Kammerling, 1991).

In an effort to determine the effectiveness and identifying features of green-dyed Opticon, the investigators made a number of attempts to mix Opticon with green coloring agents. These experiments met with limited success. In one, a small amount of Opticon was put in a test tube and a green coloring agent, marketed to color polyester casting resins, was added a drop at a time and mixed until a very dark green color was obtained. A near-colorless (very, very light blue) beryl that had been quench-crackled to produce numerous surface-reaching fractures was treated with the colored Opticon plus a hardener to seal the breaks. The resulting stone appears a light yellowish green face-up. Magnification combined with darkfield illumination revealed orangy yellow and blue dispersion flashes from the filled fractures, while diffused transmitted light revealed concentrations of light green color in the filled areas.

Figure A-1. The combination of Opticon treatment and green dye in a colorless quartz similar to the stone on the left produced a radical change (right). Photo by Maha Smith.

In two other experiments, Opticon was mixed with powdered green dyes, one a commercial fabric dye and the other a substance marketed for dyeing gems. While both appeared to produce a dark green liquid Opticon, the quench-crackled colorless beryls and quartzes treated with these dyes remained essentially colorless, albeit nicely filled with essentially colorless Opticon. A commercial treater of emeralds who at one time had experimented with colored filling substances (T. Lee, pers. comm., 1991) indicated that the mixture of powdered dye and Opticon, rather than producing a solution, resulted in a fine suspension of dye particles in the Opticon. In the filling process, the dye was "filtered" out of the Opticon at the surface of the fractures, resulting in an essentially colorless filling.

The final experiment involved essentially colorless quartzes that had previously been quench-crackled and dyed green. The original transporting agent for the dye had since evaporated, leaving particles of green dye lining the walls of the fractures. These stones were then treated with undyed Opticon. The results (figure A-1) give an indication of the significant amount of color that might potentially be added to a stone with colored Opticon.

To document the appearance of stones before and after treatment, the authors developed their own treatment methods. Stones to be treated (all from the GIA reference gem collection) were first sent to CRI Laboratories in Grand Rapids, Michigan, for thorough cleaning to remove residue of previous fillings in the fractures. Tom Lee, president of CRI, indicated that they had found that the most effective cleaning method was to place the stones in methylene chloride under a pressure of 50 p.s.i.

At GIA, the cleaned stones were placed in a heat-resistant glass beaker and heated for 10 to 20 minutes in an oven set at approximately 95°C (203°F). While the stones were heating, a small amount of Opticon was placed in a small Pyrex test tube fitted with a rubber stopper pierced by a glass tube. The test tube containing the liquid was placed in a pan of water and heated on a gas range until the water started to boil; in a few minutes, the viscosity of the liquid was significantly reduced. The stones were then removed from the oven and placed in the preheated Opticon. After the stopper was replaced, the glass tube that pierced it was attached to a hand pump. A partial vacuum was then drawn and maintained at 0.5–0.8 atm. for 10–50 minutes. The length of the treatment time varied with the size and number of stones being treated, as well as with the apparent success of the treatment process. Every few minutes the test tube was removed from the water and the contents visually examined. If any minute bubbles were still rising from the stone(s) or significant unfilled areas were noted in fractures, the treatment was continued. When it was felt that no further filling would take place, the vacuum was released and the test tube was removed from the water and allowed to cool to room temperature. At this point, the stones were removed from the Opticon and were wiped clean of any excess liquid that remained.

The stones were next immersed for a minute or two in Opticon hardener and then allowed to sit in air for 10 minutes before the excess hardener was wiped off. Last, the treated stones were rinsed in water and dried with a soft cloth.

It appears that there are also variations with respect to the filling material itself. Stone dealers in Brazil told one of the authors (RCK) of a number of hardening agents, produced for use with other resins, that have proved somewhat successful in extending the "life" of the filling when they are mixed with Opticon before it is used to fill fractures. One published report confirms this (Themelis and Federman, 1990). The newest popular hardening agent mixed with Opticon is a product called Nu Seal, which is commonly used to harden dental resins (J. Crescenzi, pers. comm., 1991). However, there is some bias in the trade against this practice because it makes removal of the filling very difficult, should this become necessary. In this study, we used the hardener only as a sealant.

For comparison purposes, the authors also treated emeralds, synthetic emeralds, and other beryls with cedarwood oil and with Canada balsam, the two most familiar "traditional" fillers. The same filling procedure as for Opticon was used, except that no hardener or other agent was applied to seal the filled breaks.

THE EFFECTIVENESS OF OPTICON TREATMENT

The refractive index of Opticon, 1.545, is higher than that of either the cedarwood oil (1.512) or Canada balsam (1.520) we used and thus closer to the R.I. range of natural emerald (1.577 to 1.583 ± 0.017). Opticon may be, therefore, somewhat more effective in improving the apparent clarity of emeralds that have surface-reaching fractures. Individual fractures that were easily seen with the unaided eye before treatment often could not be detected without magnification after the filling procedure. Some stones used in our study had localized areas of dense fractures that, before treatment, collectively reflected and scattered so much light that they gave the areas a whitish appearance (figure 5, left). After treatment, such areas appeared green (figure 5, right). In fact, in some stones this reduction in light scattering also appeared to increase the depth of color.

