THE CHARACTERISTICS AND IDENTIFICATION OF FILLED DIAMONDS

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The filling of surface-reaching cleavages, fractures, and other separations in diamonds has generated a great deal of concern during the last two years. This article reports on the study of six diamonds both before and after a filling treatment and the examination of 12 additional filled stones. Microscopy provides the best means of identifying that a stone has been filled. Chemical and other analyses indicate that the filling material is a type of glass. The filling will not withstand all standard jewelry manufacturing and repair procedures.

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Acknowledgments: The authors thank Dr. James E. Shigley, Bob Crowningshield, Tom Moses, Karin Hurwit, Loretta Bauchiero, James Lucey, Mark Mann, Duane Pebworth, and James Morton for their help in this project. John Armstrong and Mary Johnson, of the California Institute of Technology, helped with the chemical analyses. The assistance of Eli Haas and Martin Rapaport was invaluable. Unless otherwise noted, all photomicrographs are by John I. Koivula. This study was supported in part by the Dr. Byron C. Butler Fund for Inclusion Research.

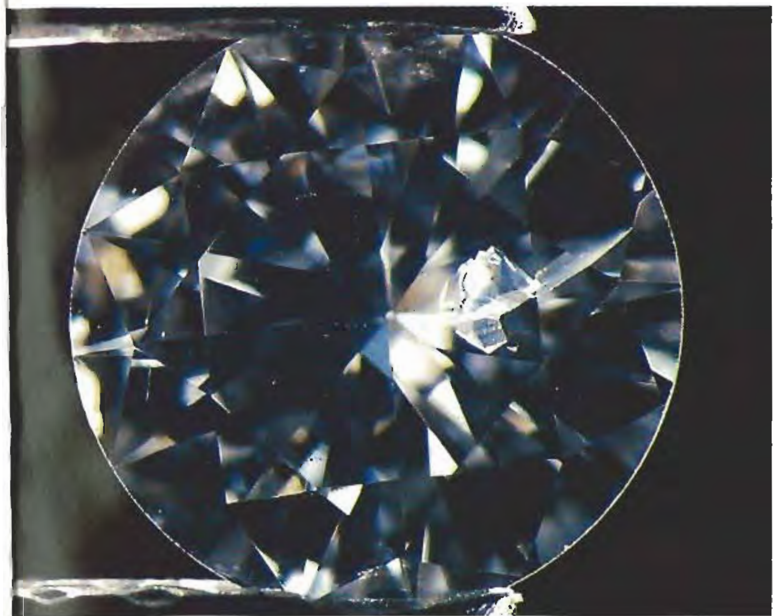
Gems & Gemology, *Vol. 25, No. 2, pp. 68–83*

Few would argue that the sale of loose diamonds and diamond-set jewelry is vital to most jewelers. The introduction of a new diamond enhancement process, then, is likely to generate concern at all levels of the diamond trade, from the small retail store to the boardrooms of the International Diamond Manufacturers Association and the World Federation of Diamond Bourses. The filling of surface-reaching cleavages and fractures has had just such an effect throughout the industry.

Developed by Mr. Zvi Yehuda, of Ramat Gan, Israel, this enhancement can alter the apparent clarity of many faceted diamonds. By decreasing the visibility of cleavages, fractures, and other separations that reach the surface of a stone, this treatment not only can potentially change the perceived clarity from SI to VS, but it can also give diamonds in the low-clarity "imperfect" range a much more desirable—and, therefore, salable—appearance overall (figure 1).

Since September 1987, there has been ongoing discussion in the trade press concerning such filled diamonds. According to a recent announcement (Everhart, 1989), this topic drew "major attention at the recent (24th) World Diamond Congress in Singapore." In addition, members of the International Diamond Manufacturers Association and of the World Federation of Diamond Bourses have just passed resolutions declaring that "knowingly selling diamonds treated by this method without disclosing that fact is a fraud and conduct not becoming to a member" (Everhart, 1989; Maillard, 1989b).

This form of diamond enhancement was first encountered by GIA in January 1987, when a colleague submitted a few of these treated diamonds for examination. On August 14, 1987, GIA issued a press release to alert the trade to the existence of this filling procedure; just one day earlier, Mr. Nubuo Horiuchi, of the Central Gem Laboratory in Japan, had issued International Colored Stone



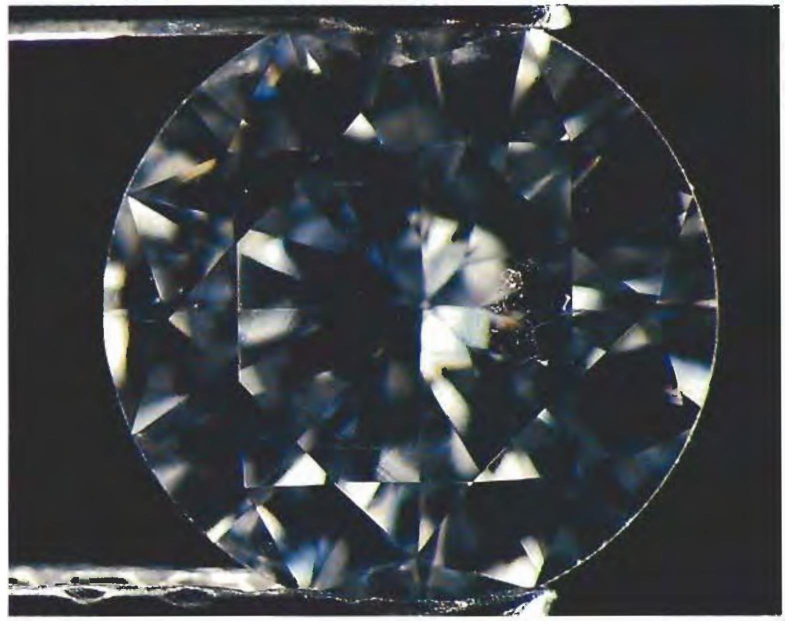


Figure 1. The filling of fractures, cleavages, and other separations in a diamond can have a significant effect on the apparent clarity of the stone. Before enhancement (left), this 0.25-ct diamond, with its eye-visible inclusion under the table, was clarity graded as an I_1. After the stone was treated by the Yehuda filling method at Dialase Inc., the apparent clarity (right) had risen to a grade of SI_2.

Association Lab Alert No. 7 on this treatment. Both reports described the effectiveness of the treatment in enhancing apparent clarity, with the GIA statement indicating that stones that would have originally graded I_2 and I_3 might be improved to the equivalent of an I_1. Both notices also stated that the stones examined thus far were reportedly treated in Israel; Mr. Horiuchi suggested that the filling material might be silicone oil.

In September 1987, GIA's Research Department acquired a 1.22-ct round brilliant-cut diamond that had been treated, reportedly by the filling process, and subsequently boiled in sulfuric acid in a diamond boiling kit. This cleaning procedure had partially removed the filler to a uniform depth from the system of intersecting cleavages reaching the table and crown facets of the stone, which resulted in an obvious white cruciform pattern just below the surface (figure 2). A close examination of this diamond provided some visual clues to the microscope-aided identification of filled diamonds (Koivula, 1987). More recently, with the opening of a diamond-filling operation in New York City (Maillard, 1989a), a number of articles have appeared in the trade press regarding these and other clues to identifying diamonds enhanced in this manner (see, e.g., Koivula et al., 1989; Shor, 1989).

