AN UPDATE ON FILLED DIAMONDS: IDENTIFICATION AND DURABILITY

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The increasing numbers of fracture-filled diamonds present a major challenge to the diamond industry, especially with regard to detection and durability of the treatment in routine jewelry manufacturing and wear. This report focuses on recent products from Yehuda/Diascience, Koss & Shechter Diamonds (Genesis II), and Clarity Enhanced Diamond House (a subsidiary of Goldman Oved Diamond Co.). Like the Yehuda treatment, the latter two processes were found to be effective in improving the appearance of most of the samples examined for this study. Treated diamonds from all three firms were damaged by direct heating and by repolishing facets intersected by filled breaks. Some stones were adversely affected by some standard cleaning procedures and wear conditions. Although the lead-based glass filling materials may be detected by X-radiography and EDXRF spectroscopy, as well as by certain internal features, we found flash effects to be the most distinctive characteristic of fracture filling—observed in all the treated diamonds examined from all three firms.

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One of the most controversial gemstone treatments to appear in the last decade is the filling of surface-reaching breaks in faceted diamonds. Jewelers, *diamantaires,* and gemologists at all levels of the industry must deal not only with the detection and durability of this treatment, but also with how properly to disclose the treatment to their customers. The trade has already been confronted with media exposés about misrepresentation in the sale of filled diamonds by some U.S. retailers (see, e.g., "Five on Your Side . . .," 1993; "Prime Time Live . . .," 1993).

Because more of these stones are now entering the market, it is increasingly likely that jewelers will encounter them in their day-to-day operations. Yet the challenge of identifying filled stones and working with them at the bench is further complicated by the fact that a growing number of firms are producing and/or marketing them, as loose stones and even in fashioned goods (figure 1). Thus, the present study was undertaken to investigate fracture-filled[1] diamonds from two well-publicized diamond treatment firms that began marketing these stones after publication of our original comprehensive study (Koivula et al., 1989), as well as recently treated stones from the firm (Yehuda) that we studied initially. The present article can be viewed as an update to our 1989 study, as we again focus on the issues of identification and durability.

BACKGROUND

The first commercially available diamond fracture-filling treatment was developed in the 1980s by Zvi Yehuda of Ramat Gan, Israel, with diamonds treated by this process marketed by Yehuda Diamond Co./Diascience Corp., New York. Briefly stated, this technique proceeds as follows: The diamonds are first cleaned, then "filled" with a molten glass at high temperatures (presumably under vacuum, to prevent diamond burning), cooled, and—last—cleaned again to remove the glass from the stones' surfaces (Nelson, 1994;

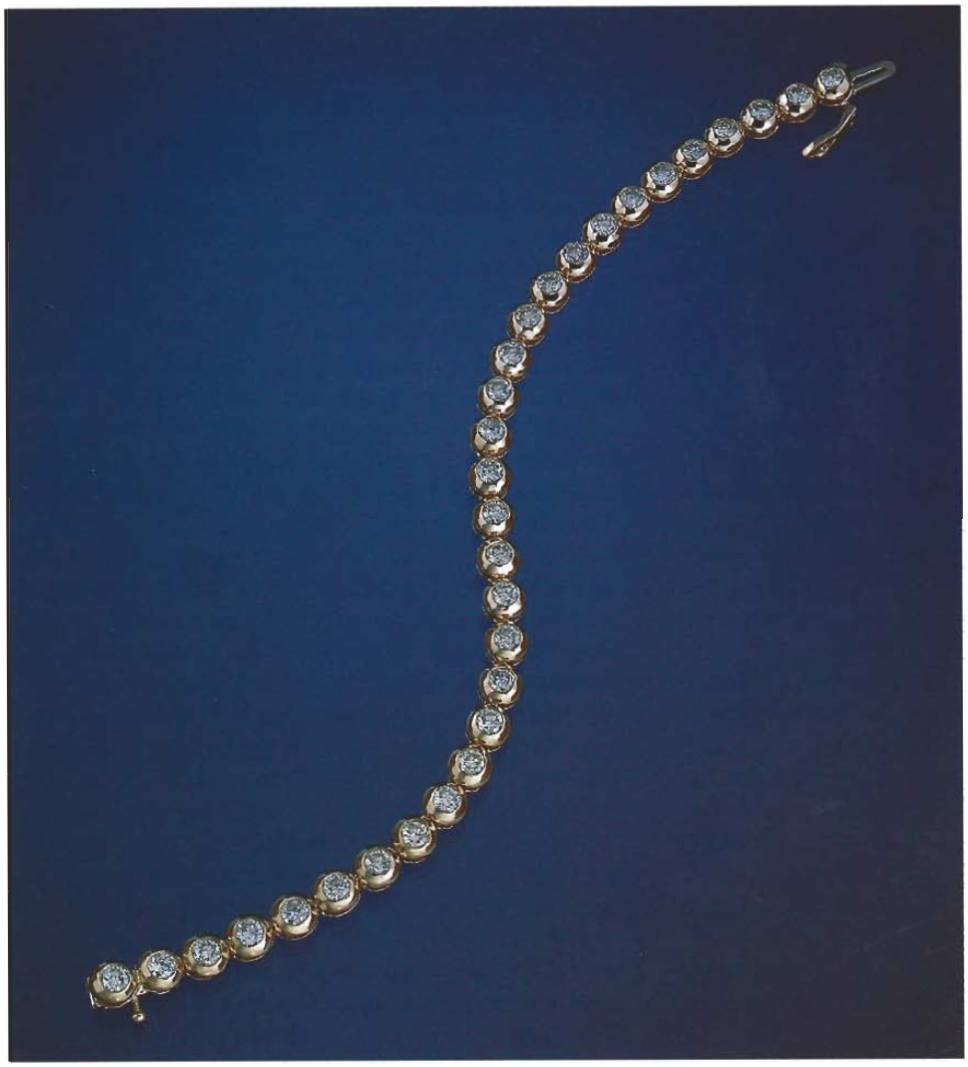


Figure 1. All of the 31 diamonds (about 0.13 ct each) in this strip bracelet had been fracture filled by Goldman Oved Diamond Company, New York, which also manufactured the bracelet. Photo by Shane F. McClure.

Nassau, 1994). Koivula et al. (1989) focused on the effectiveness of the Yehuda treatment, diagnostic features by which it could be detected using standard gemological equipment, and the durability and stability of diamonds treated in this manner. Results of a follow-up investigation of Yehuda-treated diamonds were briefly summarized by Koivula and Kammerling (1990).

Since publication of these reports, fracture filling has grown to become one of the first truly widespread treatments to be used on diamonds in the colorless-to-light yellow range, and it is also being used on fancy-colored diamonds. Even so, many jewelers still may not think to examine diamonds to determine if they are fracture filled.

Although it is virtually impossible to determine exactly how many such treated stones are in the marketplace, a number of statements indicate the magnitude of the situation. For example, in an advertisement for Yehuda-treated diamonds ("Yehuda diamonds offer great option . . .," 1994), jeweler Lloyd Drilling of Thurston Jewelers in Minneapolis, Minnesota, estimates that 20%–30% of the diamonds he sells are so treated. (When contacted by the authors in September 1994, Mr. Drilling clarified that this amounts to 30 filled diamonds annually, between 0.5 and 2.5 ct each.) In the same advertisement, Harris Fleishman of C. Harris Goldman in New York reports that over the last five years, Yehuda-treated diamonds have grown to account for about 50% of all diamonds sold by his firm. (According to our September 1994 personal communication with Mr. Fleishman, this represents about 3,000 filled stones per year.) He

[1] *Throughout this article, we use the trade term* fracture filled *to describe diamonds in which surface-reaching separations such as cleavages, fractures, voids, laser drill holes, and other partings have been filled with a foreign substance. Although the "fracture filling" designation may not be strictly accurate, it is both widely used and understood in the trade.*

further estimates that 75% of all diamond merchants on 47th Street handle at least some "clarity-enhanced" stones. In mid-1993, Dror Yehuda indicated that "tens of thousands" of diamonds treated by his family's firm were already in the U.S. market (Brown, 1993).

It appears that these Yehuda-treated stones represent some of the larger filled diamonds on the market. According to Ron Yehuda (pers. comm., 1994), his family's firm prefers not to treat clients' stones smaller than 0.25 ct, and the number of such stones under 0.50 ct that the firm has treated is "negligible." Already-treated stones in the company's sale stock are primarily 0.50 ct and larger, with fully 50% of demand being for stones over 1 ct. He adds that these stones are marketed primarily in the U.S. and Canada, the two countries in which the firm has focused its efforts to date.

The proliferation of filled diamonds can also be explained by the fact that several other firms now offer commercial treatment services and/or diamonds already so treated. Two of these appear to be especially visible in the marketplace. Genesis II—Enhanced Diamonds Ltd., in New York, a division of the Israel-based firm Koss & Shechter Diamonds Ltd., offers its own product. Originally described in the firm's marketing literature (and often referred to in the trade) as "Koss clarity-enhanced diamonds," it has more recently been marketed in the U.S. under the trademark name of "Genesis II" clarity-enhanced diamonds ("A new stone is born . . .," 1994)—the name under which it was first marketed in Australia. In terms of the numbers of these stones on the market, a startling figure is provided by Managing Director Daniel Koss, who reports that in a three-year period his firm had treated "over half a million stones from 0.01 ct to 50 cts" (Shor, 1994). We have examined Koss-treated stones as small as 0.02 ct.

The other firm, Clarity Enhanced Diamond House, a division of Goldman Oved Diamond Company, New York, offers treatment services and also sells treated stones; the treatment is performed in Israel and New York (J. Oved, pers. comm., 1993). Goldman Oved has treated stones between 0.02 and 15.5 ct (J. Oved, pers. comm., 1994), and we have examined Goldman Oved–filled diamonds as small as approximately 0.13 ct (figure 1).

Other firms, such as Chromagem of New York, operate on a relatively smaller scale, selling filled diamonds that are treated by an independent chemist. Still other New York diamond treaters (such as S&I Diamond Drilling) provide filling services to the diamond trade only. Firms in other cities, such as Diamond Manufacturers in Los Angeles, are now treating diamonds (U. Uralevich, pers. comm., 1994). In addition, there are wholesale firms that market to the retail trade (in some instances, under their own trade names) diamonds treated only by others. For example, Doctor Diamond, a division of Kami & Sons, New York, markets "Doctor Diamond clarity enhanced diamonds" ("Clearly better . . .," 1994), which reportedly include diamonds treated by both the Yehuda and Goldman Oved firms (J. Oved, pers. comm., 1993).

In part because so many companies are now offering treated stones, some confusion has developed about the identifying features of filled diamonds. For example, claims have been made that Koss-filled diamonds exhibit little or no telltale flash effects (Shor, 1994); that the presence of certain flash-effect colors—purple, orange, blue, green, and red—identify a filled stone as being Yehuda treated (*Canadian Jeweller*, 1994); and that the optical properties of the Koss filler are such that the color of the diamond is unaffected, any cracks disappear completely (with no bubbles trapped inside the filler), and rays of light travel through the diamond "with no distortion or deflection whatsoever" (1994 Koss promotional brochure).

Claims have also been made concerning the stability and durability of various products. For instance, recent product literature by Koss states that Koss clarity-enhanced diamonds withstand temperatures to 450°C, acid-based cleaning, and "ultrasound treatment." Controversy has also arisen in this area, with recent research findings by gemologist Sharon Wakefield of Boise, Idaho, indicating that the filling material used in at least one of the treatment processes (Koss) may decompose when exposed to a short-wave ultraviolet lamp or ultrasonic cleaning (Wakefield, 1993, 1994a; Even-Zohar, 1994b). It is also relevant to consider what Quam (1993) has referred to as the "longer historical perspective": that is, with the passage of time, the durability of the filling materials may prove to be less than had been believed initially. Some additional confusion—and controversy—about the identification and durability of filled diamonds may be a consequence of the claims and counterclaims made in the trade press by firms providing treatment services (see, e.g., Yehuda, 1993, 1994a,b; Koss, 1993, 1994a,b).

With the proliferation of filled diamonds in the market, concerns have been voiced at all levels of the industry. The topic of diamond treatments—and their proper disclosure—was a major focus of discussions during the biannual congress of the International Diamond Manufacturers Association (IDMA) and the World Federation of Diamond Bourses (WFDB) in Antwerp in June 1993 (Rapaport, 1993; Bates, 1993a; Shor, 1993). In late 1993, as the result of a vote taken by its board of directors, the Diamond Club West Coast in Los Angeles issued a statement asking all major grading labs not to grade filled diamonds ("Labs asked . . .," 1994; Shapiro, 1994). A similar resolution was passed by the combined leadership of the IDMA and WFDB in Antwerp in June 1994 ("Diamond leadership . . .," 1994). Also at this latter meeting, a resolution passed that prohibits the filling of rough or the selling of filled rough (Even-Zohar, 1994a). Concern about nomenclature relating to filled diamonds has been voiced by the Diamond Manufacturers and Importers Association, which has asked the trade to refer to them as "treated" rather than "enhanced" (Roisen, 1994; Bates, 1994a).

Fracture filling was also addressed at the annual meeting of the International Confederation of Jewellery, Silverware, Diamonds, Pearls and Stones (CIBJO) in April 1994, the main topic of discussions being treatment disclosure (Bates, 1994c; "Annual CIBJO Conference," 1994). Joel Windman (1994), executive vice president of the Jewelers' Vigilance Committee, has warned jewelers of their potential liability if they do not disclose to customers both that a diamond has been fracture filled and that, as such, there may be durability considerations beyond those of untreated diamonds. Some of the best-known U.S. retail chains—including Zale Corp., Sterling, Carlyle & Co., Karten's Jewelers, and Helzberg's—and at least one major jewelry manufacturer (Suberi Brothers) have notified their suppliers that they will not accept filled diamonds (Bates, 1994b; Beasley, 1994; Shor, 1994; "Manufacturer places burden . . .," 1994). Jack Gredinger, president of the Independent Jewelers Organization (IJO), has gone so far as to call the treatment "an infectious disease that undermines jewelry retailers and wholesalers. It's the single most serious and important problem our industry has faced in years" ("IJO takes stand . . .," 1994). IJO has asked its member suppliers not to sell fracture-filled diamonds (Shuster, 1994).

This concern about fracture filling became a public issue in the United States when an exposé televised locally in August and September 1993 accused two St. Louis jewelers of selling filled diamonds without disclosing the treatment to customers ("Five on Your Side . . .," 1993). The report was subsequently broadcast nationwide ("Prime Time Live . . .," 1993), and was even addressed by a member of the U.S. House of Representatives (Everhart, 1993b). The exposé affected jewelers who were not involved in the incident (Everhart, 1993b–d; Bates, 1993b). Such problems have not been confined to the U.S. market: Last year, isolated instances of filled diamonds being sold without proper disclosure were reported in a number of countries, including the United Kingdom (Levy, 1993; Shor, 1993/1994) and Australia (Kusko, 1993/1994).

The current article presents the results of research on recent production from three of the most prominent commercial sources: Yehuda, Koss, and Goldman Oved. (Note: For consistency and simplicity, in the following discussions we will refer to the filled diamonds from Yehuda/Diascience as "Yehuda-treated" [or just "Yehuda"] stones, to the Koss & Shechter/Genesis II product as "Koss-treated" [or "Koss"]; and to the Goldman Oved/Clarity Enhanced Diamond House product as "Goldman Oved–treated" [or "Goldman Oved"] diamonds.) We will examine the effectiveness of these treatments, and provide techniques for identifying them. Of particular interest are the results of testing the durability and stability of some treated stones. As will also be discussed, the GIA Gem Trade Laboratory is continuing its policy of not grading such treated diamonds.

MATERIALS AND METHODS

Samples. For our primary gemological investigation, we obtained 67 diamonds that had already been filled: 18 Yehuda-treated diamonds, ranging from 0.31 to 1.68 ct; 24 Koss-filled diamonds, ranging from 0.02 to 0.82 ct; and 25 Goldman Oved–treated diamonds, ranging from 0.18 to 1.91 ct. Some of these were purchased by GIA directly from the manufacturer; others were obtained through third parties directly from the manufacturer. We also examined 31 Goldman Oved–treated diamonds that were mounted in a tennis bracelet loaned to the authors by Goldman Oved (again, see figure 1). These mounted diamonds were all approximately 3.3 mm in diameter and were estimated by formula to weigh about 0.13 ct each.

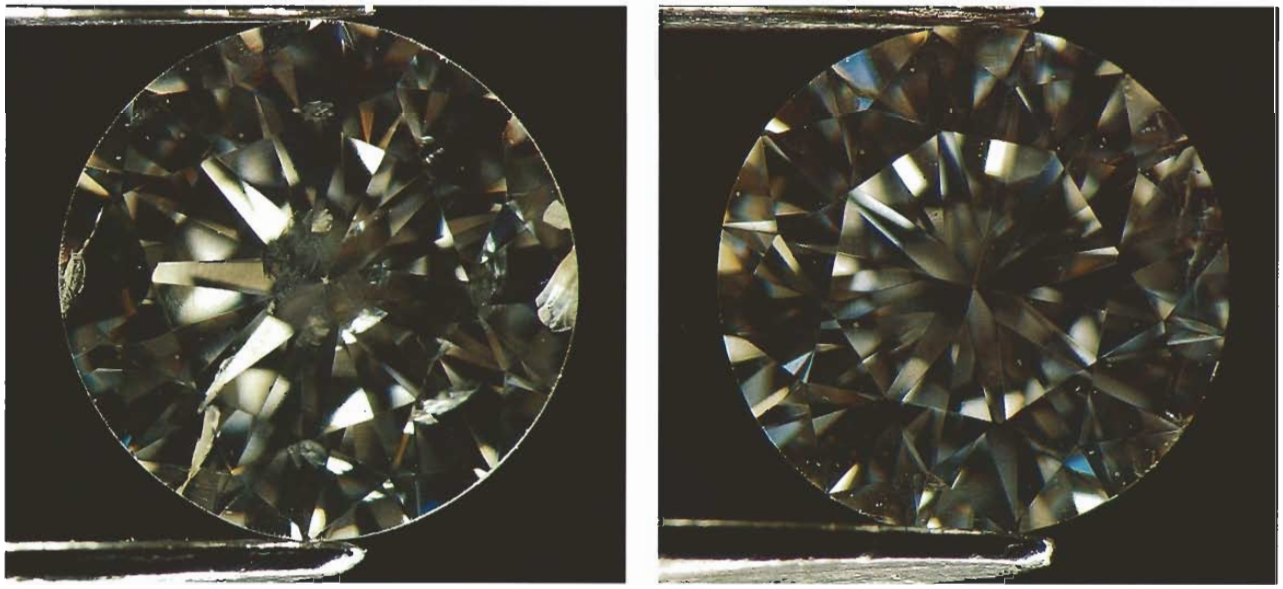


Figure 2. This 0.27-ct diamond (unfilled, left) was submitted to the Koss firm for treatment in 1992. It shows a noticeable improvement in appearance (right) after treatment. Photomicrographs by John I. Koivula.