IDENTIFYING FEATURES NOTED IN OPTICON-TREATED STONES

The authors performed some preliminary tests on the liquid resin itself. Opticon is transparent and near-colorless, showing only the slightest hint of yellow. As mentioned above, it has a refractive index of 1.545. It fluoresces weak to moderate white-blue to long-wave ultraviolet radiation, with no phosphorescence; it is inert to short-wave U.V.

The characteristics described in this section were determined on the following Opticon-treated stones: three emeralds treated and provided by Stone World (1.03, 1.19, and 2.65 ct); 24 emeralds treated by the authors (ranging from 0.20 to 5.74 ct); and one hydrothermal synthetic emerald (0.37 ct) that was first intentionally fractured ("quench crackled") by the authors using thermal shock.

Visual Observation. As mentioned above, Opticon treatment produced a marked improvement in apparent clarity. In some cases, the reduction in reflections and light scattering appeared to have improved the color as well.

Although for the most part the treated breaks were not visible to the unaided eye, some treated stones—especially those with the greatest number of filled breaks—showed a less-than-ideal transparency, or an optical distortion, that did not appear to be due to inclusions and would not be expected in a "flawless" single-crystal gem material (again, see figure 1 and figure 5, right). The

Figure 5. Before treatment (left) the whitish, reflective areas of dense fractures are easily seen in this 5.71-ct emerald. After treatment (right), most of the whitish areas are no longer visible. Photo © GIA and Tino Hammid.

effect is reminiscent of the so-called "heat-wave effect" noted in some hessonite garnets and the "treacle" color zoning often observed in rubies from Burma.

Ultraviolet Fluorescence. A relatively small percentage of the Opticon-treated fractures fluoresced a weak, chalky white to white-blue, similar to Opticon itself, to long-wave ultraviolet radiation. The others did not fluoresce, and all were inert to short-wave U.V.

Magnification. Magnification in conjunction with various lighting techniques revealed numerous identifying features. We did not see all of the features described below in all of the Opticon-treated emeralds examined, but we observed at least one in every stone.

Locating Where Filled Fractures Reach the Surface. The most constant visual feature of filled fractures is their very low relief. An untreated break—that is, one that contains air rather than a filling material such as Opticon—would have high relief, making it quite noticeable, even to the unaided eye. However, in addition to fractures, emeralds often contain numerous crystalline and fluid inclusions (see, e.g., Gübelin and Koivula, 1986) that can complicate the location and identification of filled fractures.

Thus, the first step in detecting possible filled fractures is to locate where any breaks reach the surface. An effective way of determining this is to position a light source close to the surface of the gemstone, so that only the surface is viewed in reflected light. The best results are obtained with an intense incandescent light source—such as that provided by fiber-optic illumination, a Tensor lamp, or a coaxial illuminator system—rather than fluorescent light. In this surface-reflected light, the fine, hairlike lines that mark the entry points of the fractures will often be readily visible (figure 6).

Another method is to use darkfield illumination and, holding the stone low in the microscope well, rock it until light reflects off the surface being examined. If entry points are detected, it is then easy to examine the interior of the stone immediately under those points.

Dispersion Flash Effects. The majority of the filled breaks exhibited flashes of dispersion color similar to the orange and blue flash effects shown by some diamonds with filled separations (see, e.g., Koivula et al., 1989). Examined nearly edge-on—that is, approaching a direction parallel to the plane of the fracture—some filled breaks in the Opticon-treated emeralds exhibited a slightly orangy yellow dispersion color: The entire fracture or a large portion of it seemed to "light up" with this color (figure 7, left). When the stone was rocked very slightly—in some, but not all instances—this changed to blue (figure 7, right). In instances where the blue flash was not noted, the filled break would

Figure 6. The use of surface-reflected light with the microscope enables the location of surface-reaching fractures in treated emeralds. Photomicrograph by John I. Koivula; magnified 25×.

flash orange, seem to disappear, and then reappear as the stone was rocked back and forth. At first, we thought that the green body color might be partially masking the blue flash. However, Opticon-filled fractures in near-colorless beryls that we treated also showed the orange flash alone more consistently than with the blue flash.

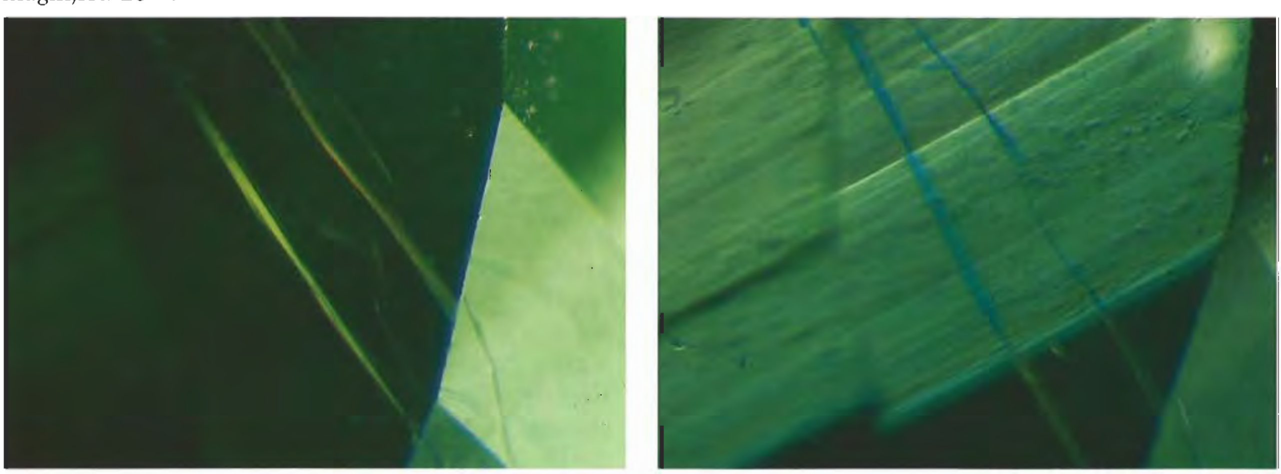
Both darkfield and oblique fiber-optic illumination were effective in revealing these flashes. Horizontal fiber-optic illumination was also effective alone or in conjunction with darkfield lighting. In some instances, this latter technique revealed a blue flash that was not noted with darkfield conditions alone. In many instances, we detected at least an orange flash using transmitted light; and in some, we detected dispersion flash colors with diffused overhead illumination.