To more accurately evaluate the effectiveness of this filling procedure, and to thoroughly characterize these fracture and cleavage fillings for identification purposes, we documented six diamonds both before and after filling and an additional 12 diamonds that were known to be filled. Tests not only included microscopic examination of these stones, but also analysis of the filling material and

Figure 2. A white cruciform pattern appeared in this diamond after an acid bath removed part of the filling compound that lay near the surface of the stone. A close examination of this 1.22-ct diamond provided the first visual clues to the microscope-aided identification of filled diamonds. Magnified 10×.

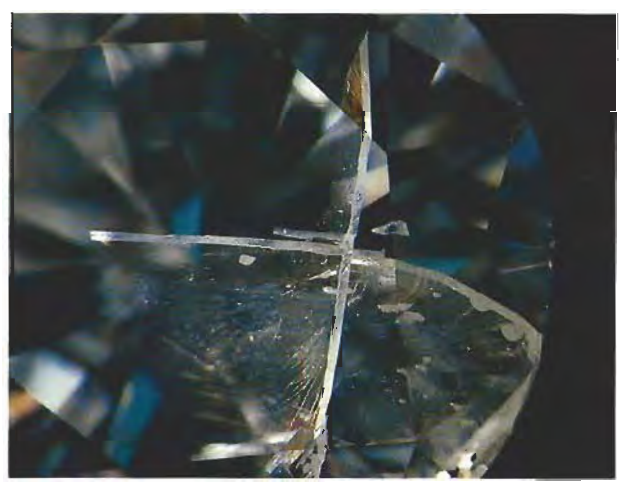


TABLE 1. Color and clarity of six round brilliant-cut diamonds before and after filling by the Yehuda method.[a]

No.	Measurements (mm)	Weight (ct)	Before		After	
			Color	Clarity	Color	Clarity
1	6.21–6.28 × 3.72	0.92	**L**	**Below I_3**	**M**	**I_3**
2	3.76–3.81 × 2.43	0.22	K	I_1	K	I_1
3	6.21–6.28 × 3.74	0.90	**K**	I_3	**L**	I_3
4	5.24–5.31 × 3.09	0.51	I	**SI_1**	I	**VS_2**
5	4.22–4.28 × 2.34	0.25	**E**	**I_1**	**F**	**SI_2**
6	4.06–4.18 × 2.39	0.25	**I**	I_1	**J**	I_1

[a]*All stones were graded independently at the GIA Gem Trade Laboratory for research purposes only. The laboratory does not offer grading reports on filled diamonds.*

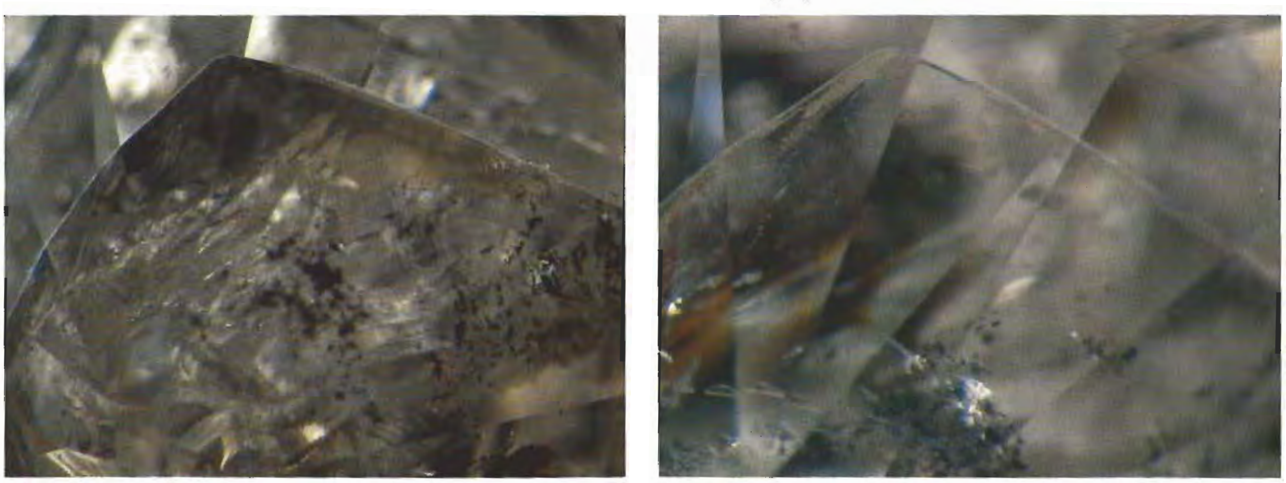
evaluation of the durability of the treatment when a filled stone is exposed to routine cleaning and jewelry manufacturing and repair procedures.

EFFECTIVENESS OF THE FILLING PROCEDURE

The stated purpose of this treatment is to improve the clarity of a diamond. In reality, this treatment has its roots in the oiling of emeralds, a practice that dates back to the ancient Roman Empire (Nassau, 1984). According to Ringsrud (1983), "The principle of oiling is simply that while air-filled fractures in gemstones are highly visible, a fracture filled with a transparent oil or some other suitable material will be much less apparent." The principle of the diamond-filling procedure is the same: replace the air that normally fills such breaks with a transparent substance that has an index of refraction close to that of diamond. The result is a much less visible separation that improves the overall appearance of a diamond to the unaided eye and, according to the trade press, greatly enhances the apparent value of the stone (Everhart, 1987; "U.S. Diamond Dealers . . . ," 1987; Weil, 1988; "Filler Must Be Disclosed," 1988). The GIA Gem Trade Laboratory in New York has even examined samples of laser-drilled diamonds that have been treated in an attempt to fill fractures reached by the laser.

According to the *Rapaport Diamond Report* (September 4, 1987), the filling compound developed by Yehuda is introduced into the diamonds at relatively high pressures (in the range of 50 atmospheres) and a temperature of 400°C. This procedure has not, however, been confirmed; and it may actually involve the use of a vacuum rather than the more technically difficult high-pressure environment suggested. The precise composition

Figure 3. A large cleavage system reduced the transparency of this 0.92-ct diamond (no. 1 in table 1) before treatment (left). After treatment (right), there was a dramatic reduction in the visibility of the cleavage, with a significant improvement in the apparent clarity. Magnified 15×.