X-radiography was performed on some of the same diamonds referred to above: three (0.32–0.40 ct) stones treated recently by the Yehuda firm, three Koss-filled diamonds (0.10–0.82 ct), and three Goldman Oved–treated diamonds (0.20–0.41 ct). EDXRF chemical analysis was performed on eight stones recently treated by Yehuda, five early Koss-treated stones, six commercially available Koss diamonds, and 11 Goldman Oved–treated diamonds.

In addition to the core 67 stones, we submitted five untreated diamonds (0.24–0.28 ct) in 1992—and nine (0.20–0.84 ct) in 1994 (see box A)—for treatment by Koss so that we might document them before and after filling, both photographically (figure 2) and for apparent color and clarity grades. The untreated stones were specifically chosen with fractures in certain orientations and positions. Six diamonds (0.20–0.41 ct) treated by the Goldman Oved process in 1994 were also photographed and "graded" for clarity and color before and after treatment (figure 3). All of these Goldman Oved stones were also examined in our primary gemological investigation.

We used selected treated diamonds for durability and stability testing. Three filled stones—a 0.31-ct Yehuda, a 0.29-ct Koss, and a 0.36-ct Goldman Oved—were subjected to steam cleaning. Ultrasonic cleaning was performed on 0.34-ct Yehuda, 0.29-ct Koss, and 0.32-ct Goldman Oved diamonds. Sizing and, subsequently, retipping were performed on mountings containing 0.37-ct Yehuda, 0.32-ct Koss, and 0.19-ct Goldman Oved diamonds. We subjected three filled diamonds (a 0.36-ct Yehuda, a 0.30-ct Koss, and a 0.36-ct Goldman Oved) to thermal testing in a furnace, and we had one treated diamond from each firm (a 0.37-ct Yehuda, a 0.45-ct Koss, and a 0.36-ct Goldman Oved) repolished. For ultraviolet testing, we selected two filled stones from each of the three firms: Yehuda (0.35 and 0.38 ct), Koss (0.29 and 0.30 ct), and Goldman Oved (0.29 and 0.38 ct). We exposed 0.32-ct Yehuda-, 0.32-ct Koss-, and 0.35-ct Goldman Oved–treated diamonds to daylight-equivalent illumination. Low-temperature testing was performed on 0.36-ct Yehuda-, 0.29-ct Koss-, and 0.20-ct Goldman Oved–filled diamonds. For durability and stability testing,we only used commercial stones obtained from the treaters directly or through third parties. We did not use any of the diamonds that were documented before and after filling.

Gemological Methods. A standard gemological microscope is sufficient to identify most fracture-filled diamonds. For this study, we performed microscopy using GemoLite Mark VII gemological microscopes with a 10×–63× magnification range, in conjunction with several illumination methods (separately and combined): darkfield, brightfield (direct transmitted), pinpoint fiber-optic, oblique overhead, and shadowing. With the exception of fluorescent oblique overhead lighting, all examinations used incandescent light sources. Where specific types of lighting are critical to the resolution of microscopic features, they are described in the appropriate portion of the "Microscopic Features" section below. Other standard gemological tests—for example, ultraviolet fluorescence and visible-light spectroscopy—were explored but found to be of no help in detecting the treatment.

Figure 3. This 0.30-ct diamond, which had highly visible breaks before fracture filling (left), showed significant improvement in apparent clarity—from I_3 to I_1—after treatment by Goldman Oved (right). Photomicrographs by Shane F. McClure.

Laboratory Methods. We used X-radiography and energy-dispersive X-ray fluorescence (EDXRF) chemical analysis, which require equipment usually found only in gem-testing laboratories, both to determine if these methods would reveal the presence of a filling and to learn more about the composition of the filling substances used.

Detection of filler by X-radiography depends on three factors: the thickness of the filler; the exposure geometry (we recommend X-raying diamonds in at least two mutually perpendicular orientations to increase the likelihood of positioning a filled fracture so that it can be detected); and experimental conditions such as film resolution, X-ray source intensity, and the like. For this study, X-radiography was performed using a Hewlett-Packard Faxitron unit with a tungsten anode and beryllium window, run at 30 to 60 kV and 2.5 mA. The distance from the X-ray tube window to the target was approximately 15 inches (38 cm). We used Fuji IX50 photographic film, with exposure times in air ranging from a few to about 30 seconds. In our experience, thin or subtle fillings are more likely to be seen on the developed film when lower energy conditions and longer exposure times are used.

For qualitative chemical analyses, we used a Tracor X-ray Spectrace 5000 EDXRF spectrometer, with operating conditions appropriate for the detection of heavy elements (35–40 keV, 0.35 mA, pumped vacuum, and 1.27-mm-thick rhodium filter). Light elements that might be constituents of the filler glass, such as boron and oxygen, cannot be detected by this method.

Durability Testing. The stones used in this phase of the investigation were obtained both directly and indirectly from all three treaters in 1994, to ensure that we tested the most current commercial products available. For these tests, we attempted to use equipment available to most jewelers. The mention of a particular brand of equipment here does not mean that other brands will not give comparable performance; nor should it be taken as an endorsement.

Steam Cleaning. We used two commercially available units: a Gesswein electric boiler and a Reimers model JR electric steam generator. The filled diamonds were first mounted in a four-prong head on a 14k gold solitaire ring and then subjected to cleaning at a distance of about one inch (2.5 cm) from the steam nozzle. Each diamond was directly exposed to steam for a total of 20 minutes at one-minute periods; all of the surfaces of each stone were exposed to the steam as they might be during a routine cleaning procedure. The intervals between steam exposures were just long enough for the steam pressure to build in the cleaning unit for the next exposure; pressure was maintained between 40 and 70 psi. We examined the diamonds visually—both with and without magnification—at five-minute intervals.

Ultrasonic Cleaning. We used a Gesswein Ultrasonic Cleaner model 87 containing BRC, a standard jewelry-cleaning solution. The unit was set at "high" and the heating element was turned on

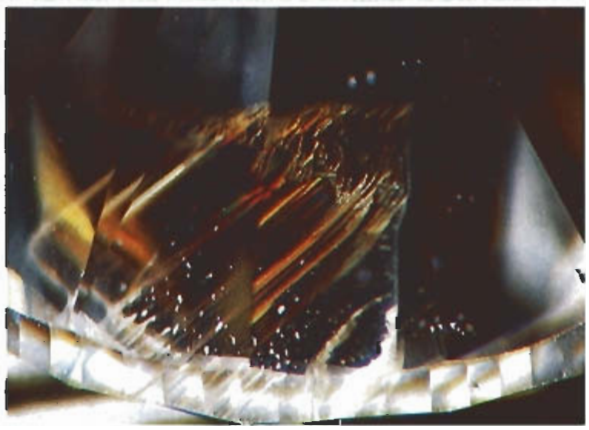
BOX A: Fracture Filling Is an Evolving Field

Figure A-1. Although the Koss firm considered the fractures in this diamond to be less than ideal for filling, the treatment was still quite effective in improving the stone's appearance, as can be seen in these photos of a 0.20-ct diamond before (left) and after (right) treatment. Photomicrographs by Shane F. McClure.

The stones that we examined in the body of this study may not be representative of all fracture-filled diamonds in the market now or in the future. The technology for fracture filling continues to evolve as producers try to improve their products.

So that the samples used for this study would reflect the most current technology, David Shechter of Koss & Shechter Diamonds attempted to treat additional diamonds for us in mid-1994, reportedly using two processes. One, based on halogen glasses, is the commercial process presently being used; the other, based on halogen-oxide glasses, is in an experimental stage (D. Shechter, pers. comm., 1994). Not all of the approximately 20 stones originally submitted for this phase of the project successfully took the treatment: For instance, five were returned by the firm untreated, with explanations that either (1) "the crack is too thick," or (2) "the fissure reaching the surface is broken" (D. Shechter, pers. comm., 1994; we interpreted the latter comment to mean that the surface-breaking entry point was a cavity rather than a narrow fracture). According to Mr. Shechter, his firm cannot get satisfactory results on diamonds with such features.

Figure A-2. Unlike the vast majority of the Koss-filled diamonds acquired for this study, this stone (submitted by the authors directly to Koss for treatment) displays orange and yellow flash colors in darkfield illumination. Photomicrograph by Shane F. McClure; magnified 29×.



Three round brilliants, ranging from 0.20 to 0.41 ct, were successfully filled by the first (halogen-based) process (figure A-1). Two other round brilliants (0.38 and 0.43 ct) were not filled successfully (i.e., completely) by this process but were included in the examination. As a group, these diamonds showed microscopic features that were inconsistent with those documented in Koss-filled stones purchased previously (including earlier in 1994) for the identification and durability-testing phases of this study.

The atypical features, as seen with magnification, are as follows: (1) *Flash effects:* Only orange and yellow were seen with darkfield illumination (figure A-2), and only blue and violet were seen with brightfield (figure A-3). (2) *Gas bubbles:* All of these stones exhibited gas bubbles which, unlike the other Koss-treated stones we examined, were relatively large and numerous. (3) *Flow structure:* Two of the five diamonds exhibited prominent flow structures in the filled breaks. (4) *Apparent color of filler:* A definite yellow cast was noted in some of the filled breaks. (5) *Crackled texture:* The fine, nearly parallel lines noted in the other Koss-treated diamonds were absent in this group of stones. In fact, as a group, the internal features in these diamonds were more reminiscent of those documented in early Yehuda-treated diamonds than those we have seen in other Koss-filled diamonds. However, EDXRF analysis revealed that all these stones, even the unsuccessfully (partially) filled ones, contain Pb and Br (i.e., unlike Yehuda-filled diamonds; refer to box C).

Four round brilliants (0.23 to 0.44 ct) were pro-

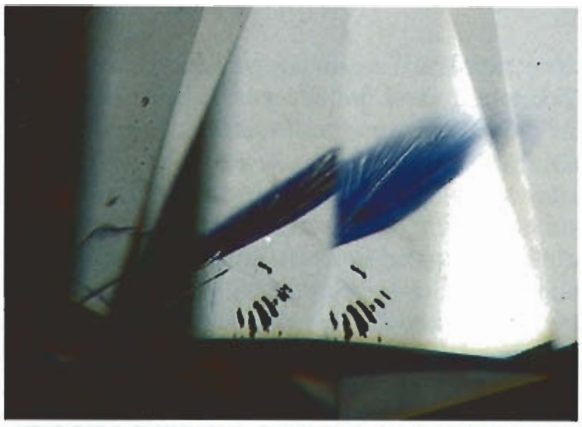


Figure A-3. Also unlike most other Koss-filled diamonds examined as part of this study, the brightfield flash colors in this group of stones (which were sent directly to Koss for treatment) were blue and violet. Photomicrograph by Shane F. McClure; magnified 33×.

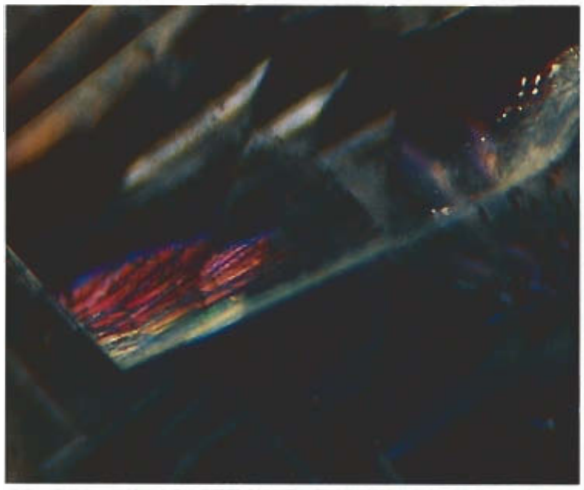


Figure A-4. The authors had not seen red flash effects in filled diamonds until they examined stones treated by S&I Diamond Drilling, as shown here. Photomicrograph by Shane F. McClure; magnified 30×.

cessed using the second (experimental) technique, but Mr. Shechter did not consider any of these to be filled successfully. Of these stones, the ones displaying any evidence of filled breaks had microscopic features consistent with those in the group described above. These also showed traces of Pb and Br in their EDXRF spectra.

Recently, it was reported in the trade press that the Koss firm was experimenting with additives to make the filling easier to detect by causing it to fluoresce yellow to ultraviolet radiation ("Koss to make fill more visible," 1994). However, no such fluorescent reaction—to either long- or short-wave UV—was noted in any of the Koss-treated diamonds, including the nine stones reported in this section. Even using cathodoluminescence, we detected no reaction from any of the filled diamonds.

To test the effectiveness of these treatments, we graded these nine stones before and after treatment, with results shown in table A-1. As can be seen from the table, these treatments improved the apparent clarity by one grade in three of the diamonds. However, they also *lowered* the apparent color by one grade in four of the nine stones, including all where treatment was considered "successful."

To get a better idea of what happens when the treatment is not successful, and to obtain some preliminary data on another treater's product, we selected eight diamonds that had been rejected as unsuitable by various diamond treaters. These were processed by Ivan Perlman of S&I Diamond Drilling, who provides diamond drilling and treatment services exclusively to the trade. These diamonds, ranging from 0.30 to 0.52 ct, were photographed and graded for clarity and color before and after treatment.

Although none of these diamonds was successfully (i.e., completely) fracture filled, we were able to document some of the features of stones treated by this process. The one relevant microscopic feature after treatment was a flash effect. In darkfield, all but one of the stones showed one or more of the following colors: orange, pink, yellow, blue, purple and red (figure A-4), with orange and pink being the most prevalent. In brightfield, the flash colors noted were blue-green, green, and greenish yellow. It should be noted that this was the first time we saw a red flash effect in a filled diamond from any treater.

TABLE A-1. Apparent color and clarity of nine round brilliant-cut diamonds before and after filling by Koss & Shechter Diamonds using two processes.[a]

Sample no.	Measurements (mm)	Weight (ct)	Before		After	
			Color	Clarity	Color	Clarity
Method 1						
1–1	4.84–4.89 x 2.90	0.41	**G**	**I_3**	**H**	**I_2**
1–2	4.65–4.69 x 2.90	0.41	**E**	**I_3**	**F**	**I_3**
1–3	3.78–3.81 x 2.26	0.20	**F**	**I_3**	**G**	**I_2**
1–4[b]	4.41–4.49 x 2.93	0.38	H	I_2	H	I_2
1–5[b]	4.57–4.63 x 2.97	0.43	G	I_2	G	I_2
Method 2						
2–1	3.97–4.00 x 2.45	0.23	F	I_3	F	I_3
2–2	4.06–4.10 x 2.44	0.25	D	I_2	D	I_2
2–3	4.72–4.81 x 2.94	0.44	**M**	**I_3**	**N**	**I_2**
2–4	4.79–4.87 x 2.62	0.37	E	I_1	E	I_1

[a] All stones were graded independently at the GIA Gem Trade Laboratory (GIA GTL) for research purposes only. The GIA GTL does not offer this service for filled diamonds. Boldface type indicates where a grade had changed after filling.
[b] Not successful (according to manufacturer).

(in the course of testing, the temperature ranged from 24°C [76°F] when turned on to a maximum of 60°C [140°F]). As with steam cleaning, the diamonds to be tested were first mounted in four-prong solitaire heads on 14k gold rings. The rings were hung by their shanks from a wire hook so that they were submerged in the cleaning solution. The stones were examined first after a five-minute cleaning, then after 30 minutes of cumulative cleaning, and then at additional 30-minute intervals for a total of three hours of ultrasonic cleaning.

Thermal Exposure. In our previous study of Yehuda-treated diamonds (Koivula et al., 1989), we retipped the four prongs on a ring set with a filled diamond. This common repair procedure exposed the filled stone to the direct heat of a jeweler's torch. For the present study, we again performed a retipping experiment. A MECO Midget torch with no. 40 tip was used with natural gas and oxygen. Each diamond was mounted in a 14k gold ring with a four-prong solitaire head; care was taken to avoid placing any prong directly over filling entry points. For each ring, both the diamond and the setting were firecoated with a denatured alcohol/boric acid slurry and two prongs were retipped, using 14k white gold soft solder with a flow point of approximately 740°C. After the two prongs on each ring were retipped, we examined the stones visually and with magnification to note any damage to the fillers. We then had the remaining two prongs on each retipped, with 14k white gold hard solder with a flow point of approximately 800°C, and we reexamined the fillings for damage.

We also tested the upper stability limit of the filling materials themselves. The three samples were set in a refractory boat, which was placed in the hot spot of a Blue M model M10A-1A Lab Heat Furnace during heating steps. We monitored temperatures with a Chromel-Alumel thermocouple. For the test, the temperature was set approximately 50°C below the desired temperature, the diamonds were placed in the furnace, and the temperature was raised the final 50°C. This process took about 30 minutes, after which we removed the stones and allowed them to cool on a refractory block before visual examination. When we saw no appreciable damage to the filler, we repeated the procedure, increasing the target temperature for each subsequent test. Initial testing was at 118°C, with subsequent testing at 240°, 340°, 390°, 455°, 480°, 525°, and 600°C.

Not all jewelry repair procedures require the direct application of heat to mounted stones. Therefore, to test the effect of indirect heating, we had a filled diamond from each of the three treaters mounted in a four-prong head on a 14k gold solitaire ring and then had each ring resized larger by one size.

First, for the sample from each firm, experimental conditions reproduced a scenario in which the jeweler knows that the diamond is filled and therefore exercises special care to keep the stone cool. This was done by first firecoating the diamond and mounting with the alcohol–boric acid slurry. The diamond and prong setting were then wrapped with strips of wet paper and the ring was sized. (Alternative procedures for protecting sensitive stones during jewelry repair include coating with commercially available gel-like insulating substances such as "Heat Shield" and "Cool Jewel," or keeping the stone immersed in water during the heating phases.)

In the second scenario, the jeweler either does not know that the diamond is filled or does not know that special care may be required in sizing a ring set with a treated stone. In this test, therefore, the sizing was performed with a firecoating but with no special effort made to keep the diamond from being heated. Because we did not note any damage from the first resizing experiment, we reused the same mounted stones for this test.