We found these flash effects to be less prevalent in the Opticon-treated emeralds than in diamonds with filled surface-reaching separations we have examined (see Koivula et al., 1989). They were not observed in any of the cedarwood oil– and Canada balsam–treated stones we examined.

Trapped Bubbles. Discontinuities, actually flattened bubbles in the filling, were noted in many of the filled breaks with darkfield, oblique or pinpoint fiber-optic, or overhead illumination. Unlike natural, unfilled breaks, which have a mirror-like appearance, these bubbles were always at least partially surrounded by filled areas that were either transparent or "cloudy." With overhead illumination, some trapped bubbles exhibited bright interference colors, a thin-film effect similar to that seen in some natural, partially healed fractures. The trapped bubbles occurred in a great variety of sizes and shapes, singly and in groups (see, e.g., figure 8).

Outlining of Fractures. Careful examination with darkfield or fiber-optic illumination often revealed a faint outlining of the filled breaks. This was generally noted at an oblique angle of observation, as when the plane of the break was at approximately 45° to the line of sight.

Figure 7. A slightly orangy yellow flash (left) was observed in the filled fractures of this Opticon-treated emerald. When the stone was tilted slightly and the background became lighter, the flash turned blue (right). In some stones, only one color is seen. Photomicrograph by John I. Koivula; magnified 20×.



Flow Structure. Some filled breaks revealed a flow structure that gave them a faintly textured appearance, in some cases reminiscent of the so-called "heat-wave" effect sometimes noted without magnification. This was best seen using darkfield illumination with the stone positioned so that the background became brighter through secondary reflection from back facets (figure 9). The presence of air bubbles trapped along such "flow planes" also helps delineate these areas.

Cloudy Areas. In some of the filled breaks, we noted small, irregular, slightly whitish cloudy patches with a somewhat textured appearance. These may represent a partial alteration of the filler. One trade press report cites claims that Opticon-filled breaks become "cloudy after several months" (Everhart, 1989).

It is possible that at least some of these cloudy areas may be due to an incomplete reaction of the Opticon with the hardening agent. To test this, we placed a small amount of Opticon on a glass microscope slide and incompletely mixed it with a drop of the hardening agent. After approximately 20 minutes, we noted whitish, swirled, "cloudy" areas interspersed with still-colorless, transparent areas. This suggests that the hardening agent may partially penetrate the filled break and cause an incomplete localized reaction.

TRT Reaction. A thermal-reaction tester ("hot point") brought close to the surface of the stone will typically cause an unsealed fluid-filled break to "sweat" out some of the filler. Although we found that, for the most part, the fluid would not "sweat" out of an Opticon-treated break that had been sealed at the surface with hardener, with magnification we did observe visible movement of the still-liquid filling in the fractures. Note that because this procedure is potentially destructive, it is not recommended for routine testing.

Other Tests. One commercial treater recommends immersion microscopy as the best means of detecting fracture filling (Themelis and Federman, 1990; Themelis, 1990). With this method, the stone is immersed in a liquid with an R.I. very close to that of the stone being tested; for emerald, he suggests using bromoform, which has an R.I. of 1.56. Anything within the immersed stone should be visible to the degree to which its R.I. varies from that of the emerald. In addition to filled fractures, this would include crystalline inclusions, unfilled voids, and fluid inclusions.

Unfortunately, the immersion liquid might also act as a solvent that could partially remove near-surface filling material. As mentioned above, tests that are potentially destructive to either the host material or the treatment should be avoided for routine gem identification. Moreover, the bromoform might enter breaks in untreated emeralds,

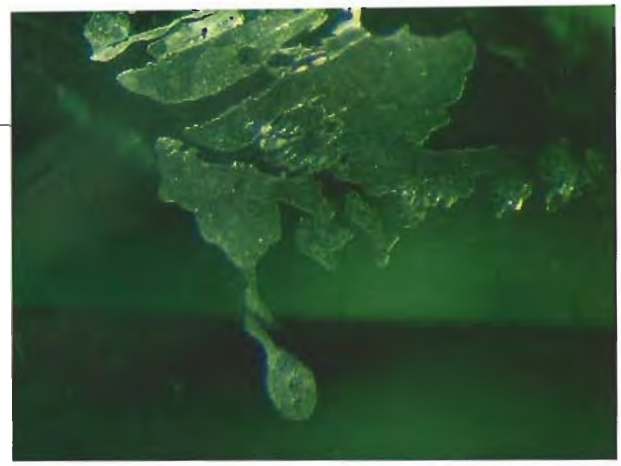


Figure 8. Trapped air bubbles may be an important identifying feature for filled emeralds. Although they vary greatly in size and shape, large bubbles with irregular outlines—such as those illustrated here in an Opticon-treated emerald—are not unusual. Some of the bubbles may be highly reflective with a silvery white hue. Photomicrograph by John I. Koivula; magnified 15×.