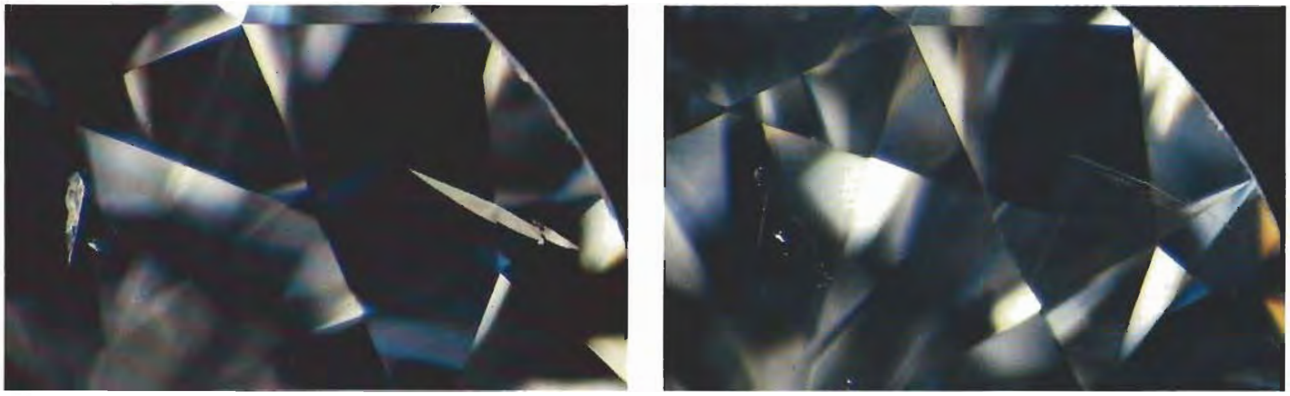


Figure 4. The small cleavages shown here contributed to the SI_1 clarity grade in this 0.51-ct diamond (no. 4 in table 1) before treatment (left). After treatment (right), the clarity was graded as VS_2. Magnified 15×.

of the filling material has not been disclosed by Mr. Yehuda; in fact, he reportedly denies that a filler is used in his process (Everhart, 1989).

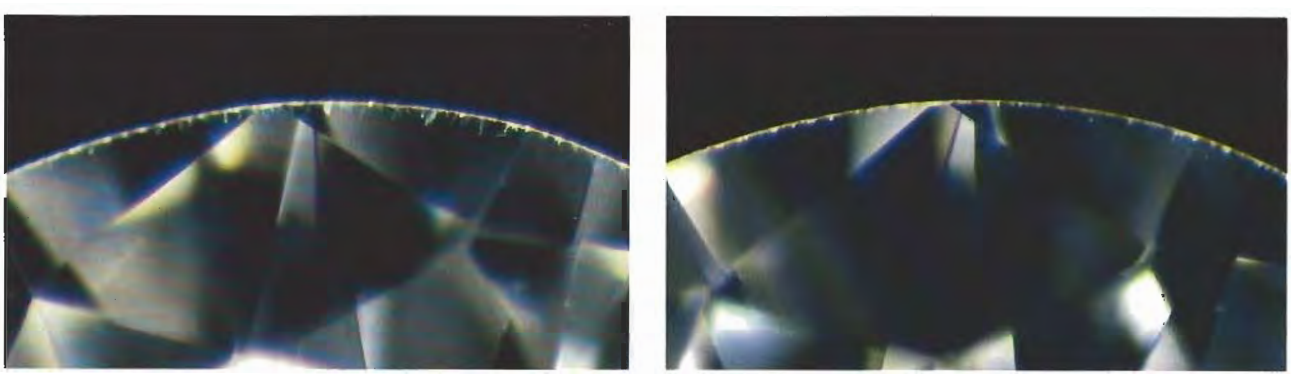
The six round brilliant-cut diamonds that served as the core of our test sample were selected from GIA's collection based on the position and appearance of the breaks that reached the surface of each. Prior to undergoing the filling procedure, all six stones were thoroughly documented photographically, weighed on an electronic balance, and independently graded for color and clarity by GIA Gem Trade Laboratory diamond graders (table 1). The six stones were then submitted by members of the trade for enhancement in New York at Dialase Inc., which purports to use the filling procedure developed by Mr. Yehuda in Israel (Maillard, 1989b).

After treatment, the stones were thoroughly re-examined and then photographed, weighed, and regraded for clarity and color. It is important to emphasize that this "before-and-after" color and clarity grading was performed by GIA Gem Trade Laboratory graders for research purposes only; it is *not* a GTL service offered on filled diamonds (as is discussed later in this article).

The results of the examination after treatment are also reported in table 1. They indicate that the treatment appeared to be very effective in some stones (figure 1; stone 5 in table 1), while in others there was no significant change. In the cases that showed significant clarity improvement, the specific changes were often dramatic. As evident in figure 3, the appearance of large cleavages was improved radically. Figure 4 illustrates an equally impressive degree of clarity enhancement of much smaller features once they have been filled. Even the general appearance of bearded girdles was improved by the filling procedure (figure 5).

One of the diamonds selected for this experiment was chosen because it contained a large diamond crystal (knot) under the table from which a large cleavage extended into the crown (figure 1).

Figure 5. Before treatment (left), the slight bearding along the girdle of this 0.25-ct diamond (no. 6 in table 1) is relatively easy to see. After treatment (right), the visibility of the bearding appears to be significantly reduced. Magnified 20×.



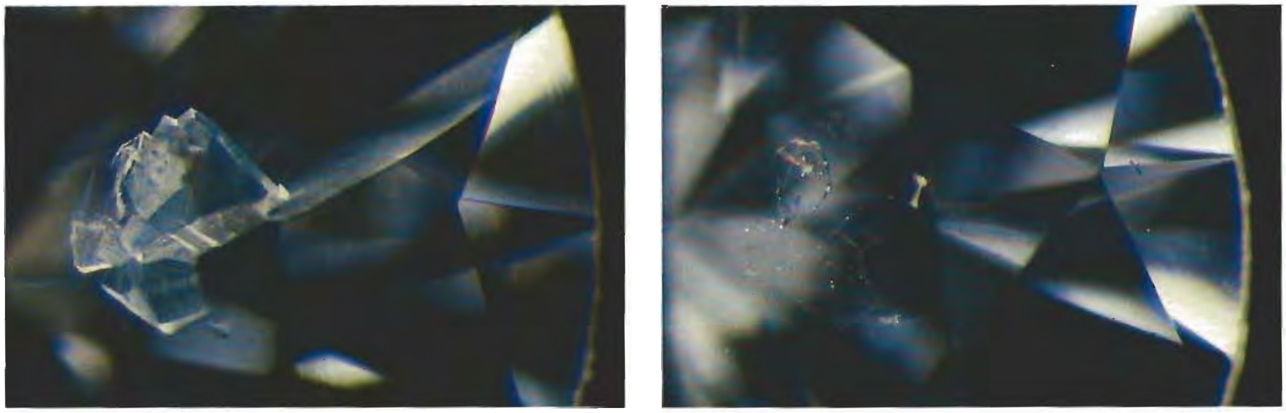


Figure 6. The white-appearing interface around the diamond crystal inclusion (knot) in the diamond shown in figure 1 (stone 5 in table 1) is readily visible before filling (left). After filling (right), the interface and knot-associated cleavage both seem to have virtually disappeared. Magnified 20×.