Repolishing. Past reports (e.g., Koivula et al., 1989; Crowningshield, 1992) have documented how the heat generated during repolishing of a diamond can damage the filling material. To investigate this further, we submitted one filled diamond (with one or more filled fractures breaking the table surface) from each of the three firms to a Los Angeles diamond manufacturer for repolishing of the table facet.

Daylight Equivalency Testing. We used an Oriel model 81150, 300-watt solar simulator to produce daylight-equivalent illumination. A xenon light source combined with a series of lenses and filters creates an output emission that approximates the daylight spectrum at 1.7× its intensity (Oriel Corp., 1982). We allowed the lamp to warm up for 30 minutes, after which we exposed each unmounted diamond, placed face up on a white refractory tile, to the light source for the specified period of time. Periodically, we removed each sample from the simulator, examined it visually and microscopically for any damage to the filler, and then returned it to

the simulator for further exposure. Time intervals (cumulative) were 20 minutes and one, three, 10, 30, 60, 100, and 200 hours, corresponding to 34 minutes, 100 minutes, and five, 17, 51, 100, 170, and 340 hours of sunlight, respectively. We monitored the light intensity throughout the exposure period, using an Oriel model 81020 solar-simulator radiometer.

Exposure to Ultraviolet Radiation. We used the long-wave radiation source of a GIA GEM Instruments long-wave/short-wave unit, in conjunction with a GIA GEM ultraviolet viewing cabinet. With a median wavelength of 365.4 nm, long-wave UV radiation is not only a component of sunlight but is also the radiation source used in so-called "UV-A" tanning booths (the latter also presenting a scenario for exposure of a filled diamond to long-wave UV). After the lamp had warmed up for five minutes, we exposed each unmounted diamond to the long-wave UV source for the specified period of time, with the fracture facing the source of the radiation. Samples were periodically removed from the viewing cabinet, examined visually and microscopically for any damage to the filler, and then returned for further exposure. Time intervals (cumulative) were 20 minutes, and one, three, 10, 30, 60, 100, and 200 hours for three stones (three others were examined after a single 100-hour exposure). These exposures correspond to almost six, 17, 50, 170, 500, 1,000, 1,700, and 3,400 hours exposure to daylight.[2] We confirmed that short-wave ultraviolet light was effectively filtered out by observing a short-wave-fluorescing material (scheelite) after each exposure.

Low-Temperature Testing. We performed low-temperature testing first by simulating the type of cooling and warming that might occur when filled-diamond jewelry was worn in a cold climate, going from heated buildings to the outdoors and back. The test stones were placed in a cold corner of the freezer compartment of a standard frost-free refrigerator for 30-minute intervals. At the end of each cooling period—there were a total of 15 cooling sessions for each filled diamond—we removed the diamonds from the freezer and allowed them to warm to room temperature, at which point we examined them visually and with a microscope.

In the second low-temperature testing procedure, we exposed the same unmounted filled diamonds to the type of rapid cooling—to approximately –71°C (–96°F)—to which they would be exposed when sprayed with a refrigerant prior to examination with a desk-model spectroscope, a procedure available to many jeweler-gemologists. We placed each stone on the stage of a desk-model spectroscope and subjected it to the pressurized refrigerant chlorodifluoromethane (Chemtronics brand "Freez-It") for approximately five seconds. We then allowed the diamond to warm to room temperature and examined it with the unaided eye and the microscope. We repeated this routine five times for each diamond.

EFFECTIVENESS OF THE FILLING TREATMENTS

The effectiveness of the Yehuda filling process in improving the apparent clarity of diamonds has been well documented (see, e.g., Koivula et al., 1989; Koivula and Kammerling, 1990). In summary, the Yehuda process can drastically reduce the eye visibility of treatable features. However, because filled breaks may still be seen with magnification, the improvement in apparent clarity is only about one grade, and never more than two grades (table 1). In addition, some of the diamonds exhibited a drop in apparent color grade after treatment due to the inherent color of the filling material.

The Koss filling procedure is also very effective in improving the faceup appearance of diamonds (again, see figure 2). Despite this, only two of five stones submitted directly to Koss in 1992 by GIA researchers for "before-and-after" comparison showed sufficient improvement to warrant a higher apparent clarity grade (again, see table 1). However, examination of all five stones by X-ray fluorescence spectroscopy revealed lead (Pb) but not bromine (Br) in the filler, unlike the Koss-treated stones obtained by third parties for characterization of diagnostic features and durability testing. Consequently, we do not believe that any of the stones GIA submitted

[2]*The light intensity (in watts/cm^2) was estimated as follows: The source was a 4-watt mercury (Hg) arc lamp, with a special fluorescing screen to convert short-wave to long-wave UV radiation. Assuming 50% efficiency in the conversion from power in to Hg light out, 50% efficiency in the conversion from Hg light to long-wave UV radiation, and 33% efficiency due to geometric effects (i.e., one-third of the light is sent through the window and not absorbed on other surfaces within the lamp housing), then one-third watt is delivered through a 4.7 × 7.0 cm window, for an integrated flux of 10 mw/cm^2. Average sunlight has a total irradiance of about 0.6 mw/cm^2 at wavelengths below 365 nm (data integrated from Oriel Corp., 1982), so our long-wave UV source is about 17 times as powerful as sunlight in this spectral region.*

TABLE 1. Apparent color and clarity of sample round brilliant-cut diamonds before and after filling by Yehuda, Koss, or Goldman Oved.[a]

Sample no.	Measurements (mm)	Weight (ct)	Before		After	
			Color	Clarity	Color	Clarity
Yehuda[b]						
1	6.21–6.28 x 3.72	0.92	**L**	**Below I_3**	**M**	**I_3**
2	3.76–3.81 x 2.43	0.22	K	I_1	K	I_1
3	6.21–6.28 x 3.74	0.90	**K**	I_3	**L**	I_3
4	5.24–5.31 x 3.09	0.51	I	**SI_1**	I	**VS_2**
5	4.22–4.28 x 2.34	0.25	**E**	**I_1**	**F**	**SI_2**
6	4.06–4.18 x 2.39	0.25	**I**	I_1	**J**	I_1
Koss[c]						
1	4.05–4.12 x 2.60	0.28	J	**I_1**	J	**SI_2**
2	4.17–4.24 x 2.56	0.28	J	**I_1**	J	**SI_2**
3	4.13–4.18 x 2.61	0.27	F	I_2	F	I_2
4	3.95–4.00 x 2.57	0.26	**H**	I_1	**H/I**	I_1
5	4.09–4.14 x 2.37	0.24	M	I_1	M	I_1
Goldman Oved						
1	3.88–3.91 x 2.30	0.20	F	**I_3**	F	**I_1**
2	4.79–4.83 x 2.95	0.41	E	**I_3**	E	**I_2**
3	4.24–4.27 x 2.61	0.30	F	**I_3**	F	**I_1**
4	4.32–4.37 x 2.63	0.30	F	**I_2**	F	**I_1**
5	4.48–4.55 x 2.75	0.35	L	**I_2**	L	**I_1**
6	4.55–4.61 x 2.80	0.36	E	**I_2**	E	**I_1**

[a] All stones were graded independently at the GIA Gem Trade Laboratory (GIA GTL) for research purposes only. The GIA GTL does not offer this service for filled diamonds. Boldface type indicates where a grade had changed after filling.

[b] Original material manufactured by Yehuda; these results are from Koivula et al. (1989). In a later study of 34 Yehuda-filled diamonds (Koivula and Kammerling, 1990), four showed no change in color or clarity grade; 11 lost one grade in color without improving in clarity grade; and one lost two grades in color without improving in clarity grade. Eight stones that improved by one clarity grade dropped by one color grade; and one dropped by two color grades. Nine stones (believed to be more representative of the current Yehuda production) improved in apparent clarity by one grade (eight stones) or by two grades (one stone) without changing their apparent color grades.

[c] These stones were apparently not typical commercial products; see text.

directly to Koss for filling treatment are the typical Koss product.

Examination of the Goldman Oved–treated "before-and-after" samples demonstrated that this treatment is also very effective in reducing the eye-visibility of surface-reaching breaks. In all cases, the filling improved the apparent clarity by one or two grades (again, see figure 3). Furthermore, in no instance did we note a drop in the apparent color grade of the stones as a result of filling. The results of this phase of the investigation are also shown in table 1.

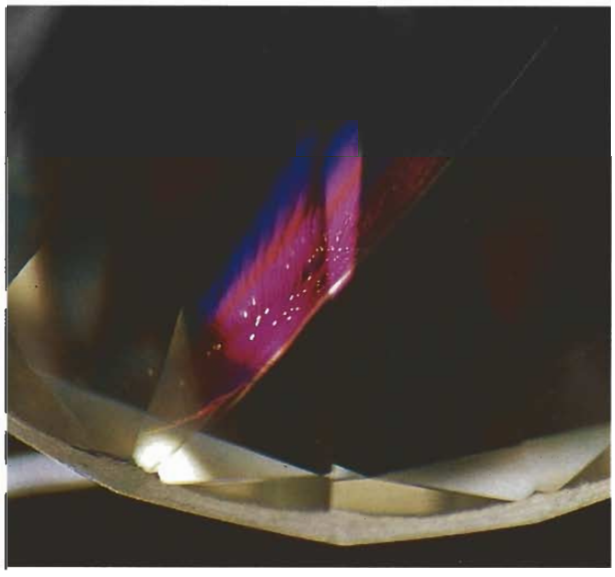
MICROSCOPIC FEATURES OF FRACTURE-FILLED DIAMONDS

In all stones examined during the current study, the fact that the diamond had been fracture filled could be determined by careful microscopic examination. The key features are described below. It is important to note that not all of the features described will be found in every stone. However, at least one of the features was detected in each stone tested.

Flash Effects. In the first Yehuda-treated stones we examined (Koivula et al., 1989), we noted a yellowish orange flash-effect color, in darkfield illumination, that changed to an "electric" blue when the stone was rocked so that the background became bright due to secondary reflection. During subsequent research, we observed a second pair of flash-effect colors in Yehuda-treated diamonds: a vivid pinkish purple seen in darkfield that became a bright yellowish green against a brightfield, secondary-reflection effect (Koivula and Kammerling, 1990).

In the most recent group of Yehuda stones examined, the colors noted in darkfield ranged from violet to purple to pink, with some filled breaks showing, for example, violet in one area and pink in another at a single angle of observation (figure 4). In brightfield, the flash-effect colors—again, often more than one from a single viewing angle—ranged from a vivid bluish green to greenish yellow (figure 5). In both brightfield and darkfield, we observed a flash-color change in all or part of the filled break when we rocked the stone back and forth very slightly. The color flashes in these more recent

Figure 4. Many of the recent Yehuda-treated diamonds display more than one vivid flash-effect color at a single viewing angle. The most predominant colors seen with darkfield illumination in recently treated stones are pink, violet, and purple. Photomicrograph by Shane F. McClure; magnified 28×.



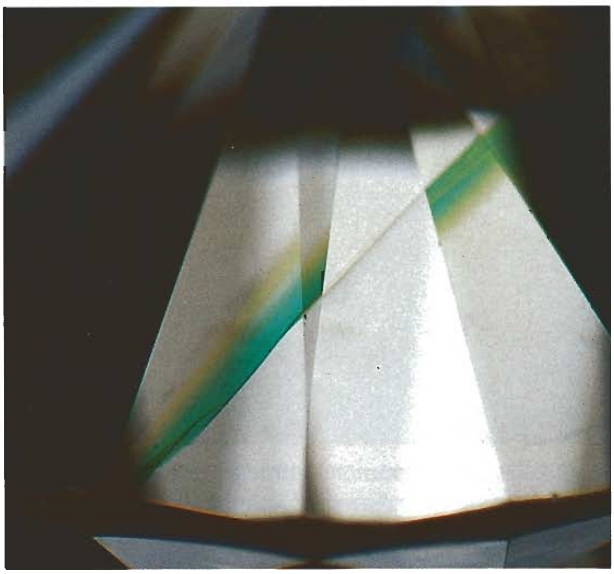


Figure 5. Brightfield flash-effect colors in the newest generation of Yehuda-treated diamonds range from bluish green to greenish yellow. Photomicrograph by Shane F. McClure; magnified 28×.

Yehuda-treated diamonds were equal to or greater in intensity than color flashes seen in the earliest Yehuda-treated stones. We also noted for the first time some flash effects associated with filled laser drill holes (figure 6).

Figure 6. For the first time, and only in diamonds treated recently by the Yehuda process, the authors saw flash effects along laser drill holes. Note the subtle orange and blue flash colors here. Photomicrograph by Shane F. McClure; magnified 40×.

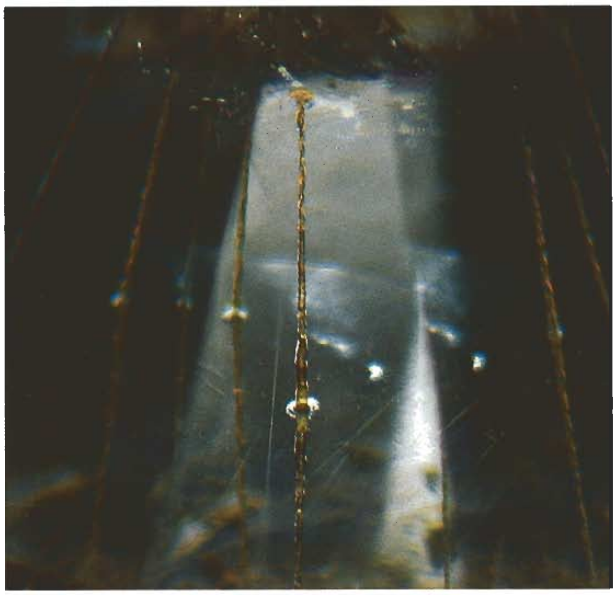


Figure 7. Koss-filled diamonds may also exhibit multicolored flash effects at a single viewing angle. Here (in darkfield illumination) we see pink, purple, and the less common desaturated slightly pinkish orange. The flash colors in the Koss stones we examined were usually less vivid than those seen in the Yehuda products. Photomicrograph by Shane F. McClure; magnified 40×.

We saw flash effects in all of the Koss-treated diamonds we examined. Most prevalent were a vivid pink and an equally saturated purple noted in darkfield illumination. As with the newer Yehuda-treated stones, at some viewing angles we could often see both pink and purple flashes in different parts of a single filled break; less frequently, we also noted a less saturated, slightly pinkish orange flash (figure 7). In a very few stones, we observed yellow and violet flash effects against dark backgrounds. In brightfield illumination, the most prevalent flash colors were bluish green and yellow (figure 8)—often at the same time in different portions of a filled break—although in some instances the color was a "pure" green. In general, the flash colors noted in Koss-treated stones were less intense than those seen in Yehuda-treated diamonds. However, in all cases—including stones as small as 0.02 ct—they could be detected using the standard gemological microscope's base-illumination (darkfield/brightfield) system.

Flash effects were also noted in all of the Goldman Oved–treated diamonds. Predominant colors in darkfield illumination were violet, purple, and pink. Less frequently, we saw a blue and (rarely) a green flash effect. When only one color was noted in darkfield, it was violet. Typically, however, we saw two or more colors at one time, the most common combination being violet and purple (figure 9).

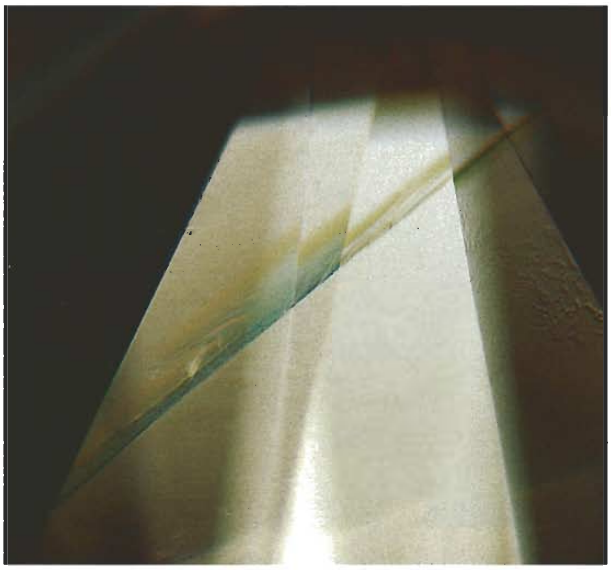


Figure 8. Flash colors noted in Koss-filled diamonds with brightfield illumination were typically bluish green and yellow—similar in hue to those seen in the Yehuda stones, but less saturated. Photomicrograph by Shane F. McClure; magnified 40×.

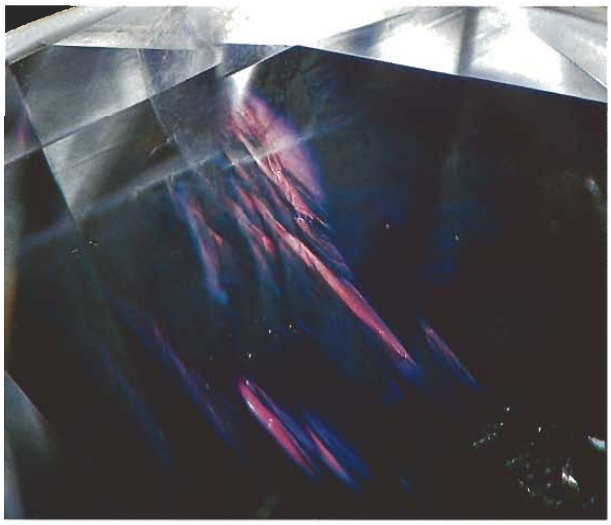
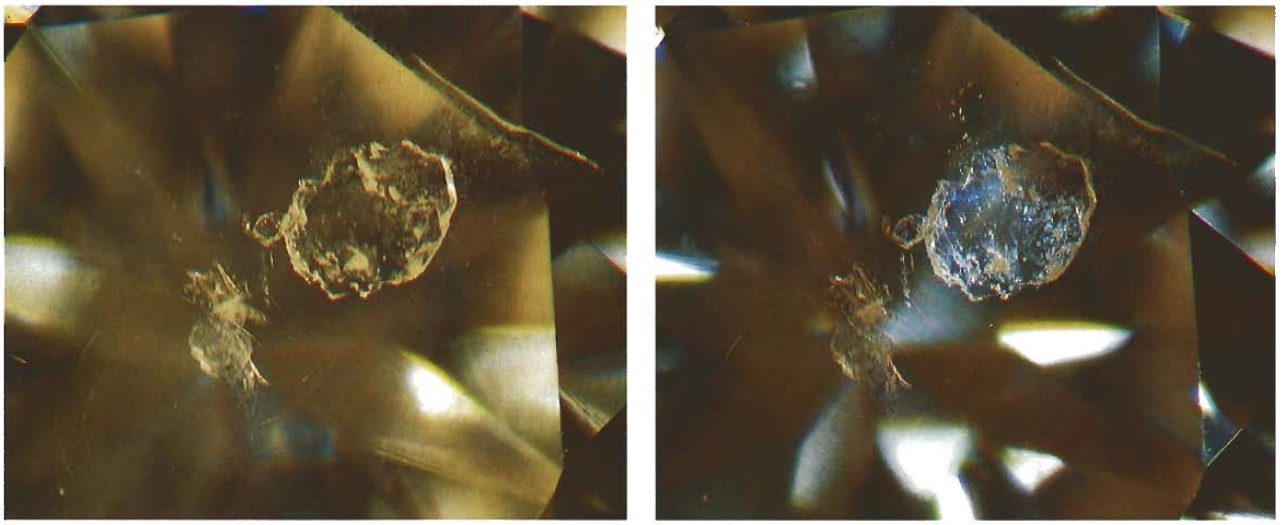


Figure 9. The most common darkfield flash colors seen in Goldman Oved–treated diamonds were violet, purple, and pink. Photomicrograph by Shane F. McClure; magnified 31×.