Figure 9. Careful examination of the areas surrounding trapped air bubbles will sometimes reveal subtle flow structures within the filling agent, which in this case is Opticon. Photomicrograph by John I. Koivula; magnified 20×.

BOX B: CAVITY FILLING

While this article focuses on the use of Opticon for filling fractures, it is apparent that other irregularities, such as surface cavities, could also be filled with hardened Opticon and similar substances. One emerald examined by the GIA Gem Trade Laboratory had a surface cavity filled with a soft, "plastic-like" substance. The stone also had been fracture-filled (Hurwit, 1989).

Discussions with one dealer familiar with Opticon treatment indicated that sometimes, in the course of treatment, the point where a fracture breaks the surface of a stone becomes enlarged, producing a cavity. Such a fracture is first filled with Opticon, after which a mixture of hardener and Opticon is used to simultaneously seal the break and fill the cavity. Alternatively, the Opticon may be mixed at a 1:1 ratio with Nu Seal (J. Crescenzi, pers. comm., 1991).

Another report suggests using Opticon to fill fractures and then a different epoxy resin, Epoxy 330 (which is marketed by the same firm that markets Opticon), to fill cavities (Jones, 1986). The authors obtained some of this product for experimentation. First, we mixed equal amounts of this epoxy resin with its hardener and allowed it to cure on a glass slide. The resulting hardened substance was transparent to semitransparent and colorless.

We then mixed additional epoxy and hardener and used it to fill surface cavities of various shapes and sizes on both natural and synthetic emeralds. After a minimum of 24 hours, the excess cured epoxy was polished down to the level of the stones' surfaces. The filled cavities can be readily identified with magnification and darkfield or surface-reflected illumination. In addition, they may contain gas bubbles (figure B-1), and some pits may be only partially filled (figure B-2). As is the case with rubies and sapphires, however, such fillers generally are not as durable as the host gem. If the cavities are large, the filler could add to the weight of the stone.

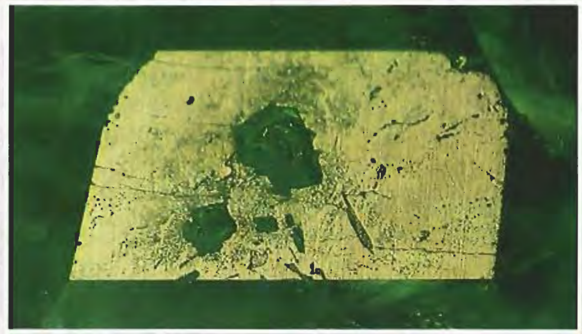


Figure B-2. Epoxy-treated surface pits in an emerald may be only partially filled. Photomicrograph by John I. Koivula; surface-reflected illumination, magnified 15×.

Figure B-1. This filled pit in an emerald shows spherical gas bubbles trapped within the epoxy. Photomicrograph by John I. Koivula; darkfield illumination, magnified 30×.

causing them to be unintentionally filled. Last, bromoform is highly toxic.

COMPARISON WITH CEDARWOOD OIL AND CANADA BALSAM

As mentioned earlier, cedarwood oil and Canada balsam have historically been the preferred substances for filling fractures in emeralds. For comparison purposes—in terms of effectiveness and identifying features—we treated two groups of five samples, one with cedarwood oil and the other with Canada balsam. Each group consisted of three emeralds ranging from approximately one-half to one-and-a-half carats, one flux-grown synthetic emerald less than a carat, and one very pale (near colorless) aquamarine of approximately 2 ct. Additional beryls were fracture filled for durability testing (see below).

All of the treated stones showed clarity improvement, although some breaks were still evident. Both fillers especially improved the appearance of the pale aquamarines, in which extensive fractures had been induced by quench crackling. With magnification, it was relatively easy to detect the filled breaks, which generally exhibited a slightly higher relief than did Opticon-filled breaks. In all cases, the cedarwood oil– and Canada balsam–treated stones could be positioned so that the fractures produced bright reflections that plainly revealed their contours – more clearly than in the Opticon-treated stones. We also noted some irregular trapped bubbles. Additionally, some fractures filled with cedarwood oil had areas that appeared to contain a whitish material, perhaps representing some of the oil that had dried out (or residue from a previous filler that had not been completely removed). Combined darkfield and pinpoint fiber-optic illumination revealed a muted orange dispersion effect in some of the breaks in the Canada balsam–filled aquamarine but not in any of the other samples.

The filled fractures in the five samples treated with cedarwood oil were inert to both long- and short-wave ultraviolet radiation. Filled fractures in all the Canada balsam–treated samples exhibited a weak greenish yellow to yellow fluorescence to long-wave U.V. radiation, with no phosphorescence; they were inert to short-wave U.V.

In general, for purposes of identification, the only consistent difference we noted between stones we treated with either cedarwood oil or Canada balsam and those filled with Opticon was the presence of the orange or blue flash in the Opticon-treated emeralds. However, we do know from our investigation of other filling materials (as will be described in part 2) that these flash effects are not specific to Opticon and can occur when other fillers are used.