The crystal was surrounded by a white interface that made the inclusion highly visible when the stone was viewed table-up (figure 6, left). We had already established that the visibility of surface-reaching cleavages and fractures could be reduced by the filling treatment, but could the visibility of a knot be reduced by filling the interface between the host diamond and the knot? The answer to this question is dramatically provided in figure 6, right. Thus, it seems likely that virtually *any* separation reaching the surface of a diamond can be filled by this process.

Another factor considered was weight. Could the filling compound also increase the weight of the original stone? The six stones treated for this report did not show any evidence of weight gain after the enhancement. This is undoubtedly because the filler incorporated is essentially a thin film, so in even the most badly cleaved and fractured diamonds its weight is negligible.

A somewhat surprising finding was a generally poorer color after treatment: Four of the six stones showed a drop of one full color grade (again, see table 1). Even two of the stones that showed no significant clarity improvement graded lower in color after treatment.

The enhancement appears, then, to be very effective in improving the apparent clarity of most diamonds with appropriate inclusions. There was no effect on the weight of the diamonds tested. However, the treatment may have a negative impact on the overall color of the stone. The next question was: How can the presence of a filling best be detected? We looked to microscopy for the answer.

For the purposes of microscopic and other testing, we also examined an additional 12 diamonds known to have cleavages and fractures filled by this or a similar process. These stones ranged in weight from 0.22 ct to almost 2 ct, and represented a variety of cutting styles, including pear shapes, marquises, emerald cuts, and round brilliants. In apparent color—again, graded for research purposes only—they ranged from colorless (E, on the GIA color grading system) to fancy brownish yellow. During the course of this study, more than 50 diamonds found to be filled were received at the GIA Gem Trade Laboratory, Los Angeles, and examined by several of the authors. Information obtained from these stones has also been included as appropriate.

VISUAL IDENTIFICATION: MICROSCOPIC EXAMINATION PROVIDES PROOF

At first glance with the unaided eye, a filled diamond may appear slightly greasy or oily with a very slight yellowish overtone. The yellowish overtone is most apparent in stones with many treated areas, and may be due to the color of the filling compound.

A person experienced with diamonds is likely to feel that something is "not quite right" when encountering one of these filled stones. This is particularly true if there are a large number of treated eye-visible breaks. To follow up on these "first suspicions," we found that examination of the fractures and cleavages with a microscope is the single best means of quickly and positively recognizing this new form of diamond enhancement. Careful study of the 18 filled diamonds used for this report revealed a number of micro-