In brightfield illumination, we noted green, yellow, and, less frequently, bluish green. In some filled breaks, only one of the colors was seen; in others, more than one could be detected at a single angle of observation, similar in hue and saturation to those seen in the Koss-treated stones. In general, of the three products, the Goldman Oved–treated stones had the most subtle flash effects. Although standard darkfield/brightfield illumination was sufficient to see flash effects in all of the loose Goldman Oved–treated diamonds, we could detect this feature only with intense fiber-optic illumination in some of the mounted stones (figure 10).

In general, mounted stones may prove a great challenge to the gemologist trying to detect flash effects. Mountings limit both the directions in which the stone can be viewed and the amount of light that reaches internal features. Fiber-optic lighting, along with careful microscopic examination, may be essential in these instances (figure 11; see also, "Techniques to Identify Fracture Filling"

Figure 10. This mounted Goldman Oved–treated diamond showed no evidence of a flash effect in darkfield illumination (left). With oblique lighting from an intense fiber-optic light source, however, the flash effect becomes clearly visible (right). Photomicrographs by Shane F. McClure; magnified 40×.



below). For more on the optics of the flash colors, please refer to box B.

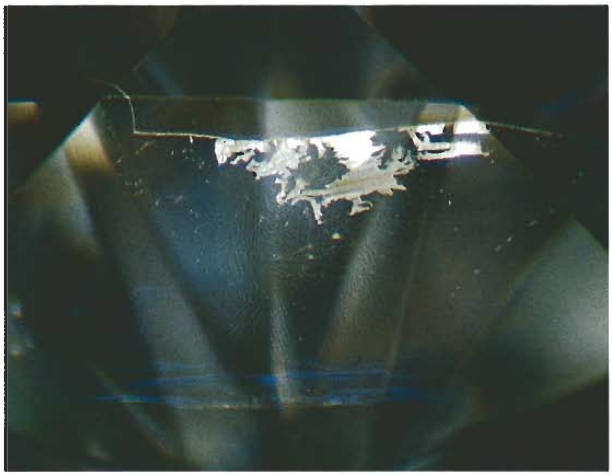
Flow Structure. A filled break may look as if a glassy substance has flowed into it, an appearance unlike anything seen in unfilled breaks. This feature was very subtle or absent in the most recent group of Yehuda-treated stones and in the Koss-treated diamonds. However, it was very subtle to fairly prominent in several of the Goldman Oved–treated diamonds (figure 12). Often, this feature can be detected only with intense fiber-optic illumination.

Trapped Bubbles. Although these voids in the filling substance (i.e., areas of incomplete filling) can be fairly large and noticeably flat, they are typically small and may occur in groups in an overall fingerprint-like pattern. Such trapped bubbles have been noted in all "generations" of Yehuda-treated stones examined by the authors.

In one stone in the most recent group of Yehuda-treated stones, we saw three-dimensional two-phase inclusions in an area of diamond intergrowth that was traversed by a filled fracture (figure 13). We believe these inclusions to be voids (such as negative crystals) that have been partially filled.

We saw at least some gas bubbles in all of the Koss-treated diamonds, although often these were extremely small and were detected only with high magnification and supplemental lighting (figure 14). Some filled breaks contained larger, flattened bubbles. In no instance did we see any of the complex,

Figure 12. A fine, transparent flow structure was detected in several of the Goldman Oved–filled diamonds examined for this study. Photomicrograph by Shane F. McClure; magnified 40×.



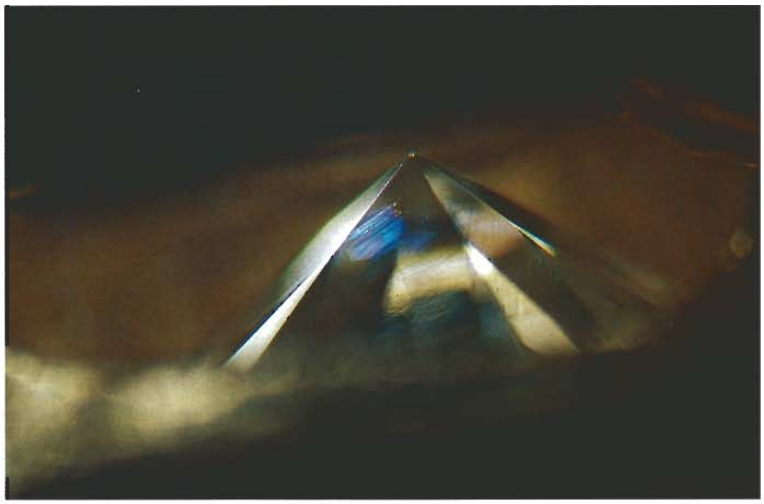
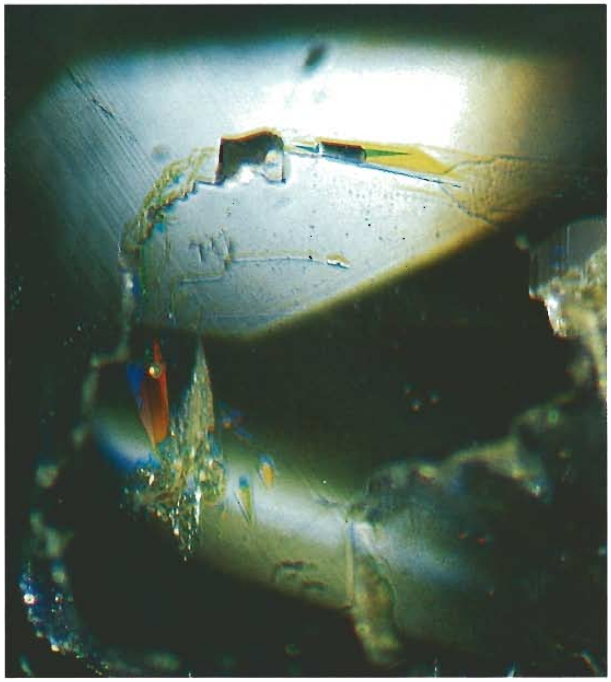


Figure 11. Only with a thorough examination under magnification, in conjunction with fiber-optic illumination, could we detect this flash effect in a filled fracture near the culet of a bezel-set Goldman Oved–treated diamond in the strip bracelet shown in figure 1. Photomicrograph by Shane F. McClure; magnified 40×.

Figure 13. These two-phase inclusions exhibiting multicolored flash effects were seen in one of the Yehuda-treated diamonds. Note the appearance of opposing flash colors—such as blue and orange—at the same viewing angle. This may be due to angled crystal faces inside the partially filled internal void. Photomicrograph by Shane F. McClure; magnified 40×.



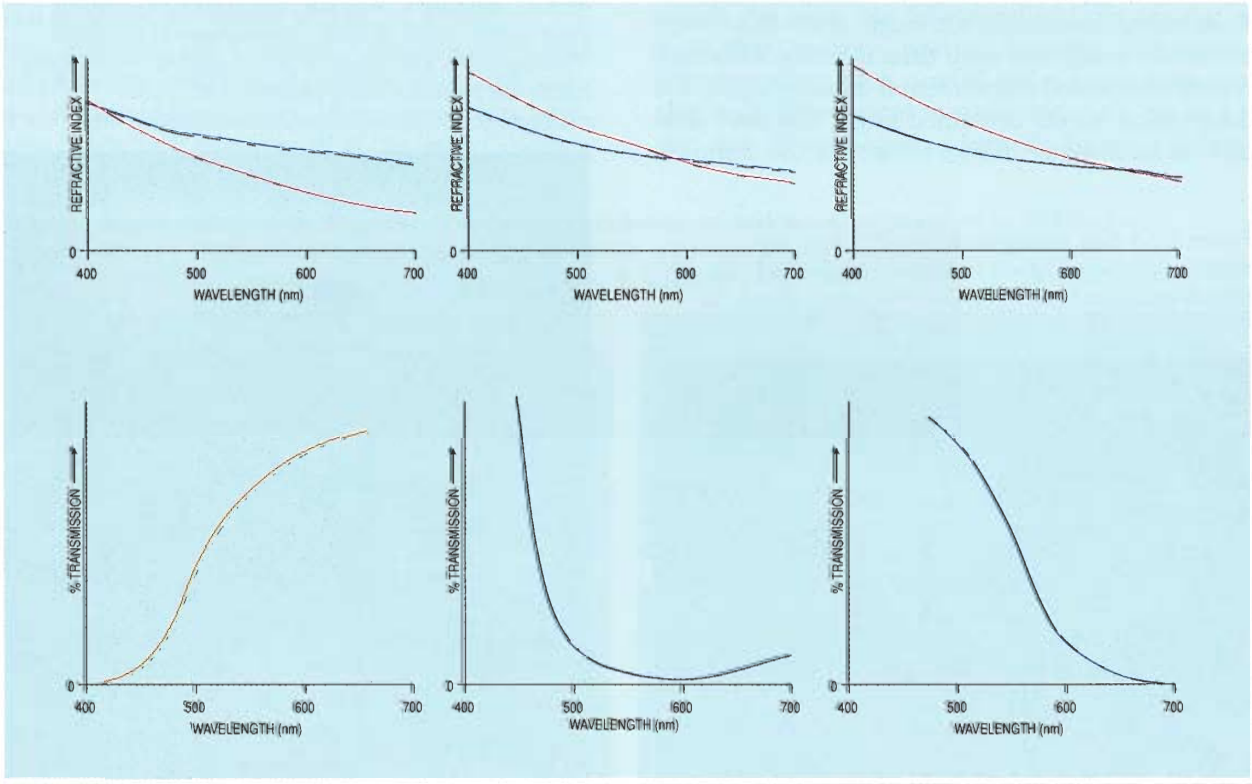
BOX B: The Optics of the Flash Effect

The first "flash effect" colors documented in Yehuda-filled diamonds were blue (when the background was bright) and orange (when the background was dark). Other flash effects were subsequently observed in products from this firm: yellow-green against a bright background and predominantly purple against a dark background (Koivula and Kammerling, 1990; Crowningshield, 1993). Other darkfield colors seen to date are: saturated pink, saturated purple, and less-saturated, slightly pinkish orange (Koss); and violet, purple, pink, "pure" blue, and green (Goldman Oved). Brightfield colors we have documented are: bluish green, yellow, and "pure" green (Koss); and green and yellow (Goldman Oved).

Initially, it was thought that the orange/blue flash colors were an interference effect, like the colors seen in natural iridescent fractures (Koivula et al., 1989); however, flash colors are more saturated and restricted in hue than interference colors, and the colors do not shift with polarization. Nelson (1993) stated that, because of these differences, the flash color cannot be due to an interference mechanism. He suggested that the flash colors are instead due to the difference in dispersion between diamond and the fracture-filling material. *Dispersion* occurs when refractive indices differ for different wavelengths of light (see examples in figure B-1). Dispersion is mathematically defined as the difference between R.I.'s for one substance (and one direction of light) between 486.1 nm (in the blue-violet) and 656.3 nm (in the red; Bloss, 1961). The value of dispersion for diamond, for example, is 0.044. A dispersion curve represents the variation of R.I. with wavelength.

In general, liquids and glasses have higher dispersions than do solids with the same R.I. Dispersion can be used to determine accurately the R.I. of a solid (the method of "colored Becke lines"); this technique was developed by Christiansen (1884, 1885) and can

Figure B-1. If a solid and liquid have the same R.I. at a wavelength in the violet region (left, top), only non-violet light is reflected (left, bottom), resulting in yellow darkfield flash colors; if they have the same R.I. in the red (right, top), non-red light is reflected (right, bottom), resulting in blue darkfield flash colors; if they have the same R.I. in the middle of the visible spectrum (center, top), darkfield flash colors are blue, or red, or purple, as other colors are not reflected (center, bottom). Figure adapted from Dodge, 1948, figure 2, p. 543.



be found in many textbooks on optical microscopy (see, e.g., Bloss, 1961). The darkfield color immersion method developed by Dodge (1948) is a variation on Christiansen's technique that provides another possible explanation for fracture-filling colors. Consider a colorless solid surrounded by a liquid with higher dispersion, but a similar refractive index (measured at the sodium D line, 589.3 nm). According to Dodge, differential refraction of light crossing liquid-solid interfaces produces spectral colors in brightfield illumination, and more intense "subtractive" colors in darkfield illumination. (That is, the spectrum of a darkfield color should equal the spectrum of the light source minus the spectrum of the brightfield flash.) Examples of pairs of dispersion curves and darkfield-color (subtractive) spectra are illustrated in figure B-1 for three cases: when the R.I.s for the stone ("solid") and filler ("liquid") match in the violet region of the spectrum (yellow darkfield flash color); in the yellow region (red, blue, or purple darkfield flash colors); and in the red region (blue darkfield flash color). Dodge comments that this mechanism also works when the dispersion of the liquid is less than that of the solid; that is, a fracture filling with a dispersion greater than that of diamond is not necessary.

The wavelength at which R.I.'s match for a solid and a liquid appears to be preferentially transmitted. Thus, Nelson (1993) found that the filling material for Yehuda-filled diamonds (with yellow-green brightfield/purple darkfield flash colors) has the same R.I. as diamond (2.421) at 560 nm (yellowish green). Also, according to Dodge, the *greater* the difference in dispersion between a liquid and a solid, the *brighter* the colors appear. Given that the Yehuda flash colors are the brightest and the Goldman Oved flash colors are in general the least obvious, the absolute difference between dispersions of filler and diamond is probably lowest for Goldman Oved, intermediate for Koss, and highest for (new) Yehuda.

Also, if darkfield colors are "subtractive" in nature, then as the color of the light source changes, the darkfield flash colors should change in the same direction. To test this, we handed a fracture-filled (Yehuda) diamond with eye-visible flash color to an experienced diamond color grader, who found that the flash color shifts from purple to violet (that is, from more red to more blue) as the illuminant color temperature changes from 3000 to 6500K (from light richer in radiation in the red region of the spectrum to light richer in blue and UV radiation).

In summary, according to this model, brightfield colors are restricted to spectral hues (i.e., red, orange, yellow. . . violet) and darkfield colors are the result of subtracting these hues from the color of the light source.

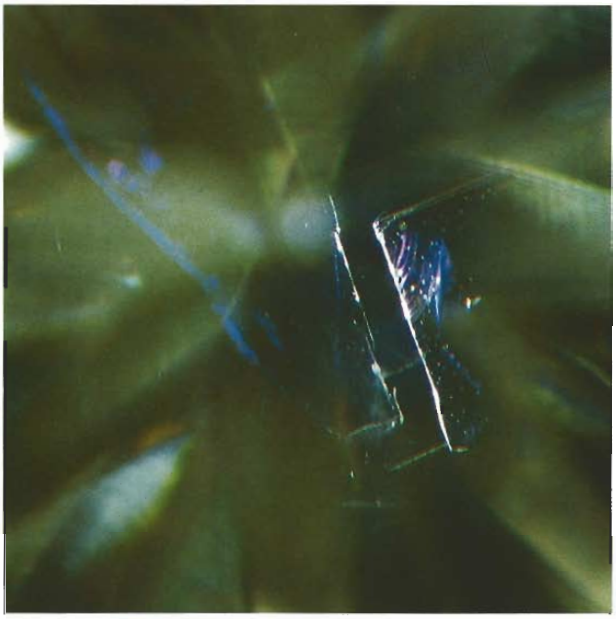
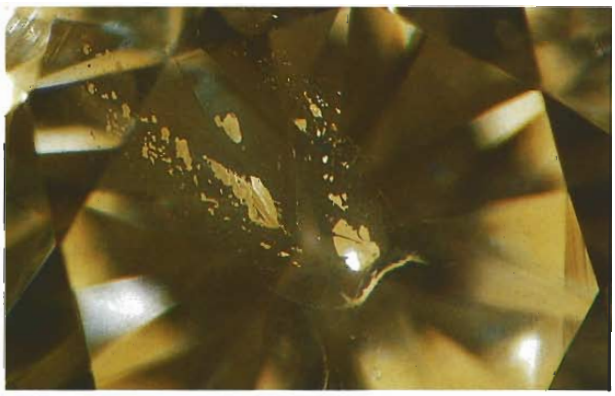


Figure 14. Many of the gas bubbles seen in Koss-treated diamonds are so small they look like pinpoints and can be detected only with fiber-optic illumination. Photomicrograph by Shane F. McClure; magnified 40×.

fingerprint-like patterns of bubbles noted in some of the earlier Yehuda-treated stones (Koivula et al., 1989).

We also saw bubbles in virtually all of the Goldman Oved–filled diamonds. In some instances, they were very few and extremely small, appearing essentially as bright, pinpoint inclusions. In other instances, they were relatively large and easily resolved as gas bubbles at fairly low magnification (figure 15).

Figure 15. Some of the Goldman Oved–treated diamonds displayed relatively large gas bubbles in their fillings. Photomicrograph by Shane F. McClure; magnified 33×.