DURABILITY OF TREATMENT

Of key concern is the durability of a filled stone during normal cleaning procedures, as well as the stability of the filling material itself. Ringsrud (1983) related the claims of several Colombian emerald dealers that "since the majority of emeralds have such fine fractures . . . the oil seldom dries out or, at the very least, the natural oils of the wearer replace the oil in the stone." Ringsrud, however, went on to state that there is variability in the permanence of this treatment. Nassau (1984) mentioned a number of specific factors – heat, reaction with atmospheric oxygen, contact with solvents, and cleaning procedures – that could negatively affect emerald fillings. Others (e.g., Crowningshield, 1972; Mumme, 1982; Webster, 1983; Martin, 1987) have also warned against using various cleaning techniques on oiled emeralds, as these could remove the filling material. In particular, oiled emeralds that have been subjected to ultrasonic cleaning or exposed to high temperatures have been known to exhibit whitish dendritic deposits in the fractures, residue of oil that has dried out (Crowningshield, 1984).

To test the durability of Opticon treatment relative to "oiling," we first subjected some of the stones we had treated by the different methods to ultrasonic and steam cleaning.

Ultrasonic Cleaning. For this portion of the investigation, we used a Gesswein Ultrasonic Cleaner model 87 containing BRC, a standard jewelry cleaning solution. The unit was set to the high setting and the heating element was turned on. Stones to be tested were put in a perforated plastic container which was then placed in the ultrasonic unit. Stones were checked at five-minute intervals for any change in appearance; total time in the unit was 30 minutes for each stone.

The results of cleaning at five-minute intervals for a 1.94-ct pale aquamarine treated with Canada balsam, a 1.78-ct pale aquamarine treated with cedarwood oil, and two Opticon-treated pale aquamarines (1.88 and 3.11 ct) are reported in table 1. Of the four stones, the two treated with Opticon appeared to have held up significantly better to this cleaning procedure than the Canada balsam– and cedarwood oil–treated stones. The overall negative effect and amount of material removed from these latter two stones appeared roughly equivalent (see, e.g., figure 10).

The ultrasonic cleaning procedure used in this phase of testing was admittedly more severe than that to which a jeweler would normally submit a stone. However, this test may realistically represent the cumulative effects of repeated ultrasonic cleanings on a fracture-filled stone. While the amount of material removed may have been minor, fractures that could not be seen without magnification before cleaning were eye visible afterward. This, in turn, could leave a jeweler open to claims of damaging (or even switching) a stone.

Steam Cleaning. Stones subjected to various filling treatments were also cleaned with a Reimers Model JR steam cleaner to test the fillings' durability to this procedure as well. In all cases, stones were initially subjected to a pressure of 60 p.s.i. and

Table 1. Results of the ultrasonic cleaning of "filled" pale aquamarines.

Weight (ct)	Treatment material	Results (minutes)					
		5	10	15	20	25	30
1.94	Canada balsam	Fine-appearing fractures visible breaking pavilion surface	Additional fractures visible	Two fractures more noticeable	Same two fractures appear larger	Same two fractures appear larger	Same two fractures more prominent
1.78	Cedarwood oil	A few surface-reaching fractures on pavilion appear as hairline marks	Numerous additional fine fractures visible breaking both crown and pavilion	Same fractures more prominent	Same fractures more visible; one breaking table surface appears iridescent in one area	Same fractures more visible	Same fractures more visible
1.88	Opticon	No apparent change	Some pavilion-breaking fractures appear as faint whitish lines	Same fractures slightly more visible	No additional change	No additional change	No additional change
3.11	Opticon	No apparent change	No apparent change	No apparent change	No apparent change	No apparent change	Three surface-reaching fractures noticeable on crown

held approximately 1/2 to 1 in. from the steam nozzle. After about five minutes, when the pressure had dropped to about 30–35 p.s.i., the stones were examined. The pressure in the unit was allowed to build to 60 p.s.i. again and the stones were steam cleaned for an additional five minutes. They were then re-examined, and the steam cleaning was subsequently repeated for a third five-minute period. During this testing, both the crown and pavilion surfaces were exposed to the steam jet for roughly equal periods of time.

The results of this testing for a 3.98-ct pale aquamarine treated with Canada balsam, a 1.75-ct pale aquamarine treated with cedarwood oil, a 2.83-ct Opticon-treated pale aquamarine, and a 3.24-ct colorless beryl treated with Opticon are reported in table 2. Although all four stones had poorer apparent clarity after the steam cleaning, the Opticon-treated stones seemed to have been affected the least. In all cases, the steam cleaning appeared to have removed more of the filling than did ultrasonic cleaning. Even the Opticon-treated stones, which are "sealed" at the surface, lost some filling material (figure 11); possibly, the prolonged attack at least partially broke through the hardened areas, creating exit points for the still-fluid Opticon. Again, although a jeweler would not normally steam clean a colored stone for five minutes or more, the results could represent the likely effects of multiple cleanings.