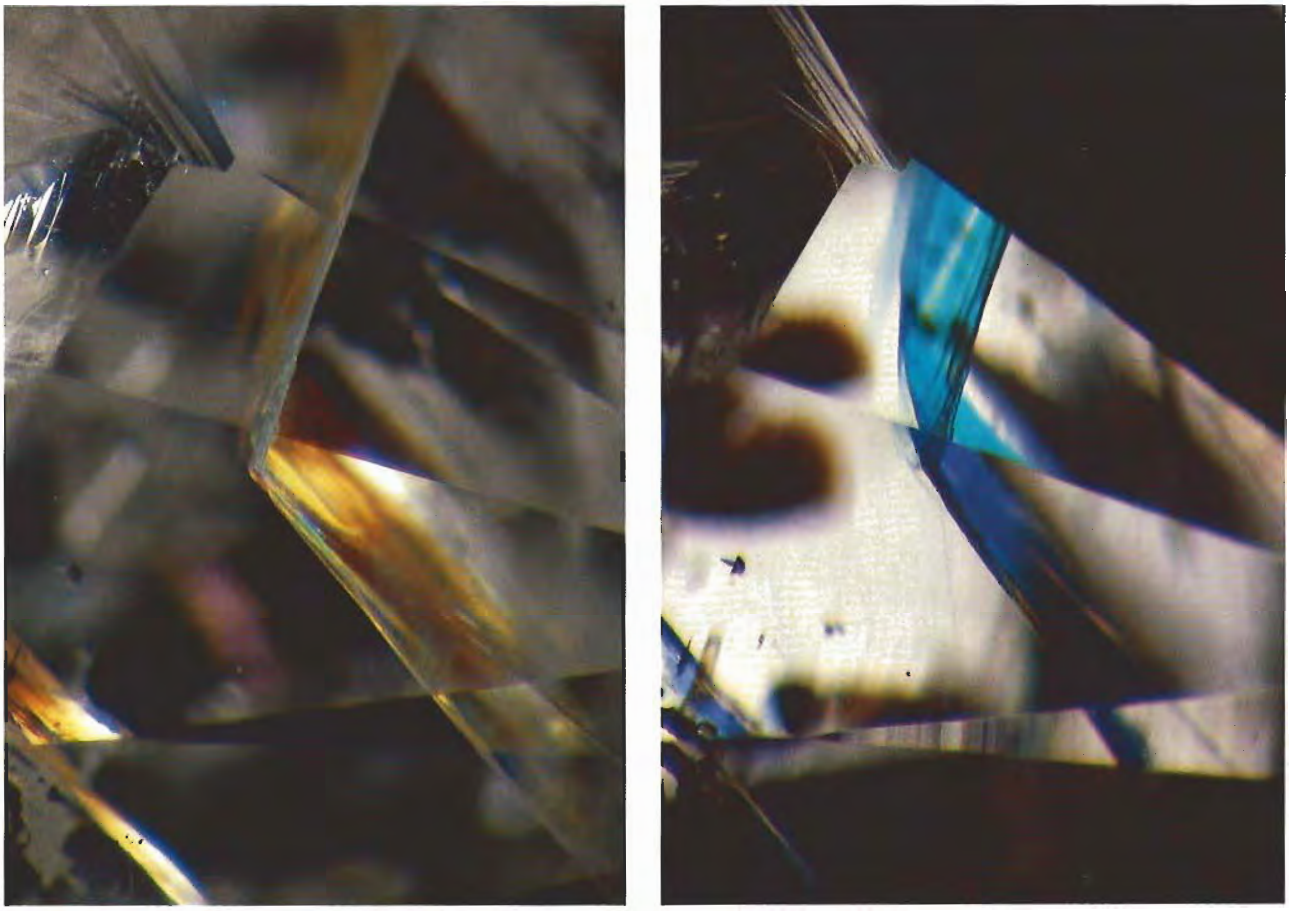


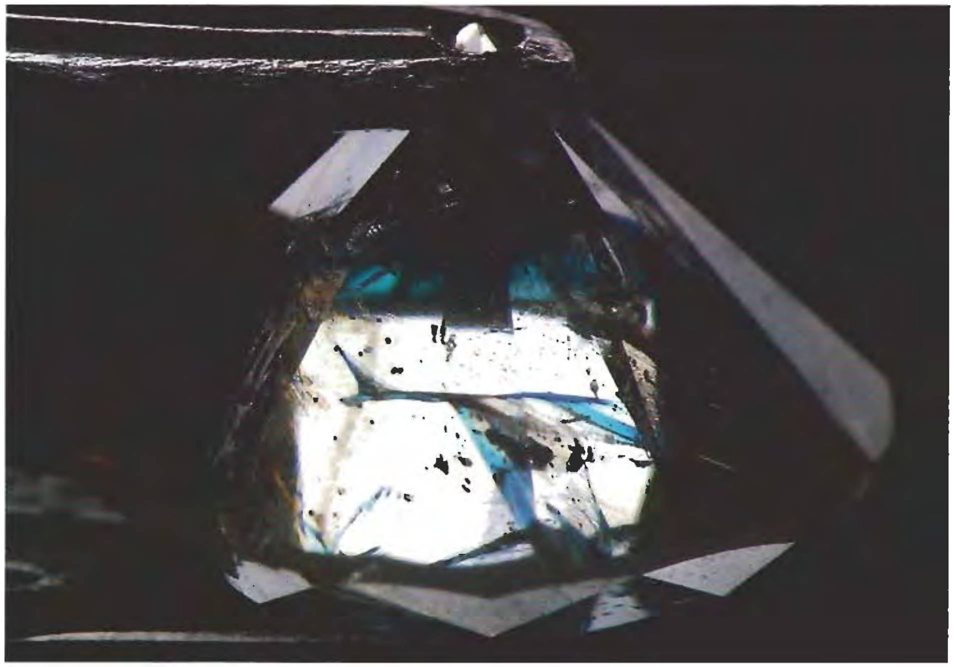
Figure 7. A "flash effect" is one of the most important visual features of filled breaks in a diamond. In darkfield illumination (left), this effect is a very characteristic yellowish orange; it changes to a distinctive blue when the stone (here, no. 1 in table 1) is rotated slightly to a position where the background becomes bright (right). Magnified 20×.

scopically resolvable features that indicate that a stone has been enhanced in this manner. Although no one feature has been seen in all of the stones examined to date, the presence of one or more of these features in a diamond can provide evidence that the stone has been filled.

"Flash Effect." One of the most obvious and most common characteristics of the filled fractures and cleavages is what we call a "flash effect." Filled breaks in most of the treated diamonds examined to date, and in all six that we had treated by Dialase Inc., have revealed a very characteristic yellowish orange interference color in darkfield illumination that changes to an intense vivid "electric" blue when the stone is rotated very slightly to a position where the background turns bright through secondary reflection (figure 7). As a treated diamond is tilted back and forth, these interference colors will change from orange to blue and back to orange again in a flashing manner—hence, the "flash effect" designation. These "flash effect" colors are seen when the viewing angle is very steep, approaching a direction parallel to the plane of the treated fracture or cleavage (i.e., edge-on with the separation).

Provided the proper illumination is used, the "flash effect" can be so obvious that it is easily seen even at low magnification (figure 8); in some cases a standard 10× loupe is sufficient. In fact, stones with fractures or cleavages in the table-to-culet direction probably are not good candidates for filling, as the "flash effect" will be too readily visible through the table. The yellowish orange "flash effect" may be difficult to see in dark brownish yellow to orange diamonds, although, as is evident in figure 9, the vivid "electric" blue is still quite apparent. It must be noted, though, that in extremely small separations—a bearded girdle, for example—the "flash effect" may not be visible

Figure 8. Even at 10× magnification, "flash effects" may be seen in some filled diamonds.



at all. Nor was it visible even in some larger breaks in some of the filled diamonds recently examined at the West Coast GIA Gem Trade Laboratory.

Cleavages and fractures that reach the surface of an *untreated* diamond may be decorated with a rusty orange-colored stain of iron-containing naturally derived epigenetic compounds (figure 10) that could at first be mistaken for the yellowish orange "flash effect." In addition, *unfilled* fractures in untreated diamonds sometimes behave as thin films and display bright iridescent colors when viewed in certain directions (figure 11). However, neither the rainbow-like arrangement of the colors nor the "feathery" appearance of the cleavage itself has been observed to date in an artificially filled fracture or cleavage. These iridescent colors in an unfilled separation are most likely to be seen at an angle nearly perpendicular to the plane of the break. Again, the best viewing angle for a "flash effect" is near-parallel (edge-on) to the break.

Flow Structure. Another very distinctive and relatively common feature of this enhancement process is the flow structure of the filling material (figure 12). Apparently the compound is intro-

Figure 9. Even though the orange "flash effect" is difficult to see in this 1.32-ct brownish yellow diamond, the blue "flash" is clearly visible. Magnified 15×.

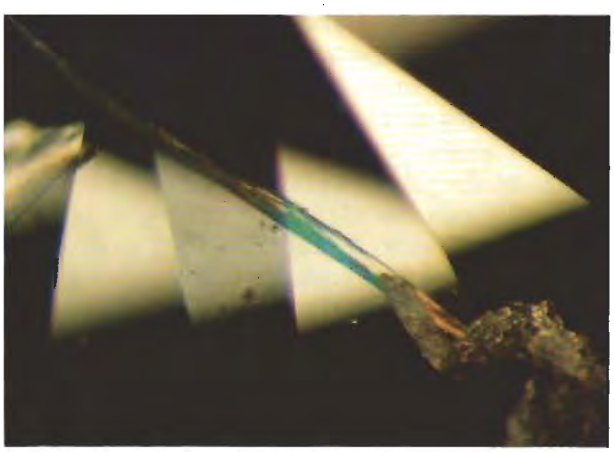
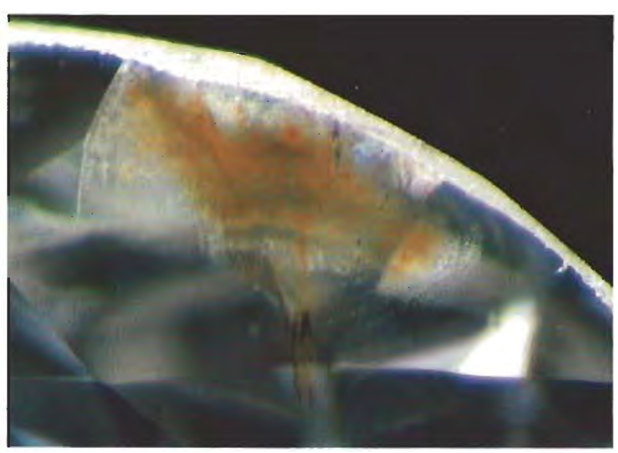


Figure 10. Epigenetic iron stains, such as the one decorating this cleavage, should not be mistaken for the yellowish orange "flash effect" observed in filled breaks. Diamond courtesy of Gregg Lyell. Magnified 40×.