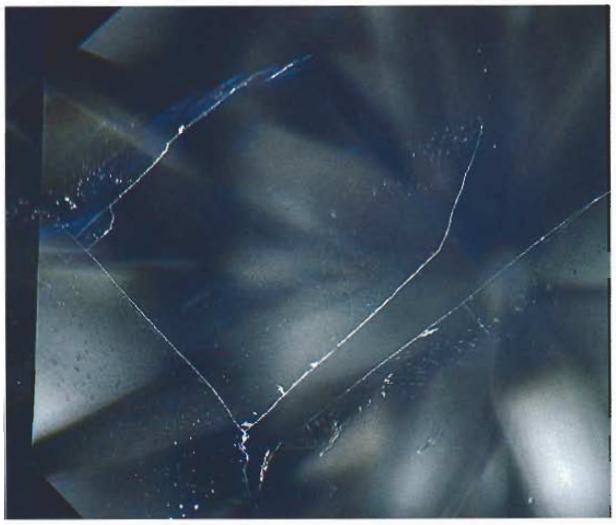
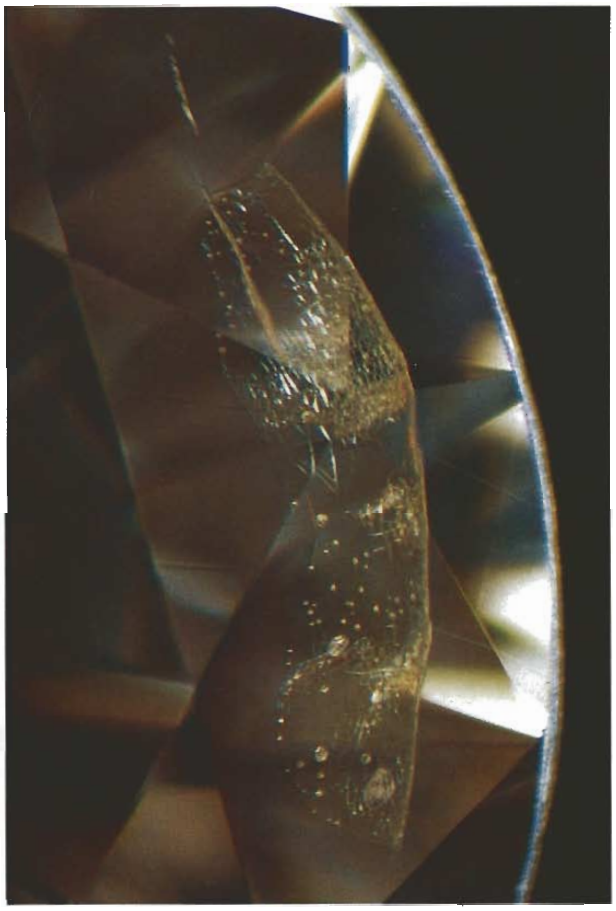


Figure 16. Areas of incomplete filling at the surface, which usually resembled fine white scratches, were present in most of the filled stones (here, by Goldman Oved) examined for this study. Photomicrograph by Shane F. McClure; magnified 38×.

Figure 17. A crackled texture was noted in some of the thicker areas of filling in Yehuda-treated diamonds. Photomicrograph by Shane F. McClure; magnified 40×.



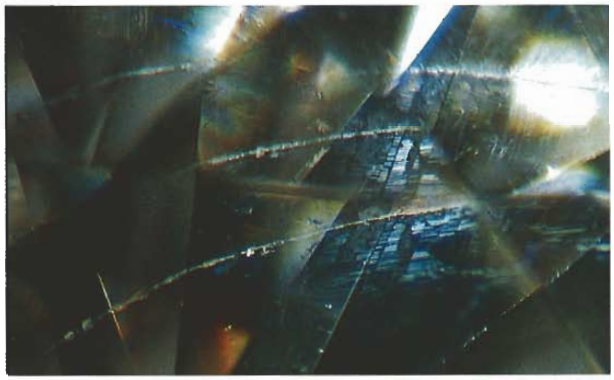


Figure 18. Extremely fine, nearly parallel whitish lines—possibly minute fractures within the filling material—were visible in some of the Koss-filled diamonds with fiber-optic illumination. Photomicrograph by Shane F. McClure; magnified 40×.

Incomplete Filling at Surface. In most of the Yehuda-filled breaks, we also noted extremely shallow areas of incomplete filling at the surface of the stone. In many instances, these had the appearance in darkfield illumination of fine white scratches. This feature may be the result of removal of a very small amount of the filler during a cleaning step performed by the manufacturer after treatment. Similar features were noted in virtually all of the Goldman Oved–treated diamonds we examined (figure 16). They were seen least frequently in the Koss stones, along the surface entry points of many of the filled fractures.

Crackled Texture. We detected cracks in the filling material, often with a web-like texture, in the thickest filled fractures of the more recent Yehuda-treated stones (figure 17), just as we had for previous Yehuda products. We also noted crackled areas in filler within laser drill holes.

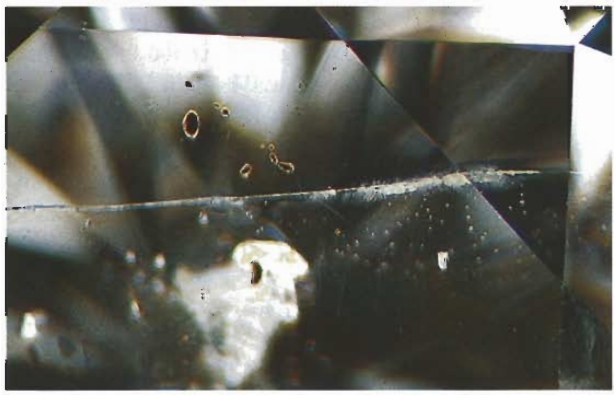
We did not detect crackled texturing in any of the Koss-filled stones. However, this feature has been seen only in relatively thick filled areas, and the Koss firm reports that diamonds with wide breaks are unsuitable for filling treatment (D. Shechter, pers. comm., 1994). What we did note in some Koss-filled breaks were extremely fine, nearly parallel whitish lines that may be minute fractures within the filler (figure 18). This feature, however, was very subtle and only seen with intense fiber-optic illumination. We did not detect a crackled texture in any of the Goldman Oved–filled fractures, although we did see some crackling in the substance partially filling a surface cavity.

Apparent Color of Filler. In relatively thick areas of the first Yehuda-treated diamonds we examined

Figure 19. Relatively thick areas of filling in Yehuda-treated diamonds showed a distinct yellow color. Photomicrograph by Shane F. McClure; magnified 40x.

(Koivula et al., 1989), the filler appeared light brown to brownish yellow or orangy yellow. Such color was not apparent in filled stones we examined later, and we speculated that the effect may have been reduced or eliminated by the manufacturer (Koivula and Kammerling, 1990). However, in the most recently examined Yehuda-treated stones, the yellow body color of the filler was quite noticeable in thicker filled fractures, cavities (figure 19), and laser drill holes.

Figure 20. Some Yehuda-treated diamonds exhibited whitish material near the site where the filler entered the breaks. This material may be filler residue. Photomicrograph by Shane F. McClure; magnified 40×.



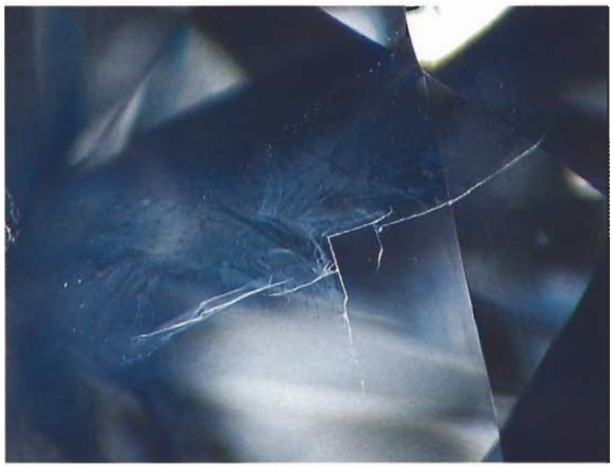


Figure 21. Cloudy areas within the filler were a common feature in Goldman Oved–treated diamonds. Photomicrograph by Shane F. McClure; magnified 40×.

We did not see any indication of inherent filler color in any of the Koss-treated diamonds (although there was a drop in apparent color grade of the diamonds described in box A) or the Goldman Oved–treated stones. For more on filler color, refer to box C.

Cloudy Surface Markings. A few of the Yehuda-treated stones examined in 1990 had cloudy, circular, surface markings that we had attributed to residue from the treatment process (see Koivula and Kammerling, 1990). In the most recent group of Yehuda-filled stones, we detected what appeared to be filling residue around the entry points of some filled breaks (figure 20). None of these surface indications of treatment were noted in any of the Koss- or Goldman Oved–treated diamonds.

Cloudy Filled Areas. In the most recent group of Yehuda-treated diamonds, we noted one feature not previously encountered in this firm's products: areas of reduced transparency ("white clouds") in a few of the filled breaks. Possible causes include groups of extremely small gas bubbles (the same mechanism that reduces the transparency of some amber), partial devitrification of the filling material, or a change in oxidation state of one or more of the filler's constituents.

Less commonly, we noted some cloudy areas in the filling material of Koss diamonds, typically near the surface entry points but also deeper in the breaks. Such "white clouds" were seen in at least one filled break in almost all of the Goldman

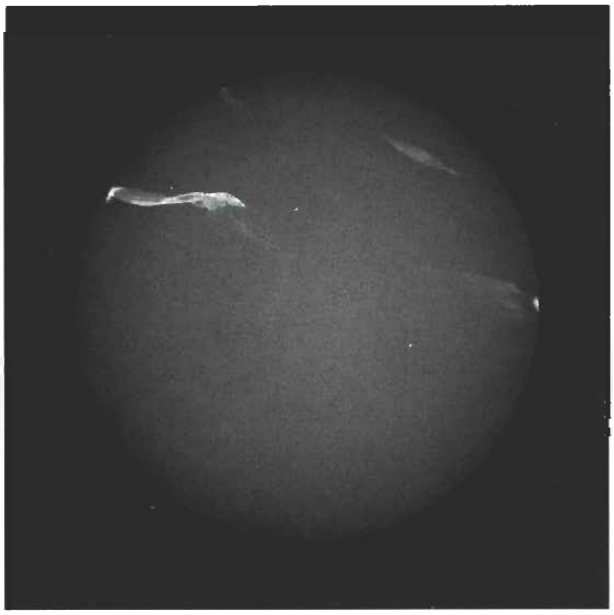


Figure 22. This X-radiograph of a fracture-filled diamond illustrates the extremes of filler visibility by this testing technique. Obvious white areas are evident on the perimeter of the stone, while the L-shaped filled break in the center is barely discernible. X-radiograph by Karin Hurwit.

Oved–treated diamonds we examined (figure 21). In many Goldman Oved–treated stones, we saw this feature before we detected a flash effect.

To summarize, with magnification and various lighting conditions, we detected flash effects in all of the sample treated diamonds. Other features associated with the presence of a filler in breaks include flow structures, trapped bubbles, a crackled texture, cloudy areas, and a white or cloudy appearance at the surface of the fracture.

X-RADIOGRAPHY

Although this technique is not directly available to most gemologists, X-radiography is particularly useful when the microscopic features in a filled diamond are somewhat ambiguous. It also helps document the extent of treatment where the filling material has been damaged, as in jewelry repair procedures involving heat (see, e.g., Hargett, 1992). In addition, it could prove to be a useful test in screening parcels of diamonds.

In most of the early Yehuda-treated diamonds, the filling material was more opaque to X-rays than was the host diamond and thus appeared as white areas on the X-radiograph (Koivula et al., 1989). Two of the three Yehuda stones tested for the current study yielded clear evidence of filling—again, in the form of X-ray-opaque white areas—on the exposed X-ray film. "White" filling areas—some faint and others quite sharp—were seen on the X-rays of all three Koss-filled diamonds. One of the Goldman Oved–treated diamonds showed clear evidence of filling (figure 22), while indications in the other two stones were present but very faint.

Several years of experience with this method have shown us that the orientation of the filled breaks relative to the X-ray film can significantly affect the outcome. Filled areas are most noticeable when the plane of the break is essentially perpendicular to the film, which results in the greatest absorption of the X-ray beam by the filling material. Also, breaks oriented essentially parallel to the film, thin breaks, or those that otherwise have relatively little filler, may not appear on the X-radiograph. The limited sensitivity of the X-ray film is another factor.

Note that there does not appear to be any correlation between the results of X-radiography and the strength of flash effects seen: A filled break that does not produce a distinct white area on the X-ray film may display a pronounced flash effect under magnification, while a thick area of filling that is clearly visible on the X-radiograph may display only a weak flash effect or none at all.

Note, too, that artificial fillings are not the only substances found in diamonds that are opaque to X-rays. Some rarely encountered mineral inclusions, such as iron sulfides, also may appear white on an X-radiograph. Furthermore, iron sulfides may occur as thin-film inclusions that could closely resemble filled fractures on an X-radiograph. Therefore, the results of X-radiography can only be interpreted in conjunction with other, especially microscopic, evidence.

CHEMICAL ANALYSIS

Some introductory comments are necessary concerning the use of EDXRF spectroscopy in the chemical analysis of diamonds. Natural, untreated, near-colorless diamonds often contain small amounts of impurities such as iron, potassium, calcium, zinc, and copper (Field, 1979), which are not discussed below. The following results focus exclusively on heavy elements, which typically are not found in untreated diamonds. More details on the chemistry and other properties of diamond filling materials can be found in box C.

The Yehuda stones treated recently contain

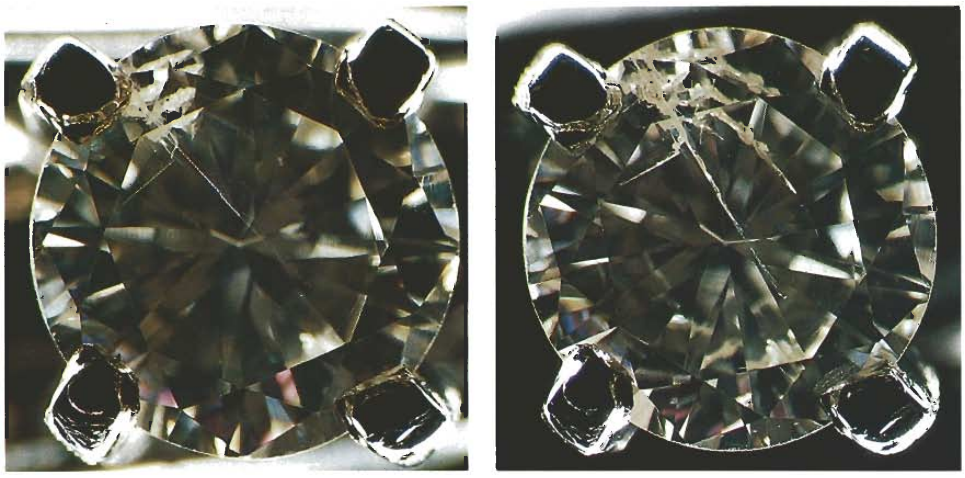


Figure 23. This 0.29-ct Koss-treated diamond had noticeable damage to the filler near the surface after steam cleaning for twenty minutes (before, left; after, right). Photomicrographs by Shane F. McClure.

both lead (Pb) and bismuth (Bi), which is consistent with the chemistry of the Yehuda diamonds treated earlier (Koivula et al., 1989). The early Koss-treated stones revealed Pb as their only heavy element, although the commercially available Koss diamonds revealed both Pb and bromine (Br). We found both Pb and Br in the Goldman Oved–treated diamonds as well. (Note that the chemistry of the Goldman Oved–treated stones was similar to that of a filled diamond from the Chromagem firm that was also analyzed by the authors.)

DURABILITY AND STABILITY OF THE FILLINGS

Over the past five years, concerns have repeatedly been voiced in the trade as to the durability and stability of filled diamonds. Treated diamonds with fillings that have been damaged by heat have been submitted to the GIA Gem Trade Laboratory for examination and damage reports (see, e.g., Hargett, 1992; Crowningshield, 1992). We have also learned that, while ultrasonic cleaning for a brief period may not damage at least some fillings, extended exposure to this process (and, by inference, numerous cleanings of short duration) may cause some shattering of, or other damage to, the filler (Crowningshield, 1992; Wakefield, 1993). Perhaps of greatest potential concern, independent research by Wakefield (1993) has shown that extended exposure to a short-wave ultraviolet lamp (and, by extension, to prolonged daylight exposure) can cause degradation of some fillers. This, in turn, has provoked a highly charged exchange (Koss, 1993, 1994a-d; Wakefield, 1994a, c-e; Yehuda, 1993, 1994a,b; Even-Zohar, 1994b).

For this study, we performed a series of durability and stability tests on diamonds filled by the Yehuda, Koss, and Goldman Oved firms. However, with the single exception noted below, only one stone from the current production of each diamond treater was used for each test. Therefore, we feel that the results of our tests are *representative,* but not *conclusive.* In other words, we do not guarantee that stones from any diamond treater will be either impervious or susceptible to a given circumstance.

Steam Cleaning. Twenty cumulative minutes of steam cleaning produced no noticeable change in the filler or the faceup appearance of the Yehuda-treated stone. The Koss-filled diamond showed some minor removal of filling material near the surface entry points after five minutes of steam cleaning. The damage continued throughout the testing procedure, with filling removed from areas deeper into the stone. Although relatively little material was removed, the damage did cause areas of the fractures at and just below the surface to become quite noticeable; at 10× magnification, they resembled deep scratches (figure 23).

The Goldman Oved–treated diamond, like many of the filled stones from this firm, had some areas of incomplete filling at the surface. After five minutes of steam exposure, there appeared to be an almost imperceptible change in the width of an unfilled area. After 10 minutes, however, there was a definite widening, with further widening noted at 15 minutes. After 20 minutes of steam exposure, this unfilled area was significantly wider than it was at the beginning of the test, with the damage having spread across the entire length of the fracture (figure 24).

It appears, then, that steam cleaning for even relatively brief cumulative time periods presents a potential durability problem.