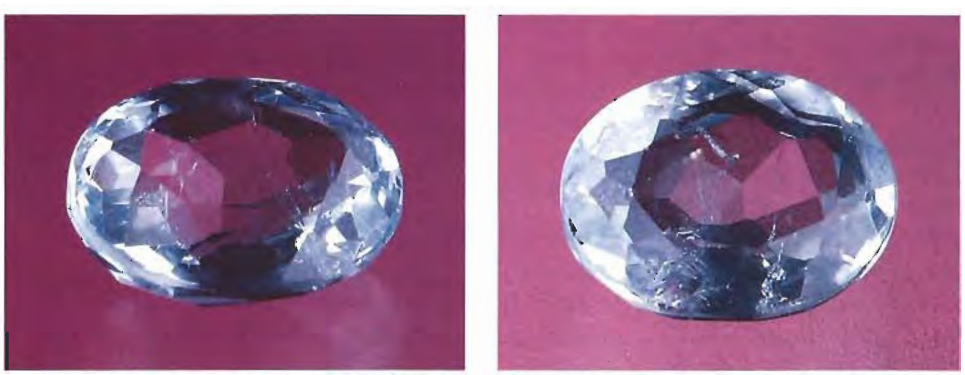


Figure 10. Before ultrasonic cleaning, no fractures were visible in this 1.78-ct cedarwood oil–treated aquamarine (left). Within 10 minutes in the ultrasonic, some of the oil had been removed; after 25 minutes, a multitude of fractures had become visible (right). Photos by Maha Smith.

Table 2. Results of the steam cleaning of "filled" pale aquamarines.

Weight (ct)	Treatment material	Results (minutes)		
		5	10	15
3.98	Canada balsam	Some filled fractures visible	Larger fractures more visible; all surface-reaching fractures had some filling removed	Some fractures more noticeable
1.75	Cedarwood oil	Large number of hairline-appearing fractures visible	Fractures more visible; slight "fuzzy" appearance to stone	"Fuzziness" more pronounced
2.83	Opticon	No apparent change	Several white hairline fractures visible on pavilion	Same fractures more visible
3.24	Opticon	One crown-breaking fracture visible	Three fractures visible	No additional change

REACTION OF OPTICON-TREATED STONES TO JEWELRY SETTING AND REPAIR

Another important durability consideration is the potential for damage to stones during jewelry setting or repair procedures. For example, a number of reports (e.g., Crowningshield, 1972; Themelis and Federman, 1990) warn bench jewelers that failure to detect filled fractures might result in excessive pressure being exerted on the stones during setting. Therefore, we performed a number of additional tests on Opticon-treated stones to determine how well they held up to other conditions that might be experienced.

Retipping Prongs. As mentioned above, Opticon is a very slightly yellow, almost colorless substance. During the treatment procedure—which included heating the Opticon-immersed stones in hot water—we noted a gradual darkening of the Opticon to a medium yellow after about two hours. This might not have a noticeable effect on the color of an emerald or other medium to dark stone that has been Opticon-treated. However, it could conceivably influence the appearance of a pale or colorless stone, although probably no more than would Canada balsam.

The retipping of prongs requires significantly higher temperatures. Although the retipping of prongs with an emerald in place is generally discouraged, it is occasionally done. There is also the risk that a stone other than emerald may have been fracture filled (see Box C). Therefore, we subjected a 2.40-ct Opticon-treated colorless beryl to the heat that would be generated in retipping prongs. To this end, the stone was secured in tweezers and held against a charcoal block. A 14k white hard solder with a 14k bead was then placed on a corner facet. The stone was evenly preheated with a #40 torch tip for approximately five seconds; then the torch was brought to bear on the solder until it flowed onto the bead, simulating retipping. This procedure caused the Opticon to flow out of fractures in the vicinity of the bead and

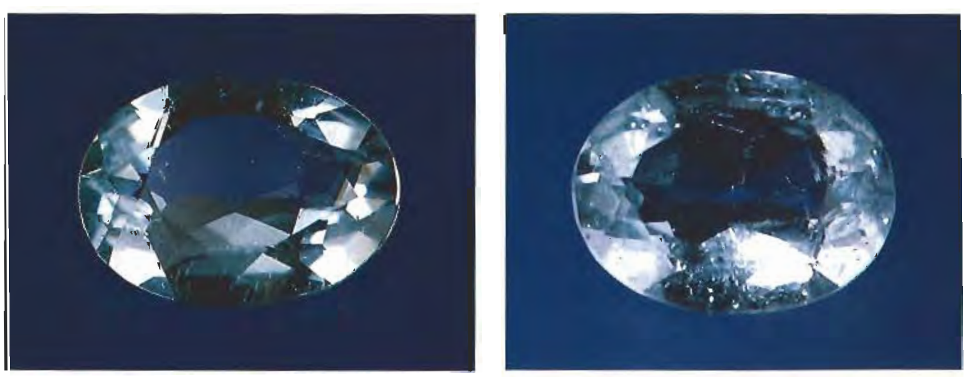


Figure 11. Before steam cleaning, no major fractures were evident in this 2.83-ct Opticon-treated aquamarine (left). Steam cleaning for 15 minutes resulted in partial removal of the Opticon, making the fractures readily apparent (right). Photos by Maha Smith.

produce a brownish residue on the surface of the stone (figure 12).