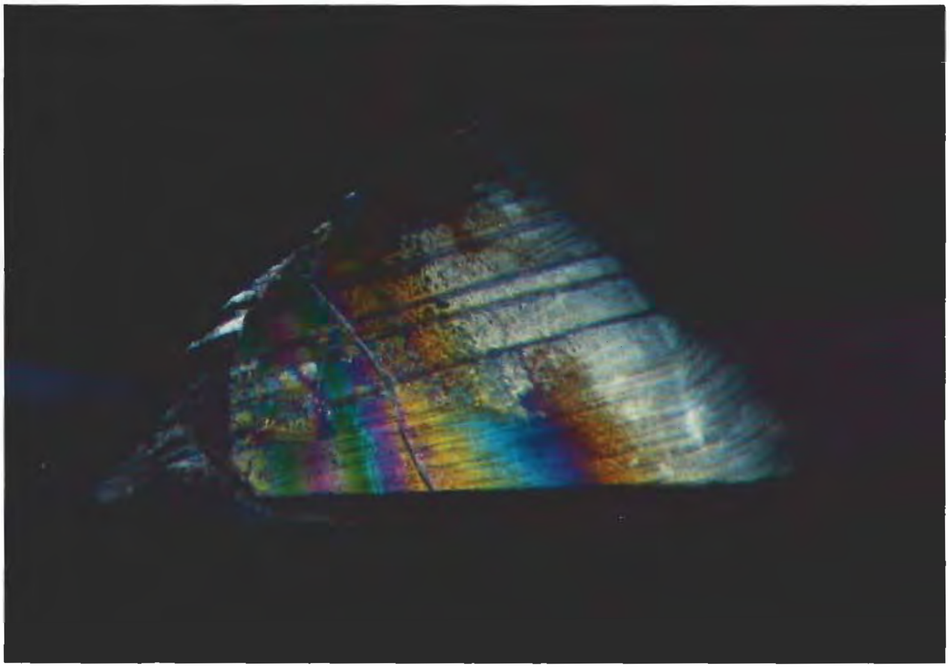


Figure 11. The "feathery" appearance and rainbow-like interference colors shown here are typical of an untreated *fracture in diamond. Similar texture and intense iridescence are not seen in filled cleavages or fractures. Magnified 35×.*

duced into the exposed separations at a high temperature in a molten state (a relatively high vacuum is probably used as well, which would then be followed by increasing pressure as the vacuum is released). The compound flows by capillary action into any open areas in the diamond, obscuring the normal feathery appearance (again, see figure 11) that is associated with breaks in *untreated* gems. Under magnification, the resulting flow structures generated by this enhancement look as if a glassy substance has been melted into the separations, creating a texture not seen in untreated diamonds.

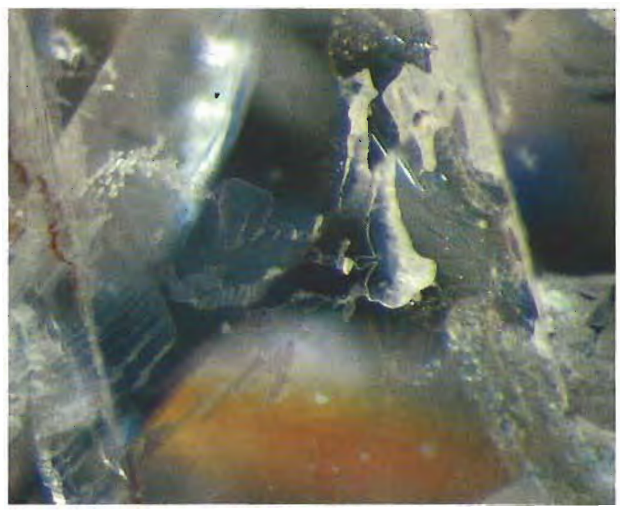


Figure 12. The melted appearance of the flow structure in a filled cleavage (here, in stone 1 from table 1) is very different from the distinctive pattern of an untreated break shown in figure 11. Magnified 35×.

Trapped Bubbles. Careful microscopic examination revealed at least a few flattened gas bubbles trapped within the filling material in a number of stones. Most of the bubbles observed are quite small, and in some areas they are rather plentiful, forming a fingerprint-like inclusion (figure 13). Some, however, were comparatively large and conspicuous (figure 14). Bubbles probably result from shrinkage of the filling material during cooling and/or from air trapped in the breaks by the liquified filling material.

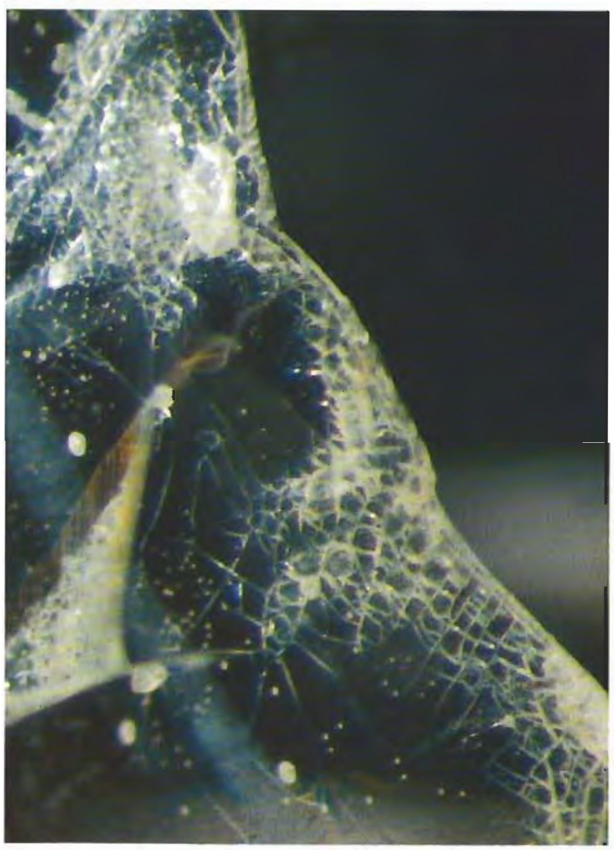
Crackled Texture. Although encountered in only three stones in our investigation, a crackled or web-like texture in a separation provides conclusive proof that it has been filled (figure 15). Somewhat akin to the mud cracks seen in dry lake beds, this feature was observed in only the thickest portions of the filled areas. Such a crackled texture may result from a combination of a partial crystallization of the filling material and uncontrollable or rapid shrinkage as it cools.

Apparent Color of the Compound. The filling agent is rarely thick enough for its true color to be judged, but when it is, as shown in figure 15, it seems to be light brown to brownish yellow or orangy yellow. This may explain the lowering of

Figure 13. These numerous small trapped bubbles form a fingerprint-like inclusion in the filled cleavage plane of this diamond (stone 3 in table 1). Magnified 35×.

the graded color in four of the six stones examined before and after treatment. One of the diamonds examined contained a thin void 0.5 mm long that was partially filled with the treatment compound, which appeared as a light brownish yellow irregular mass in direct association with a network of cleavages. We also observed a brownish compound in the laser drill hole of a diamond that was found to be filled (figure 16).

Figure 15. Although not as common as other identifying features, a crackled texture is additional evidence that a break has been filled. Note also the color of the filling revealed here. Magnified 30×.