Ultrasonic Cleaning. No change in the filler was noted in the Yehuda-treated diamond at five or 30 minutes. After 60 minutes total ultrasonic clean-

BOX C: More on the Fracture-Filling Process

The substances used for filling diamond breaks are high-refractive-index glasses related to those commercially used for glass adhesives ("solder glasses"; Nassau 1994), far-infrared optic fibers (1994 Koss promotional brochure), and other applications (Beck and Taylor, 1958). Such glasses contain large amounts of heavy elements. Solder glasses are commercially available through large glass-manufacturing companies such as Corning, Schott, and Pilkington. Glasses transmitting far in the infrared are generally not very stable physically and are produced only in small quantities (Dumbaugh, 1984). To be used as filling materials, the glasses must have an R.I. close to that of diamond in the visible range (i.e., approximately 2.4), be fairly liquid at relatively low temperatures (so that they can be easily introduced into the fractures), must not crystallize (devitrify) easily, and must be relatively free of coloration. Several of these considerations are discussed below, as they relate to diamond treatment.

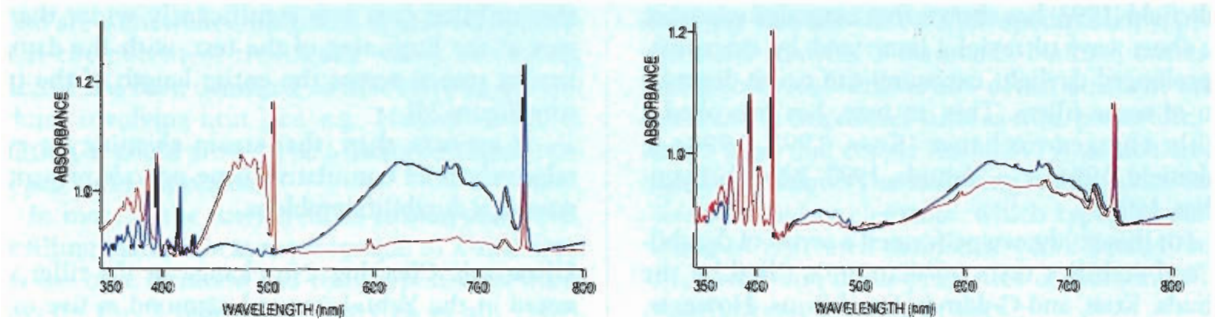
Chemical Composition. Solder glasses generally contain lead (Pb) and boron (B), sometimes with other elements. Infrared-transmitting glasses are oxides containing Pb and bismuth (Bi), as well as some additives to give the glass stability (Dumbaugh, 1984). Heavy elements (Pb or Bi) were found in the earlier Yehuda filling material, as well as possibly chlorine (Cl) and B (Koivula et al., 1989). Since then, further study has indicated that Cl is present only on or near the surface of the Yehuda fracture-filling material, but not in the bulk filling material itself. More recent commercial fracture-filling processes appear to be using similar types of materials, although with some variations in chemistry.

The following is a summary of the chemical elements found to date in the diamond fracture-filling materials currently available: for Yehuda—Pb, Bi, B, O; for Koss—Pb, Br, probably contains Cl or O, may contain B; for Goldman Oved—Pb, Br, probably contains Cl or O, may contain B. EDXRF analysis demonstrated that there is less bromine in the Koss glass than in the Goldman Oved filling material. Since these two glasses have similar optical properties, there must be another element to compensate for the relative lack of Br in the Koss glass. If it was a light element, especially at low concentration, it would not be detected by EDXRF. Candidates include oxygen or chlorine, or perhaps both. Boron is another likely glass-forming element that cannot be detected by EDXRF (Nassau, 1994).

High Refractive Index. Although we have been unable to measure the refractive index and dispersion of diamond fillings, the presence of flash-effect colors proves that, somewhere in the visible range, the R.I. of the filling glass matches that of diamond (2.435 at 486.1 nm, 2.410 at 656.3 nm; Field, 1979, p. 650). Pb and Bi in oxide glasses, as in those used for filling diamonds, are known to yield an R.I. that is considerably higher than those of normal commercial oxide glasses. Heavy-metal oxide glasses containing, among other things, PbO and Bi_2O_3 are reported to show R.I.'s in the range of 2.2 to 2.6 (Dumbaugh, 1986).

"Melting" Point and Temperature of Treatment. A glass has no melting point; it is already a liquid—albeit a very slow-flowing one—at room temperature. As it is heated, it reaches a temperature (the "softening temperature": K. Nassau, pers. comm., 1994) at which the glass begins to flow at a significant rate. The softening temperature places a lower limit on the temperature at which the fracture-filling process can be performed. Using a heating stage on a microscope, and observing the behavior of the filling in a diamond, one can estimate this temperature for fracture-filling glasses. Koss fillings show an approximate flow temperature of 370°C, and we first observed damage in the fill-

Figure C-1. Temperatures of treatment were estimated based on this comparison of the low-temperature ultraviolet-visible absorption spectra of two laboratory-irradiated green diamonds before (blue) and after (red) fracture filling: (left) a 0.84-ct round brilliant, and (right) a 0.76-ct round brilliant.



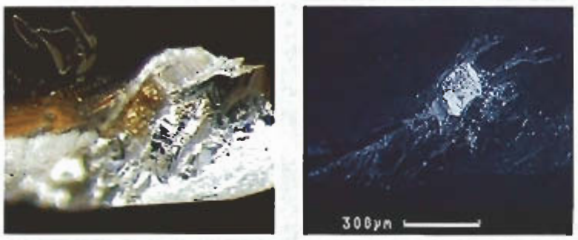


Figure C-2. A yellow mass is seen in the photo on the left, at the entrance of a filled fracture in a Yehuda-treated diamond (photomicrograph by John I. Koivula; magnified 20×). On the right, the same yellow mass is seen in a scanning electron micrograph; it has the same contrast and chemical composition as the filling material (the bar indicates the scale).

ing materials in a Koss-treated diamond in the temperature interval between 340° and 390°C (see the "Durability and Stability of the Fillings" section).

An indirect way to obtain information regarding treatment temperature is to observe changes in laboratory-irradiated green diamonds. Such diamonds are, in effect, annealed during the fracture-filling process; the behavior of various temperature-sensitive absorption features (such as GR1 at 741 nm) provides information on the temperature regime the treated diamond has undergone (see, e.g., Collins et al., 1986). Two such stones were submitted to this experiment, one filled by Koss (a 0.84-ct round brilliant), the other by Goldman Oved (a 0.76-ct round brilliant). The absorption spectra of these two diamonds before and after treatment are compared in figure C-1. On the basis of the decrease in intensity of the GR1 absorption, the intensities of the H3 (503 nm) and 595-nm features, and the presence or absence of the H1b absorption (4935 cm^{-1} in the near-infrared), the following approximate temperatures of treatment can be estimated: 600°C for the Koss process, and 500° to 550°C for the Goldman Oved process.

Coloration of the Filling Material. Because the apparent color grade of some fracture-filled diamonds drops after filling, at least some of the filling materials may themselves be colored. For instance, observations with the microscope indicated a yellow-to-brown color in the filling material in early Yehuda-treated diamonds (Koivula et al., 1989). A yellow mass found at the outcrop of a filled fracture in a Yehuda-treated diamond is shown in figure C-2, left. Scanning electron microscopy (figure C-2, right) and energy-dispersive X-ray spectrometry (SEM-EDS) analysis established that the chemistry of the yellow material was identical to the filling material. Therefore, the Yehuda filler is strongly colored. In general, oxide glasses that are suitable for fracture filling (i.e., with a high R.I. and a low softening temperature) contain large amounts of Pb or Bi, and are known to be colored yellow (Dumbaugh, 1978, 1986). The optical spectra of two representative glasses (a commercial solder glass obtained from Schott, and a lead borate glass made by Pilkington) showed UV-cutoff edges shifted toward the visible, which cause yellow color.

Optical absorption spectroscopy demonstrates that colored glass present in open fractures, even in small amounts, can decrease the apparent color grade of a diamond. Because the amount of glass present in a filled diamond is very small, we expect to see only subtle changes in the absorption spectrum of a filled diamond compared to its spectrum before filling. We were able to measure these changes in a Yehuda-treated diamond cut into a slice that contained a large filled fracture almost parallel to the sides of the slice. Absorption spectra were recorded for both the filled and unfilled regions (figure C-3). A second experiment was carried out by creating a diamond–solder glass (10 µm)–diamond "sandwich," with similar results. The filling material shifts the UV-cutoff by over 25 nm toward the longer wavelengths. The edge of this spectral feature extends into the visible range—up to approximately 450 nm—and therefore induces a weak absorption in the violet. This creates a pale yellow coloration and explains why some diamonds may have a slightly lower apparent color grade (i.e., are slightly more yellow) after fracture filling.

Infrared Absorption. Although solder glasses themselves show broad, weak absorptions in the mid-infrared range, at 500, 700, 900, and 1220 (strongest) cm^{-1}, in general there is too little filler present to be detected in nitrogen-bearing (i.e., most) diamonds, which also have absorptions in the mid-infrared. As we found previously (Koivula et al., 1989), infrared spectroscopy is not a useful technique for routine testing of fracture-filled diamonds.

Figure C-3. Ultraviolet-visible absorption spectra are shown for (a) a filled region and (b) an unfilled region of a laser-sawn and polished slice from a Yehuda-filled diamond. The absorption shifts (right) toward the visible, causing the yellow color (see text).

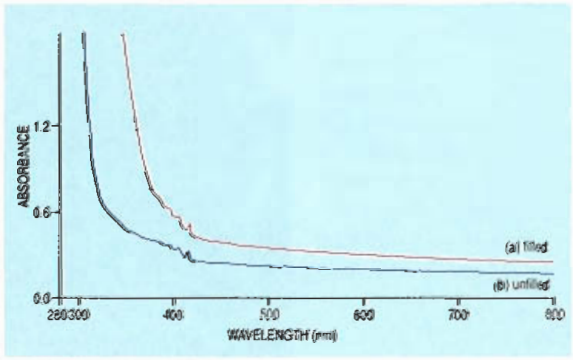


Figure 24. Obvious damage to the filler was also seen in this 0.36-ct Goldman Oved–filled diamond after a prolonged steam cleaning (before, left; after, right). Photomicrographs by Shane F. McClure.

ing, however, we noticed damage to the filler at the surface of one filled break—minute, but enough to make the fracture visible as a white line. After 90 minutes, the damage was significantly more apparent: At the entry points of all surface-reaching fractures, the filler was easily seen as thin, bright lines resembling scratches (figure 25). These damaged areas appeared about the same after two hours and 2 1/2 hours, with perhaps slightly more damage evident after three hours. (Note that although we did not observe any changes in the Yehuda-filled stones tested with ultrasonic cleaning in our 1989 study, we ran that test for only 30 minutes.)

Neither the Koss- nor the Goldman Oved–treated diamonds showed any removal of, or damage to, the filler after three hours of cumulative exposure to ultrasonic cleaning, although the filling was removed (essentially, all at once) from a wide cavity on the surface of the Goldman Oved–treated stone.

Direct Heating. Another durability concern with filled diamonds is how such treated stones will react when exposed to the high temperatures employed in various jewelry repair procedures. The retipping tests caused major damage to the filled fractures in all three stones. The Yehuda-treated diamond showed a significant loss of filling material, with most of the remaining filler turning cloudy and containing many gas bubbles. The Koss- and Goldman Oved–treated (figure 26) diamonds lost even more filling material, although the remaining filler did not drop as much in transparency as it did in the Yehuda-treated stone.

In all three stones, the second phase of retipping—of the other two prongs on each ring—caused additional damage to the remaining filler.

In a separate test, furnace heating produced degradation in the fillings of all three sample stones at 390°C. In the Yehuda-treated stone, which was extensively fractured, the filling material became cloudy at that temperature, and the flash colors shifted and became less prominent (figure 27). No other change was noted until 600°C, at which point

Figure 25. Extended ultrasonic cleaning caused a loss of filler in this 0.34-ct Yehuda-filled diamond (before, left; after, right). This damage is seen at the surface entry points of the fractures as bright lines resembling scratches. Photomicrographs by Shane F. McClure.

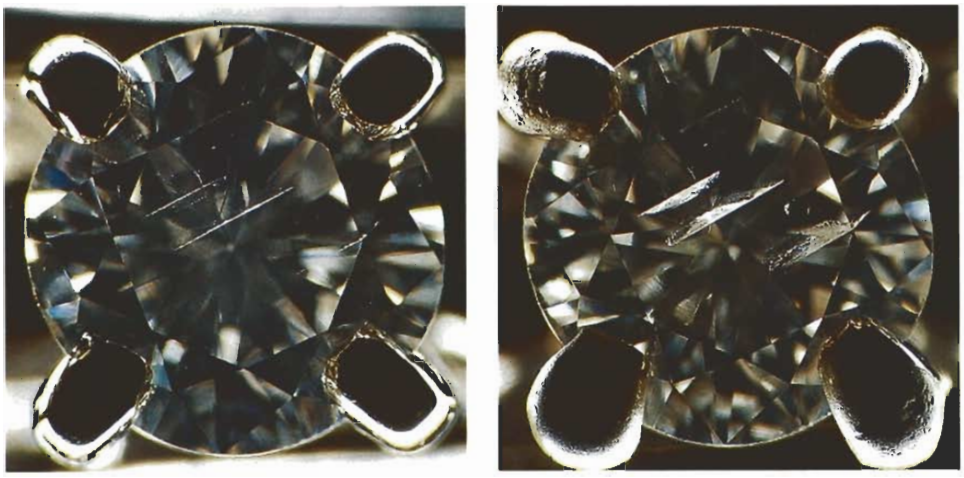
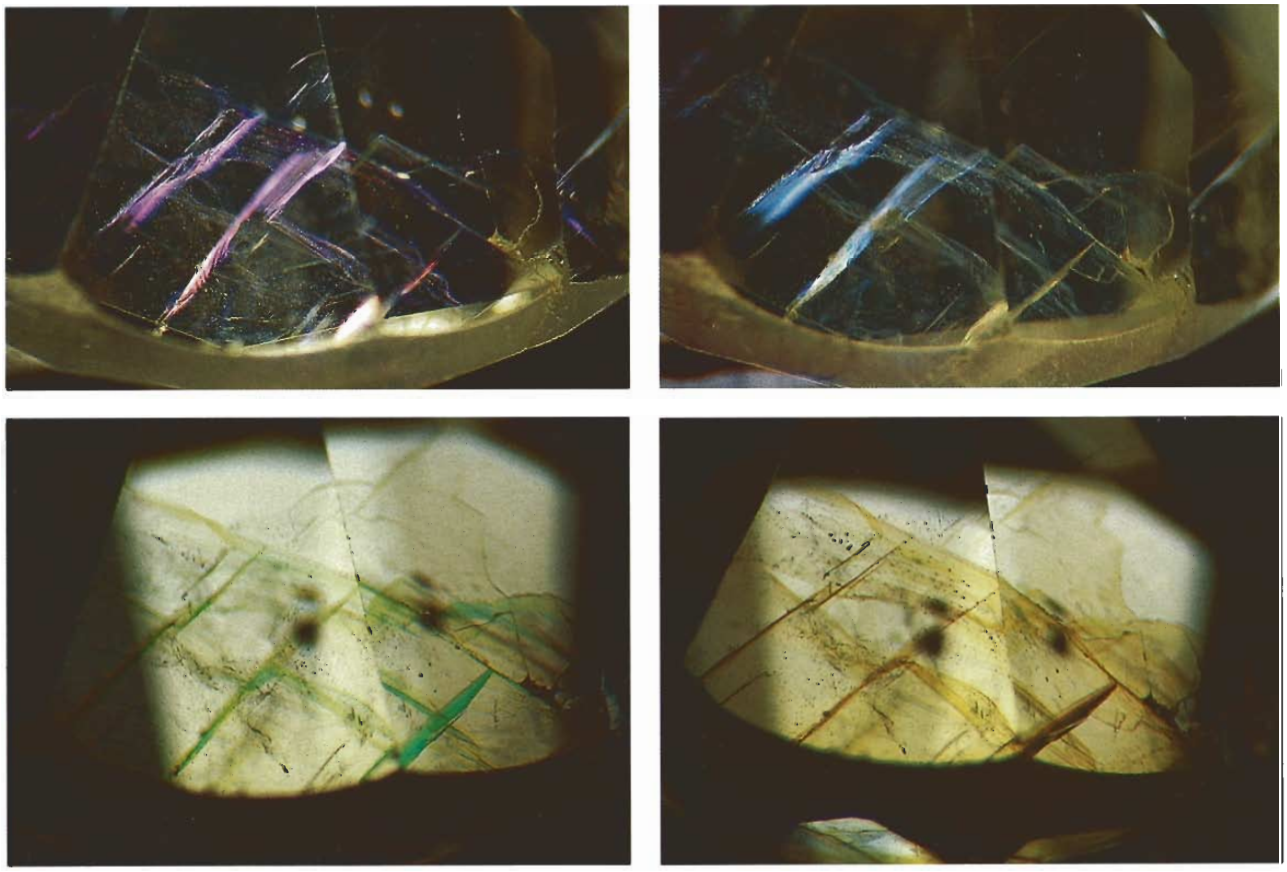


Figure 26. The vast majority of the filling material was removed from this mounted 0.19-ct Goldman Oved–treated diamond during the retipping of the prongs. Note the significant difference in appearance before (left) and after (right) the retipping procedure. Photomicrographs by Shane F. McClure.

we saw the first evidence of filler loss from the outermost edges of the fractures. In the Koss stone, we saw some loss of filler from the edges of the fractures after heating to 390°C (figure 28), with about half the filler gone after 45 minutes at 420° to 455°C. The Goldman Oved stone showed migration of the filler within the break into an apparently cellular structure at 340°C, loss of filler from the edges of fractures at 390°C (figure 29), and significant loss of filler at 455°C.

Figure 27. After this 0.36-ct Yehuda-treated diamond was heated to 390°C, the filler began to degrade and a distinct change in the flash colors was observed. In darkfield illumination before heating (top left), a pink flash is evident; after heating to 390°C (top right), the filler is cloudier and the flash color has changed to blue. The same diamond in brightfield illumination before heating (bottom left) shows a green flash color; after heating to 390°C (bottom right), the filler appears darker and the flash color is yellow. Photomicrographs by Shane F. McClure; magnified 30×.