Metal Polishing. Both friction-generated heating and abrasive action take place during the polishing of metal prongs. To determine what, if any, negative effect this might have on a mounted stone with Opticon-filled fractures, a 1.20-ct treated colorless beryl was held in grooved locking tweezers, which were then "polished" for 30 seconds on a high-speed (3450 r.p.m.) 4-in. (10-cm) stitched muslin buff impregnated with tripoli compound. This had no effect on the appearance of the stone to the unaided eye. The procedure was then repeated, using a rouge buffing wheel impregnated with Bendick's rouge compound. Although we saw no effect with the unaided eye, magnification revealed some extremely fine whitish areas along a large filled fracture that broke the surface of the stone close to where the metal had been polished

BOX C: OPTICON AND OTHER GEMS

Opticon has also been recommended for treating cracks in fashioned stones and mineral specimens of agate, quartz, beryl, topaz, tourmaline, and "any other transparent hard gem with a refractive index close to Opticon" (Jones, 1986). From time to time, the GIA Gem Trade Laboratory has detected fillings in fractures in other faceted gemstones—including pink, green, and blue tourmalines, blue sapphire, and green zoisite—although the feeling is that these usually have been treated with oils. To explore the effectiveness of Opticon in filling fractures in other gem materials, the authors induced fractures in three (1.88, 2.83, and 4.54-ct) faceted, light greenish blue aquamarines; a 7.58-ct faceted medium purple amethyst; a 2.59-ct dark green tourmaline cabochon; a 2.87-ct faceted light, slightly greenish blue synthetic spinel; and a 3.34-ct faceted medium-dark green-blue synthetic spinel. Also treated were two essentially colorless cat's-eye tourmaline cabochons of 1.81 and 3.68 ct.

The improvement in appearance was most noticeable in the faceted aquamarines (see, e.g., figure C-1) and amethyst (figure C-2); fractures that showed high relief before treatment could not be detected

Figure C-1. The fractures in this 4.54-ct aquamarine were readily apparent before Opticon treatment (left) and almost invisible afterward (right). Photos by Maha Smith (left) and © GIA and Tino Hammid (right).

Figure C-2. Because its refractive index is close to that of Opticon, quartz also responds favorably to filling with this substance, as seen in this 7.58-ct amethyst before (left) and after (right) treatment. Photos by Maha Smith.

(figure 13). Apparently, the hardened Opticon had been abraded by the action of the wheel.

Ultraviolet Radiation. Another concern is the stability of the filling material itself. According to one report (Themelis, 1990), the various materials used to fill emeralds, including Opticon, are not stable to light unless various "plasticizers" are used. As one test to determine the stability of Opticon, an Opticon-treated near-colorless beryl was exposed to short-wave ultraviolet radiation from a 4-watt fluorescent lamp for approximately 36 hours. This appeared to have no effect on the filling material.

DISCUSSION

Opticon would seem to offer some advantages over Canada balsam and cedarwood oil for filling fractures in emeralds. Because it is closer in refractive index to emerald than either of the other sub-

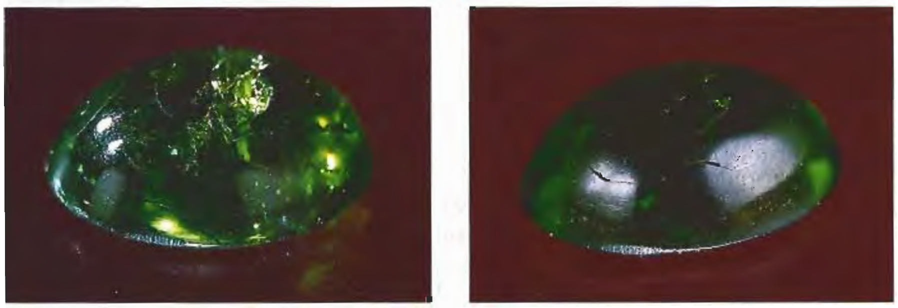


Figure C-3. Even though the refractive index of tourmaline is somewhat higher than that of Opticon, treatment of this 2.59-ct tourmaline (left) still produced significant improvement in the apparent clarity of the stone (right). Photos by Maha Smith.

with the unaided eye after filling. This is to be expected, as the refractive indices of these two gem materials (aquamarine = 1.560–1.566 and amethyst = 1.544–1.553) are quite close to that of Opticon (1.545). The R.I. of tourmaline, about 1.624–1.644, is further from that of Opticon; even so, the improvement here, too, was very good (figure C-3). Only when the difference in refractive index between the gem and the filling material was considerably greater, as in the case of the synthetic spinel (R.I. 1.728; figure C-4), did the fractures remain visible to the unaided eye. Yet even in this case, we saw an overall improvement in apparent clarity. The filling of the growth tubes in the cat's-eye tourmalines caused these to become much less noticeable, so that the stones appeared more transparent after filling.

All features of filled breaks in the aquamarines were consistent with those previously noted in emeralds. This was also the case with the amethyst, with the exception that overhead fluorescent lighting revealed blue but no orange flashes from many of the filled breaks (both colors were noted under darkfield conditions). Irregular gas bubbles, but no dispersion colors, were noted in filled fractures and filled growth tubes in the green tourmaline. Both darkfield and overhead illumination clearly revealed the outlines of the filled breaks in both synthetic spinels; again, no flash effects were noted.