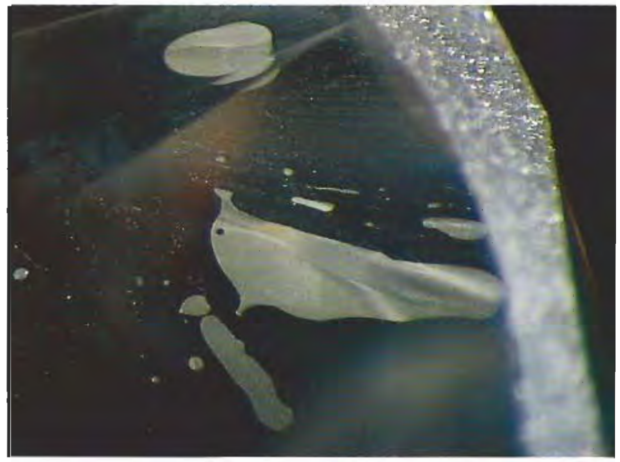
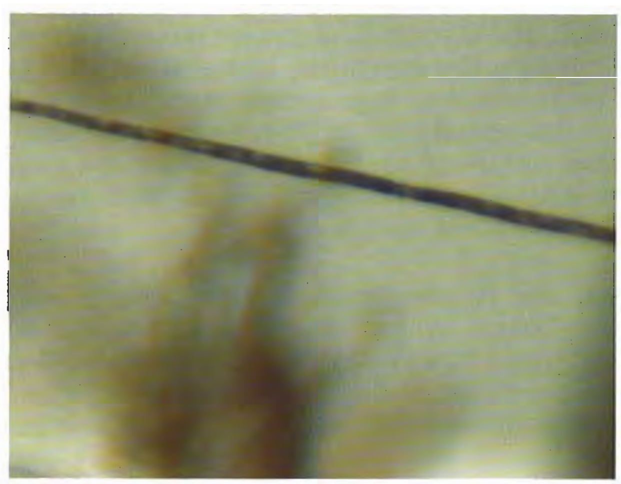


Figure 14. Large trapped gas bubbles such as those shown here probably result from the incomplete filling of the fracture in this diamond. Magnified 35×.

Estimating the Refractive Index of the Filling. Where the filling agent is in direct contact with the diamond, and there is no interfacial separation, it is possible to do a crude form of the Becke line test to determine which of the two—diamond or filler—has the higher refractive index. The results of such observational testing revealed that diamond, with a refractive index of 2.417, is the higher of the two. However, the bright-line appearance, together with the extremely low relative

Figure 16. The yellowish to brown color of the filling material is also evident in this filled laser drill hole. Photomicrograph by David Hargett; magnified 60×.



relief between the diamond and the filler, suggests that the refractive index of the filler is very close to that of diamond.

X-RADIOGRAPHY

X-radiography can also help identify filled diamonds. Working together in the New York GIA Gem Trade Laboratory, Bob Crowningshield and Tom Moses have had considerable success using X-radiography to separate diamond from many of its simulants, especially when dealing with melee-size stones (Tom Moses, pers. comm., 1989). Therefore, they decided to see if X-radiography could also be useful in determining the presence of filled cleavages and fractures in a diamond. Using a technique similar to that used for pearls, but with a lower voltage and current because of the high transparency of diamond, they took an X-radiograph of three diamonds that were known to have been filled. On the processed film, the filled areas appeared much more opaque to X-rays than did the surrounding diamond (Koivula and Kammerling, 1988; Koivula et al., 1989). This same test has since been done several times in the GIA Gem Trade Laboratory. Although in most cases the lab has observed the same positive results, in some stones fillings that had been observed with microscopy did *not* show up as opaque on the developed X-ray film. In addition, certain mineral inclusions have also proved to be much more X-ray opaque than diamond. Thus, while this test may be particularly helpful for a preliminary assessment on large lots of diamonds, we recommend that it always be used in conjunction with microscopy.

Inasmuch as Mr. Horiuchi's August 1987 ICA Lab Alert had suggested that the filling material might be a silicone oil, Messrs. Crowningshield and Moses decided to coat an untreated diamond with silicone grease to see if this coating would be less transparent to X-rays than the underlying diamond. There was no difference in transparency between the coating and the stone, so silicone was ruled out as the filling agent in the Dialase process.

On the X-ray film, the filled areas appear as distinct white, underexposed patches and zones on the diamonds, as illustrated in the stone shown in figure 17. On the basis of this appearance, it was evident that one or more atomically heavy, X-ray-opaque elements was present in the filling material. Further work was now necessary to determine its precise nature. For this purpose, a number of filled diamonds were studied using a variety of techniques to identify the presence and the composition of the filling material.

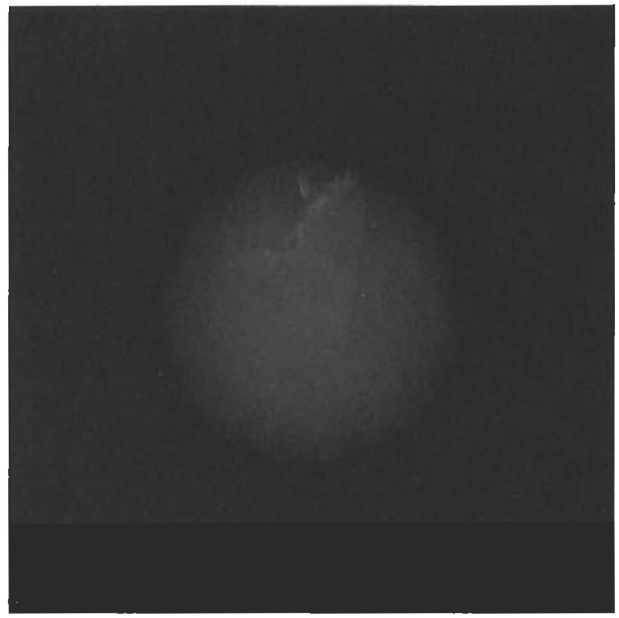


Figure 17. This X-radiograph of the 0.90-ct Yehuda-treated stone (no. 3 in table 1) shows the filled areas as opaque, unexposed white patches. X-radiograph by Karin Hurwit.

ANALYSIS OF THE FILLING MATERIAL

Impregnation with an organic substance in other gem materials can usually be detected using infrared absorption spectroscopy in the mid-infrared range (400–4000 cm^{-1}). Therefore, five known filled diamonds were run on a Nicolet 60SX Fourier Transform spectrometer (Fritsch and Stockton, 1987) to determine whether the filling was organic or inorganic in nature. To visualize the material in the fractures and obtain a local semiquantitative chemical analysis, a Camscan scanning electron microscope (SEM) in the Division of Earth and Planetary Sciences at the California Institute of Technology was used (analysts: John Armstrong and Mary Johnson) on stone 1 of table 1. For a precise chemical analysis and measurement of the concentration of light elements (oxygen in particular), a Jeol Superprobe 733 in the same laboratory was used (analyst: John Armstrong) on the same stone. Additional semiquantitative chemical analysis was performed on the six Dialase-treated stones and more than 25 additional filled diamonds with a Tracor X-ray Spectrace 5000 X-ray fluorescence spectrometer in GIA's Research Department.