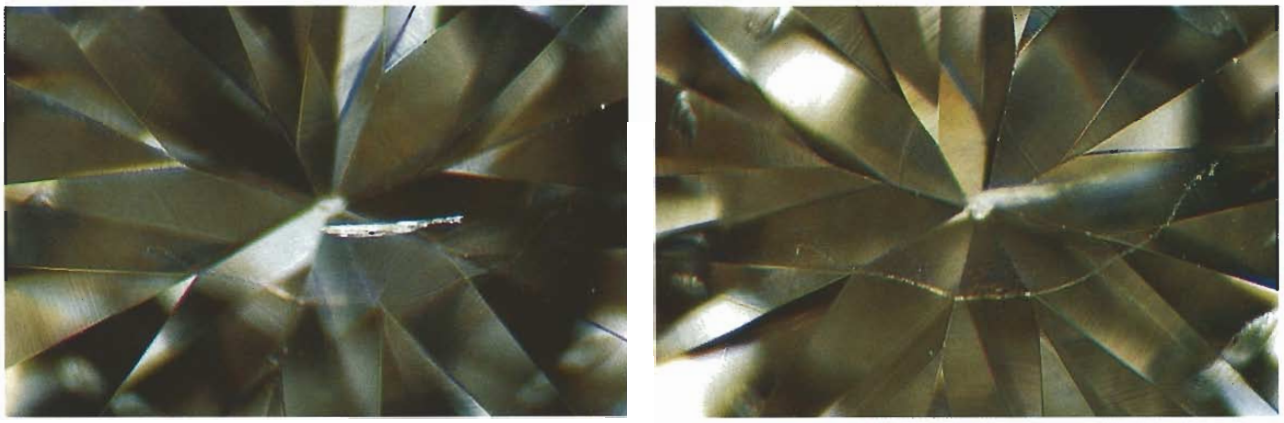


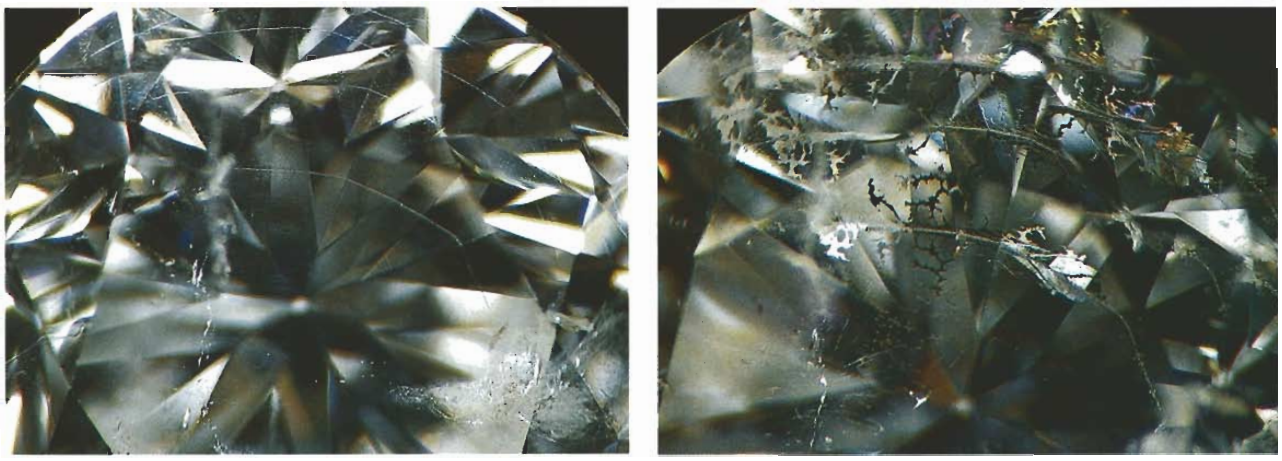
Figure 28. After heating to 390°C, this Koss-treated diamond showed a minor loss of filling material at the surface, apparent as a white line resembling a scratch (before heating, left; after, right). Photomicrographs by Shane F. McClure; magnified 33×.

Indirect Heating. Careful resizing (as when the jeweler is aware of the potential problems associated with fracture-filled diamonds) caused no visible damage to any of the three stones tested. Nor was any damage seen when resizing was performed with no special effort made to keep the diamonds from being heated. Note, however, that the results may differ depending on such factors as the karatage of the gold, thickness of the ring shank, and the length of time the filled areas are exposed to heat.

Repolishing. Repolishing produced different degrees of damage to the filler in all three test stones. Although the Yehuda-treated diamond lost only a small amount of filler from the treated breaks within a few tenths of a millimeter from the surface, almost all of the remaining filler became cloudy and more small gas bubbles had formed (figure 30). Substantial amounts of the filling material were removed from the Koss-treated diamond, resulting in large unfilled areas near the surface and some minor clouding but many gas bubbles (producing a fingerprint-like pattern, as in figure 31) appearing throughout the remaining filler. The Goldman Oved–filled diamond showed damage similar to that of the Koss-treated stone.

Laser Inscribing. Because of industry concerns about detection and disclosure, it has been suggested that all such stones be laser-inscribed with initials that disclose the treatment. One retailer (Blando, 1994) proposed "CL-E" (we assume for "clarity enhanced"); Daniel Koss has reportedly

Figure 29. A Goldman Oved–filled diamond showed extensive loss of filler after heating to 390°C. This damage was easily seen with a 10× loupe (before heating, left; after, right). Photomicrographs by Shane F. McClure; magnified 27×.



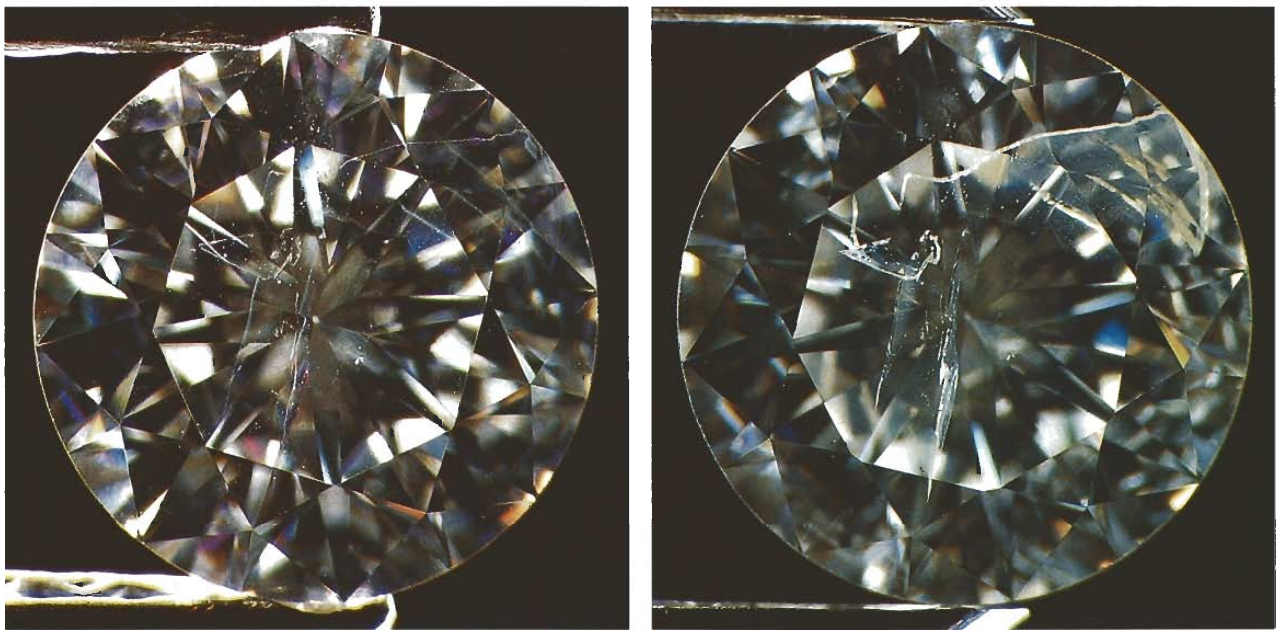


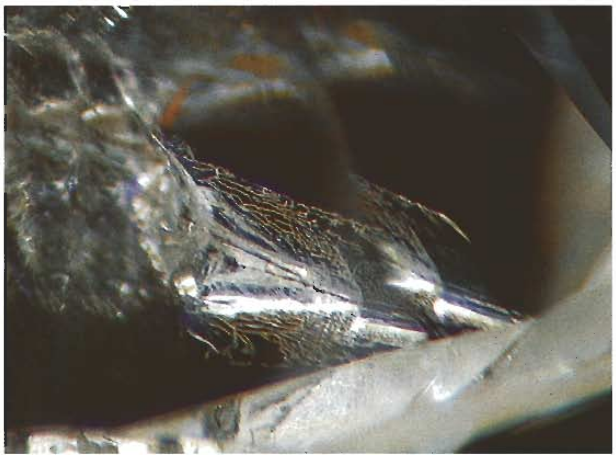
Figure 30. Repolishing the table facet of this 0.37-ct Yehuda-treated diamond caused almost all of the filling material to become cloudy, the formation of new gas bubbles, and removal of some of the material near the surface (before repolishing, left; after, right). Photomicrographs by Shane F. McClure.

pledged to laser inscribe "F.F." (fracture filled?) on the girdles of his stones (Federman, 1994). If such markings were supported broadly in the trade, a standard symbol (like a hallmark) might be adopted. Because the laser-inscription process generates high temperatures, however, this could pose its own durability problem.

Therefore, the authors laser-inscribed the GIA logo and identifying letter(s) on the girdle of one filled diamond from each of the three manufacturers. In each test, care was taken to inscribe an area of the girdle that was close to, but not intersected by, an entry point. Since many filled diamonds have more than one fracture that intersects the girdle, we chose a position approximately 45° from where the nearest fracture crossed the girdle. The diamonds were examined and photographed before and after laser inscription. We did not detect (with magnification) any deterioration in the filling in any of the stones.

As with the other processes described here, however, the outcome may differ depending on the amount of the filling material or its proximity to the inscription.

Figure 31. Repolishing this 0.45-ct Koss-filled diamond removed substantial amounts of the filler, producing the fingerprint-like pattern seen here in the remaining filling material. Photomicrograph by Shane F. McClure; magnified 40×.



Daylight Equivalency Testing. To simulate extended exposure to sunlight, the authors subjected filled diamonds to radiation in a solar simulator. No obvious changes were noted in the Yehuda-, Koss-, or Goldman Oved–treated diamonds after 200 hours (equivalent to only 340 hours of exposure to sunlight; see next section for results relating to a longer daylight-equivalent exposure).

Exposure to Ultraviolet Radiation. As noted above, Wakefield (1993) has reported that exposure to a short-wave ultraviolet lamp—which she equated to a longer daylight equivalency—can cause degradation of the filling material in Koss-treated dia-

Figure 32. Noticeable clouding of the filler in this large break in a 0.29-ct Koss-treated diamond was seen after 100 hours of exposure to long-wave ultraviolet radiation (before exposure, left; after, right). Photomicrograph by Shane F. McClure; magnified 37×.

monds. Some have questioned both the relevance of using a short-wave UV unit for the testing (something to which she has responded—see Wakefield, 1994e) and her correlation of the exposure to actual lighting conditions that might be encountered by filled stones (Koss, 1994d; Even-Zohar, 1994b). Wakefield subsequently (1994e) suggested that the damage to the filler from a short-wave UV unit she had documented was caused by the low levels of long-and medium-wavelength UV radiation, because most diamonds do not transmit short-wave UV.

Our testing with long-wave UV radiation showed no obvious changes to one Yehuda-treated diamond after 100 hours—and a second Yehuda-treated diamond after 200 hours—of exposure. However, we first noted minor discoloration of the filling material in one Koss-filled diamond after 60 hours (equivalent to 1,000 hours of daylight), with discoloration becoming visible table up after 100 hours, and further discoloration noted at 200 hours. A second Koss-treated stone showed discoloration and clouding of the filling (figure 32) after a single 100-hour exposure. One Goldman Oved–filled stone showed no apparent change after 100 hours' exposure, but obvious discoloration and clouding after 200 hours (figure 33). A second Goldman Oved–treated diamond showed no damage after 100 hours' exposure.

The presence of Br in the Koss and Goldman Oved fillers may explain their discoloration and clouding when exposed to long-wave UV radiation. Many compounds containing halogens such as Br and iodine are known to decompose spontaneously when exposed to strong sunlight (see, e.g., Turro 1978, pp. 568–569); an example well known to gemologists is the degradation of methylene iodide.

Low-Temperature Testing. Neither of the low-temperature tests had a noticeable effect on any of the three samples.

GIA GEM TRADE LABORATORY POLICY ON GRADING FILLED DIAMONDS

As stated by Koivula et al. (1989), the GIA Gem Trade Laboratory has an established policy of not issuing grading reports on diamonds determined to have been filled. This policy was adopted on the basis of two considerations: (1) the true—that is, untreated—color and clarity grades of such treated stones cannot be determined after filling (unless the filling material is completely removed); and (2) the treatment—like diamond coatings but unlike laser drilling—is not permanent. The implication of this second item is that, because of durability and stability concerns, even the *apparent* color and clarity grades of treated stones may change.

With respect to color, initial research showed that at least some filling treatments may lower the apparent grade due to the inherent body color of the filling material (Koivula et al., 1989). More recent research (Wakefield, 1993), and the results of this study, have indicated that some fillers may darken after exposure to ultraviolet radiation like that contained in sunlight.

With respect to apparent clarity, it has been shown that the filling may be damaged by extended ultrasonic cleaning (Crowningshield, 1992; this study). As Wakefield (1993) pointed out, the cumulative effect of repeated ultrasonic cleanings could have a similar effect. Acid boiling (Rapaport, 1987), repolishing (Crowningshield, 1992; this study), and jewelry repair procedures involving heat above 400°C (Koivula et al., 1989; Hargett, 1992; this study) may also adversely affect the filling material. All such damage makes the filled break more visi-

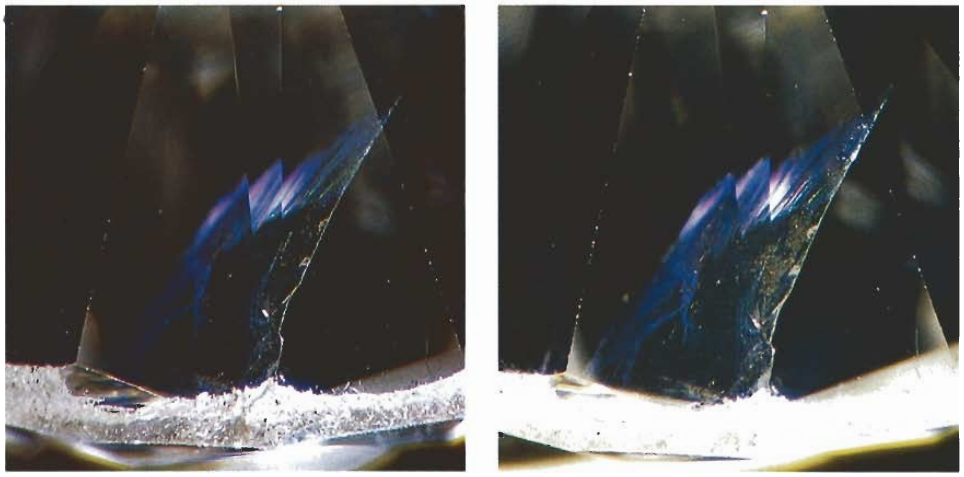


Figure 33. Obvious discoloration and clouding were seen in this filled break in a Goldman Oved–treated diamond after 200 hours' exposure to long-wave UV radiation (before exposure, left; after, right). Photomicrographs by Shane F. McClure; magnified 27×.

ble and, in general, lowers the apparent clarity grade.

An additional grading consideration relates to diamonds that are so heavily included that they fall outside the GIA clarity grading scale. Essentially, such stones are below I_3 in clarity and for GIA GTL purposes are classified as "rejection grade." Diamonds in this category include those that have so many inclusions or inclusions that are so large that the stone is no longer completely transparent. The GIA Gem Trade Laboratory does not issue grading reports on such low-clarity diamonds. Yet diamonds that fall in this "rejection grade" might be good candidates for fracture filling, and it would be inconsistent to grade filled stones that—in their unfilled state—would not be graded.

Therefore, the GIA Gem Trade Laboratory continues this policy of not grading fracture-filled diamonds. Any filled diamond that is submitted for quality analysis is issued an identification report stating the identity of the stone (i.e., diamond) and noting that a clarity-enhancing foreign material has been artificially introduced into surface-reaching features, which precludes quality analysis. As discussed earlier, this position has now gained formal support among diamond dealers.

TECHNIQUES TO IDENTIFY FRACTURE FILLING

At the time of our initial study, magnification with darkfield illumination was sufficient to detect fracture filling (Koivula et al., 1989). This lighting method is the standard for diamond clarity grading as well as for locating and interpreting inclusions in gem identification. More recently, however, we have encountered filled diamonds for which darkfield illumination is insufficient to detect conclusively the diagnostic features of this treatment. Following is a review of supplemental methods that have proved useful, as well as some precautions to keep in mind when examining diamonds for evidence of fracture filling.

Microscopic Techniques. *Fiber-Optic Illumination.* Through 1991, we saw distinctive flash effects in most fracture-filled diamonds we examined (Koivula et al., 1989; Koivula and Kammerling, 1990; DelRe, 1991). Filled breaks that lacked the flash effect included some with relatively thick fillings and some that were very small (see the example of bearded girdles in Koivula et al., 1989). Since 1991, GIA GTL gemologists have examined several fracture-filled diamonds in which the flash-effect colors were much less intense. We have found that, for such stones, the intense light from a pinpoint fiber-optic illuminator can make the flash effects significantly more noticeable, as well as reveal the extent of the filled breaks and any hairline fractures in the filling material (Kammerling and McClure, 1993).

Flash effects are often first noticed as reflections in facets around the stone (figure 34), rather than directly from the break itself. These can be extremely helpful with mounted stones, where viewing angles are restricted.

In some instances, flash effects can be detected in filled diamonds with intense fiber-optic illumination *without* magnification. To perform this test, place the stone table down on the end of a vertically positioned fiber-optic light wand and then rotate the stone: This can reveal flash effects from filled breaks in the pavilion that are parallel or nearly parallel to the girdle plane. However, even if a flash is seen using this test, the presence of filled breaks should be confirmed with examination under magnification.

Figure 34. In this 0.32-ct Yehuda-treated diamond, all of the flash effects visible are reflections of a single large filled break. The flash of the filled break itself is not seen at this viewing angle. Photo by Shane F. McClure.

It is important to emphasize that failure to see a flash effect does not prove that the stone is untreated. Care must also be taken to make sure that dispersion or interference colors (figure 35) in unfilled fractures are not mistaken for a flash effect. (See also "Thin-Film Iridescence" under "Precautions," below.)