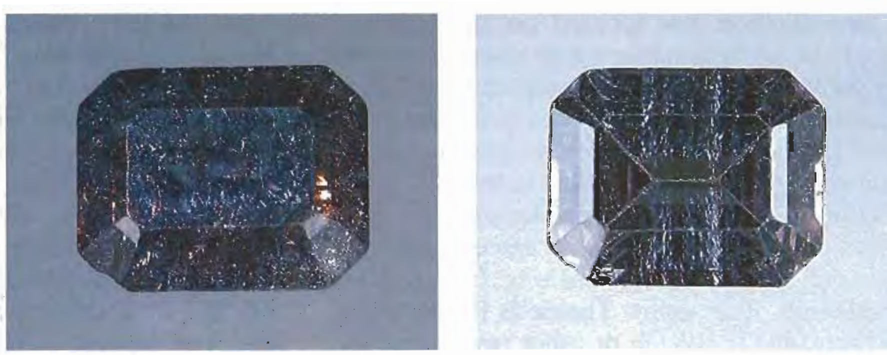


Figure C-4. Because the refractive index of spinel is considerably higher than that of Opticon, treatment is less effective. Many of the fractures visible in this 3.34-ct stone before Opticon treatment (left) were still apparent after treatment (right). Photos by Maha Smith.

Figure 12. When this Opticon-treated beryl was exposed to the heat of a jeweler's torch during a procedure approximating the retipping of prongs, Opticon boiled out of some of the fractures and darkened, leaving a brown residue on the surface. Photomicrograph by John I. Koivula; magnified 30×.

Figure 13. Rapid polishing of the prongs that held this Opticon-treated beryl resulted in abrasion of the hardened Opticon at the surface of the stone. Photomicrograph by John I. Koivula; magnified 50×.

stances, it may be more effective in masking surface-reaching breaks. Detection of any of these treatments is best accomplished using magnification with a variety of lighting techniques.

With respect to durability, extended ultrasonic and steam cleanings appear to remove less filling material from sealed, Opticon-filled breaks than from breaks filled with either Canada balsam or cedarwood oil, indicating that the former is a more durable treatment.

The long-term durability of Opticon-filled fractures is not known at this time. It is possible that the sealing of filled fractures at the surface of a stone may inhibit or retard alteration/decomposition of the filler itself by preventing direct exposure to air, airborne caustic agents, or solvents. Such sealing at the surface might also extend the effective life of other fracture-filling substances.

CONCLUDING COMMENTS

This investigation has focused on the fracture filling of emeralds using the epoxy resin marketed under the brand name Opticon, which is sealed at the surface of the stone to which it has been applied, and has compared this to fracture filling using cedarwood oil and Canada balsam.

However, it appears that a number of substances—or combinations of substances—are now being used to fill surface-reaching fractures in emeralds and other gems. Themelis (1990) mentions that Canada balsam or other resins may be mixed with cedarwood oil or other oils. Various other oils have also been used, including castor, coconut, corn, linseed, lubricating, mineral, Neatsfoot, olive, palm, peanut, rapeseed, soybean, tung, and whale (Nassau, 1984). In the course of this investigation, the authors experimented with additional substances for fracture filling, all of which improved the apparent clarity of the treated stones to some extent; two in particular, a U.V.–curing glue and a polyester casting resin (the latter, R.I. of 1.555), were very effective.

More recent reports note that an apparently new type of low-polymer epoxy resin is being used to treat the majority of emeralds being imported into Japan from Colombia ("Fluid Epoxy Resin Reported in Emerald Fractures," 1991; "Filled Emeralds," 1991). The substance, referred to as "palm oil," reportedly has an R.I. of 1.57 and exhibits trapped bubbles and weak bluish green to orangy red dispersion colors. The reports also mention emeralds filled with a cyano-acrylic that exhibits a white brushmark-like appearance in fractures. A third filling substance mentioned is a hardened epoxy resin that exhibits very few bubbles and no dispersion colors.

As mentioned at the beginning of this article, we do know of other, proprietary treatments being used on emeralds, including the one offered by the firm that pioneered what has become known colloquially as the "Yehuda" procedure for filling fractures in diamond; another, offered by CRI Laboratories of Grand Rapids, Michigan, which has received widespread notice in the trade press; and a third, being offered commercially in Japan. The investigators are in the process of examining stones treated by these processes; reports on these findings will be published in the second part of this series.

What appears obvious at this stage in our investigations, however, is that there is considerable overlap in the gemological features of filled surface-reaching breaks regardless of the substances used. It would, therefore, seem both inappropriate and misleading, in describing a filled fracture, to use wording that implies that the filling substance has been conclusively identified if in fact it has not. It would also seem unwise to use the term *oiling* as a general term to refer to emeralds with filled surface-reaching breaks. Instead, a generic term referring to the treatment process would be better advised. The general process so described herein is fracture filling. The GIA Gem Trade Laboratory uses the following statement, as appropriate, on identification reports: "Note: Foreign material is present in some fractures reaching the surface."

The GIA Gem Trade Laboratory has periodically encountered gem materials other than emeralds and diamonds that have been fracture filled. It would seem prudent, therefore, to keep in mind the possibility of filled surface-reaching breaks when examining any gem material.

While Opticon-filled breaks appeared to withstand both ultrasonic and steam cleaning better than breaks treated with either cedarwood oil or Canada balsam, prolonged exposure to either cleaning method adversely affected stones treated with any one of these three substances. It would thus appear inadvisable to use these procedures when such fillings are present or even suspected (as is believed to be the case with most emeralds). High temperatures and surface abrasion, as associated with some jewelry repair procedures, were also shown to negatively affect Opticon-filled stones. It would thus seem prudent to remove stones suspected of treatment from their mountings whenever possible before repair procedures are attempted.

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