Partially Polarized Light. With a single polarizing filter placed between the microscope's objective and the diamond, light transmitted through a stone may sometimes reveal the outline of the filled areas (Kammerling and McClure, 1993).

Shadowing Technique. In this lighting technique, an opaque, black, nonreflecting light shield is inserted gradually into the transmitted light path between the gemstone and the light source (see Koivula, 1982). It has proved useful to the authors in detecting flow structures within the filling material of some treated stones. Generally, partial closing of the microscope's iris diaphragm is sufficient.

Application of Water. To perform this test, hold the diamond in question in a stoneholder on the microscope's stage with the fracture entry point facing up. While examining the stone, run a small brush (like that sometimes used by gemologists when clarity grading diamonds) that has been dipped in water across the entry point. If the water enters and fills the break—noted as a temporary lowering of the fracture's relief—then it can be concluded that the break is probably not filled (figure 36). Note, however, that failure of the water to enter the break

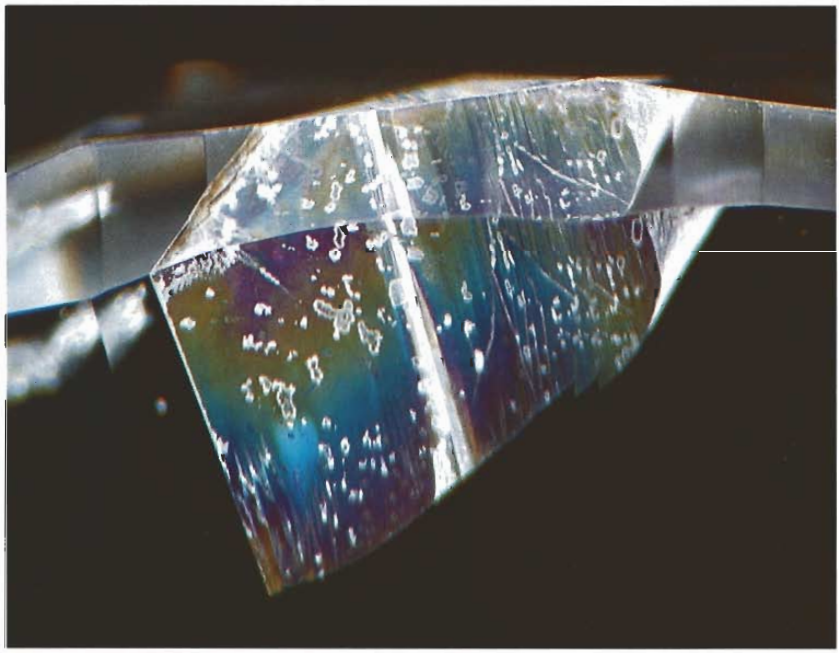


Figure 35. This unfilled fracture displays natural iridescent colors in a broad range of hues. (Note also the small, white areas within the fracture, which could be confused with the gas bubbles often seen in a filled break.) Photomicrograph by Shane F. McClure; magnified 33×.

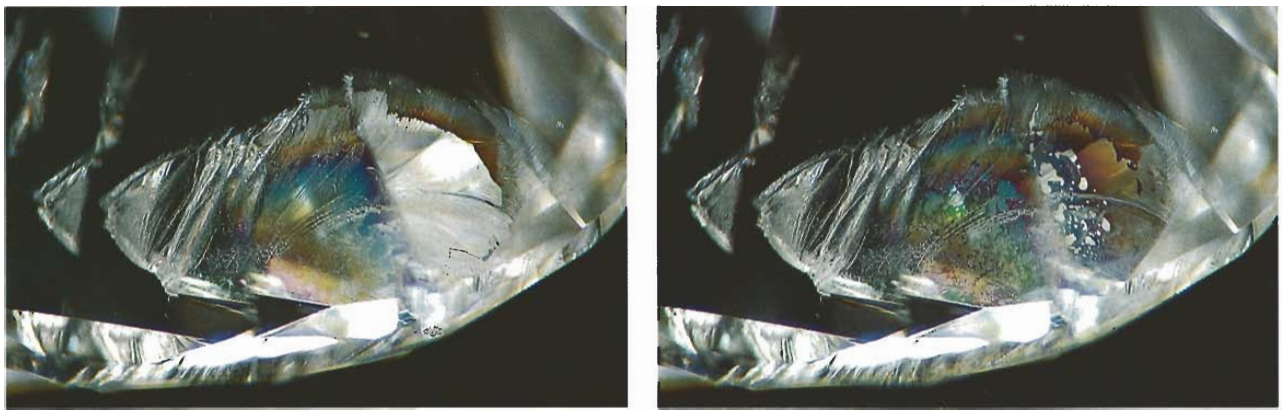


Figure 36. One method for determining whether a suspect fracture is filled is to see if water can be drawn into it. On the left is an unfilled fracture with natural iridescence. On the right is the same fracture after water was applied to its entry point at the diamond's surface; note the decrease in relief of the fracture, as well as the bubbles apparent in the water that was drawn into the break. With the microscope, one can actually see the bubbles move. Photomicrographs by Shane F. McClure; magnified 33×.

Figure 37. The interference spectrum can be seen by looking at a quartz wedge between crossed polarizers. The dark-gray color occurs where the interference film is the thinnest; colors vary to yellow, blue, red, yellow again, etc., and finally to repeated pinks and greens, as the film gets thicker. Compare this with the iridescent fracture in figure 35. Photomicrograph by John I. Koivula.

does not prove that the break *is* filled, as some unfilled breaks with very narrow surface openings will not accept the water.

Precautions. *Thin-film Iridescence.* As noted in Koivula et al. (1989), unfilled fractures can act as thin films, displaying rainbow-like interference colors that might be mistaken for flash effects (again, see figure 35). Like the flash effects in filled breaks, these iridescent effects can vary in the intensity of their colors; unlike flash effects, they should always show the same color sequence (figure 37; see also Fritsch and Rossman, 1988). The iridescence shown by unfilled breaks typically has a broad range of hues, although on occasion they may only display a few (figure 38). Flash effects often display a single color at most viewing angles, but some filled breaks can be positioned to show more than one color at a time.

One reliable feature that can be used to make the distinction is the *viewing angle.* Iridescent colors in unfilled breaks are usually seen best at a viewing angle roughly perpendicular to the plane of the break (again, see figures 35 and 36, and Koivula, 1980), whereas flash effects in filled breaks are usually detected when looking almost parallel (edge-on) to the break (figure 39). Another important distinction is the *texture* of the break: Unfilled breaks typically have a "feathery" appearance (figure 40) that we have not seen to date in filled breaks. Unfilled breaks also have much higher relief, the primary purpose of the filling being to lower the relief.

Another technique that may be helpful in separating iridescence from flash effects is illumination with polarized light. When a polarizer is placed

Figure 38. Occasionally, unfilled breaks exhibit iridescence in only one or two hues. Photomicrograph by Shane F. McClure; magnified 33×.

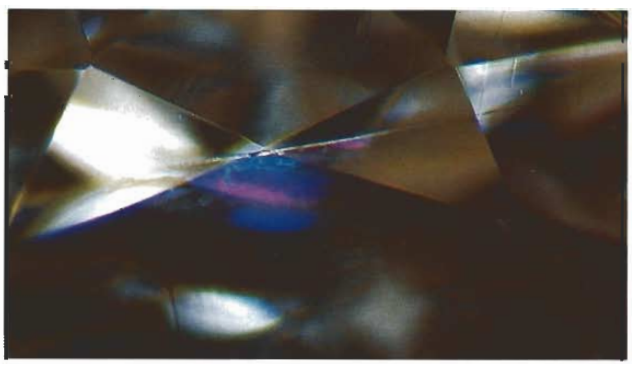
between the diamond and the observer, iridescent fracture colors will shift in position as the polarizer is rotated; flash colors, however, only turn darker and brighter, and they do not shift laterally with rotation of the polarizer.

Natural Colored Staining. Occasionally we come across surface-reaching breaks in untreated diamonds that contain an orangy brown staining of naturally occurring iron compounds (figure 41) which could be mistaken for an orange flash effect. The staining, however, should be visible throughout a broad range of viewing angles, whereas the similarly hued flash effect would typically be seen only within a very narrow range of viewing angles. In addition, a relatively thick staining may appear translucent to nearly opaque, whereas an orange flash effect would have no comparable reduction in apparent transparency. Also, a stained break should have higher relief than a filled break.

"Indirect" Surface-Reaching Breaks. It would seem obvious that a break must reach the surface of a diamond for it to be filled, and in most of the filled stones we examined, the fractures had direct surface-entry points. It is possible, however, for a break that is entirely internal to be filled by first laser drilling one or more narrow channels to it from the surface (figure 42; see also, e.g., Crowningshield, 1993). A thorough microscopic examination for possible fracture filling should take this possibility into consideration. A laser drill hole that appears to "go nowhere"—that is, that does not end at a void caused by a vaporized inclusion—should be considered especially suspect.

Body-Color Masking. The body color of a diamond can affect the ease with which flash effects are seen. In our experience, flash effects are relatively distinct when seen against the essentially colorless to very pale yellow body color of most diamonds; they may be even more obvious when the hue is complementary to the stone's body color, for example, a blue flash in a fancy yellow diamond. However, when the body color of a diamond and the flash

Figure 39. When the filled feather in this diamond is viewed perpendicular to its length (left), it is only visible at its entry point—as a white line on the surface of the crown—where it is not completely filled. Only after the stone is tilted so that the break is viewed nearly parallel to its length, can the flash colors be seen (right). Photomicrographs by Shane F. McClure; magnified 40×.



effect are of the same or a similar hue, a "masking effect" may result. Thus far, we have noticed this masking effect primarily with orange flash effects that are obscured in stones with deep yellow to brown body colors (although, as noted, the complementary flash effect should still be quite noticeable; see figure 43). A similar masking could be expected in pink stones with a purplish pink to purple flash effect.

Potentially Dangerous Use of Laser Light. In late 1993, a method for testing fracture-filled diamonds using a handheld laser pointer was suggested (Everhart, 1993a,e). The procedure calls for examining the stone in a darkened room, under low-power magnification, while illuminating it with the laser's intense red light. This will reportedly cause the entire area of a filled break to glow red, thereby clearly showing the extent of the filled area.

We strongly advise against using this technique. First, in experiments conducted by the authors using a 3.0-mw laser pointer, the results were ambiguous: Both filled and unfilled fractures reflected the laser light with no appreciable, consistent difference in their appearance. Second, laser pointing devices are labeled with clear warnings to avoid direct eye exposure. Diamond surfaces—and fractures within diamonds—are highly reflective, so the method could result in the intense laser light being reflected through the microscope's lenses and into the user's eyes. Because of this potential health hazard, as well as the ambiguous results, we recommend avoiding this method altogether.

Figure 41. Some untreated diamonds reveal an orange-brown staining in their fractures, which is actually a naturally occurring iron compound. Such staining should not be mistaken for a flash effect. Photomicrograph by John I. Koivula; magnified 35×.

Figure 40. The high relief and feathery appearance typical of many unfilled breaks have not been noted in any of the fracture-filled diamonds examined by the authors. Photomicrograph by Shane F. McClure; magnified 33×.

Inadequacy of Loupe. The last and perhaps most important precaution has to do with the type of magnifier used. As noted by Koivula et al. (1989), a 10× loupe was adequate to detect diagnostic features of filling treatment in some of the earlier Yehuda-treated diamonds. We have since seen, however, that the identifying features can be very subtle, and other features, such as iridescent feathers, might be mistaken for filled fractures. Thus, evidence of fracture filling may be completely overlooked, or misidentified, if the stone is examined only with a low-power hand magnifier. Given this situation, *it is the authors' opinion that a 10× loupe cannot be relied on to detect characteristic features in all filled diamonds. Instead, a binocular gemological microscope with a range of lighting options should be used.*

CONCLUSION

The current investigation has confirmed that the fracture-filling processes of the three firms studied—Yehuda, Koss, and Goldman Oved—can effectively improve the faceup appearance of some diamonds. All three can improve the apparent clarity of a diamond by one or sometimes two grades. The

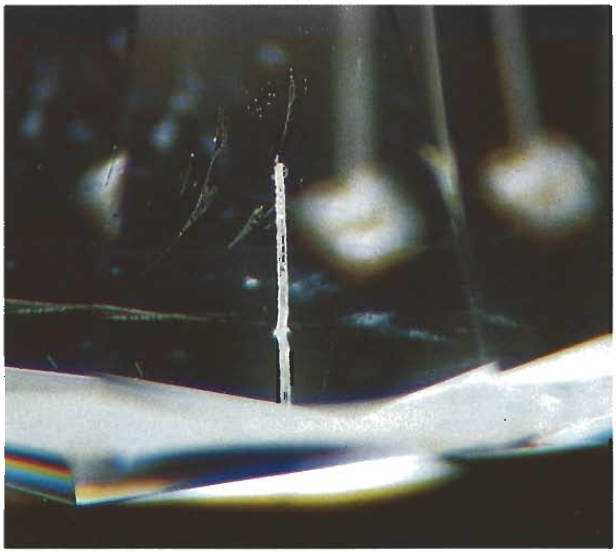


Figure 42. The laser drill hole in this diamond appears to end abruptly within the stone at no obvious feature. In reality, it terminates at a totally internal feather that was filled through the laser drill hole. Note the few minute gas bubbles in the filled break. Photomicrograph by Shane F. McClure; magnified 40×.

Yehuda treatment was found to lower apparent color grades in some but not all stones (Koivula et al., 1989; Koivula and Kammerling, 1990). In one group of Koss-treated diamonds (box A), the treatment lowered the apparent color grade of some stones, but the Goldman Oved samples showed no such effect.

Regardless of some of the claims made in the trade press, the fracture-filling treatments of *all three firms* can be detected using a binocular gemological microscope. While standard darkfield/brightfield illumination is often adequate for detecting the treatment, in some instances—especially with mounted stones—fiber-optic and other illumination techniques may be required. The most consistently encountered diagnostic microscopic features noted in all three products were as follows:

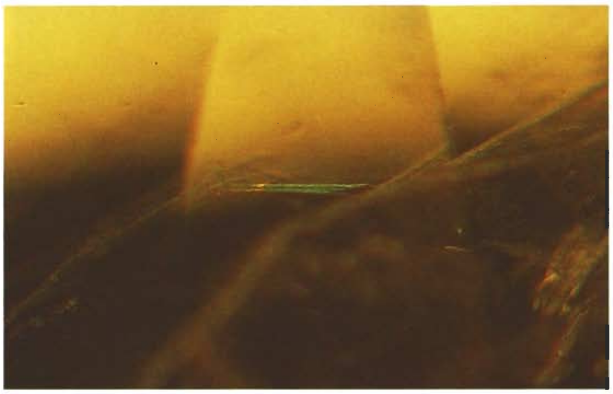
- Flash effects
- High-relief areas representing incomplete filling (trapped bubbles in the filling and/or thin, unfilled areas at surface entry points)
- Cloudy filled areas of reduced transparency that appear white

Other features noted with magnification in the products of one or more firms include a crackled texture or predominantly yellow color to the filler, as well as cloudy surface markings. Advanced laboratory tests that have proved useful in detecting the treatment are: X-radiography, which reveals the X-ray opacity of the filling material, as it typically contains one or more heavy elements; and EDXRF chemical analysis, which can detect trace amounts of heavy elements, especially lead (which was documented in the fillers of all three firms).

Although the suites of diagnostic features for filled stones from the three firms are not identical, there is significant overlap—both in the general types of features and in such specifics as the flash-effect colors. It also should be remembered that there are other firms that perform fracture filling of diamonds. It is reasonable to expect that their identifying features overlap those of the stones treated by Yehuda, Koss, and Goldman Oved. Thus, we conclude that *no individual gemological feature or suite of features will conclusively identify which firm treated a specific stone.*

The durability-testing phase of this study also provided useful information. In particular, prolonged exposure—or numerous short exposures—to commonly employed cleaning methods may damage the filling substances. Although such damage might be minor—as in the removal of a minute amount of filler at surface entry points—it could reveal treatment that had previously been unnoticed. For example, the appearance of a fine scratch on the surface where none had previously been

Figure 43. The orange flash from this filled fracture is almost completely masked by the yellow body color of the diamond. A small area of the complementary blue flash (seen here as green, because of the yellow body color) is visible in the center of the illustration. Photomicrograph by John I. Koivula; magnified 30×.



observed would be an unfortunate way of "disclosing" fracture filling to a customer. Jewelry repair procedures involving direct exposure to heat (as in retipping prongs) will damage and partially remove the filler from such treated diamonds. On a more positive note, we found that jewelry repair procedures involving *indirect* heating (as in sizing a ring) might not damage the fillings. Because exposure to long-wave UV radiation is comparable to exposure to sunlight (since natural sunlight contains a significant "UV-A" component), even prolonged exposure to daylight might have a negative effect on the appearance of fracture-filled diamonds. As with optical characteristics, the results of our durability testing do not substantiate some of the claims made in the trade press.

Because products from the various firms did not respond identically to all of the durability/stability tests—and in light of the highly publicized exchanges in the trade press concerning some of these products—it would not be surprising if these data were selectively used to help "substantiate" one product's alleged superiority over another. In this regard, there are some important considerations. First, the various durability tests in this study were, with one exception, performed on only one filled diamond from each of the three manufacturers. Therefore, *general conclusions cannot be drawn from the results of durability testing.* For example, the fact that one test diamond was not damaged by prolonged ultrasonic cleaning should not be misinterpreted to mean that all diamonds treated by that firm will necessarily be immune from such damage. Another diamond with larger or more extensive filled breaks—or one subjected to even longer cumulative cleaning times—might react differently.

Furthermore, while there may be some differences in the durability or stability of the products from the different firms, the "pedigree" of a filled diamond will probably not be known to the jeweler who takes it in for cleaning or repair. Thus, it may be best to proceed under the principle of "lowest common denominator," and not subject a filled diamond to any cleaning or repair procedure that has been shown to damage filled stones from any firm.

Because of these durability and stability concerns, it is evident that the apparent clarity and color grades of such treated stones can change over time. The GIA Gem Trade Laboratory therefore continues its policy of not grading diamonds that are found to have been fracture filled. Given the increasing numbers of fracture-filled diamonds, and the prospect that faceted synthetic diamonds will someday be commercially available, everyone in the gem, jewelry, and diamond industries should begin *now* to think of diamonds not only in terms of grading but also in terms of identification (as they currently do with colored stones).

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