Infrared Spectroscopy. None of the stones tested exhibited any noticeable additional absorption in the mid-infrared range, even when microscopic inspection had proved the presence of numerous filled breaks. This suggests that the filling material is not organic.

Scanning Electron Microscopy. Using a scanning electron microscope with the appropriate detectors (Stockton and Manson, 1981), one can obtain both a visual representation of the surface shape of the filling and a map of the spatial distribution of chemical elements in the near-surface layer of the material. The higher the atomic number (the heavier the element) is, the brighter this area of the black-and-white image will appear. Figure 18 clearly shows how at relatively low magnification (30×) the filled fractures stand out because of their very high contrast (almost white versus the very dark gray of the remainder of the stone). The fact that the tone of the material in the fractures is different from that of the bulk of the diamond confirms the presence of a foreign (noncarbon) material in the cracks. The very light tone of the filling also proves the presence of high-atomic-number elements.

Higher magnification (180×) reveals patches of various tones within the foreign material, although most of it appears white (figure 19). This means that the filling is not homogeneous.

Using the SEM's energy dispersive spectrometer (EDS) attachment, the analyst performed semiquantitative analysis on eight spots along the fracture illustrated in figure 19. All eight showed the presence of lead and chlorine as major constituents. Six also showed significant amounts of bismuth. Various other elements (e.g., sodium, potassium, calcium, aluminum, and silicon) were detected, but in negligible amounts. Similar results were obtained on other filled areas analyzed.

When both lead and bismuth were present in a filling, the lead-to-bismuth concentration ratio was generally around 2:1, although it dropped to 1:1 in one spot. The chlorine concentration consistently seemed to approximate the sum of the lead and bismuth concentrations.

Electron Microprobe. The presence of oxygen as a major constituent in the filling material was ascertained with the electron microprobe. The exact values of oxygen concentration could not be obtained because our gem specimens did not conform to the precise technical conditions required for this type of analysis.

X-Ray Fluorescence. This technique provides the overall bulk composition of the material under study. Because the instrument does not detect carbon, the bulk composition of the diamond itself is ignored, and the XRF spectrum obtained represents the composition of the inclusions, primarily the filling agent. This technique is very fast and does not require any preparation of the sample, so it also provides a rapid means of identifying filled diamonds. The diamonds were run in conditions appropriate for the detection of heavy elements. Only three of the samples examined by XRF contained lead and bismuth. The remaining stones had only lead, indicating a variable chemistry of the filling material from one stone to the next, similar to the inhomogeneities seen with the SEM in a single fracture from one spot to the next.

X-Ray Diffraction. To determine whether the filling agent has a definite structure, we decided to crush the 0.92-ct diamond (no. 1 in table 1) because it contained the greatest number of comparatively large filled areas.

To minimize contamination, the diamond was first broken with a pair of steel pliers and then crushed between two thick slabs of chalcedony. The largest of these fragments with the greatest number of freshly exposed surfaces was selected for the X-ray study. An iron scraper instead of diamond (again, to minimize contamination) was used to scrape the freshly exposed surfaces. The minute amount of powder obtained in this manner was mounted on a spindle in a Debye-Scherrer camera and exposed to X-rays from a copper target tube powered at 46 kV and 26 mA for 18 hours.

The X-ray pattern generated was very weak. The only two visible lines in the pattern could be identified as coming from the diamond (probably loosened cleavage fragments) and the iron scraper. No evidence of additional crystalline structure from the filling agent was present. This suggests that the filler is amorphous, and probably a glass.

Subsequent to this examination, we received a filled stone that had three readily discernable droplets adhering to the surface. X-radiography indicated that these droplets had the same opacity

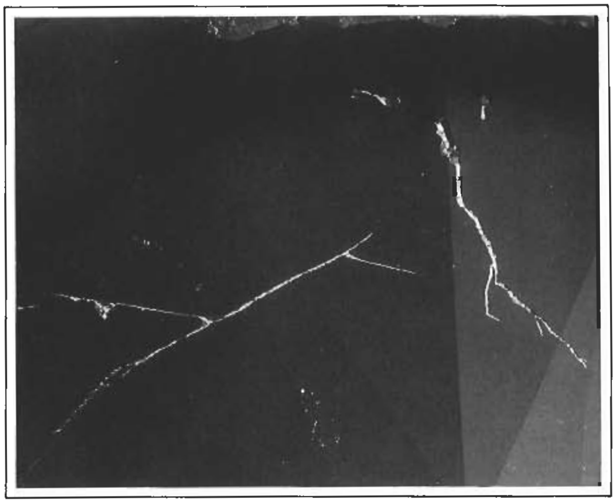


Figure 18. A scanning electron micrograph of filled breaks shows the very high contrast between the separation and the diamond. This indicates the presence of a foreign (noncarbon) material containing elements of high atomic number. Electron micrograph by John Armstrong; magnified 30×.

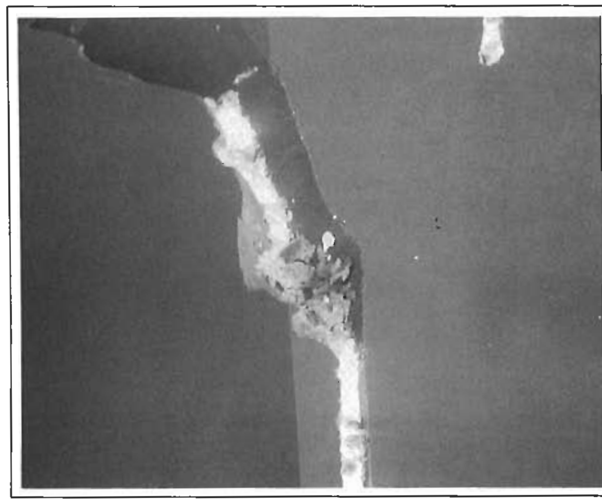


Figure 19. Higher magnification shows three different tones in the breaks illustrated in figure 18. These different tones indicate that the filling material is not homogeneous. Electron micrograph by John Armstrong; Magnified 180×.

as the filling material in the fractures of this stone. A scraping was easily made of the material from one of these droplets which X-ray diffraction proved to be amorphous, thus confirming our earlier findings.

Discussion. Chemical analysis showed that the filling material is a compound of lead, chlorine, and oxygen, with variable amounts of bismuth. (Boron is another possible component, but we do not have the means to detect its presence.) X-ray diffraction analysis proved that it is amorphous. Therefore, this particular filling material is undoubtedly a lead-containing glass-like material.

One can estimate what the optical and physical properties of this material might be by looking at materials of similar composition. There is only one natural lead-bismuth oxychloride, the very rare mineral perite, of formula $PbBiO_2Cl$. It is "sulfur" yellow in color (Gillberg, 1960), and its refractive index has been estimated to be over 2.4, very close to that of diamond (2.417). There are also various man-made "solder glasses" with high lead and bismuth contents that have low melting points and refractive indices in the 2-plus range. Because chlorides or oxychlorides of lead or bismuth typically have a high index of refraction (BiOCl: 2.15; $PbCl_2$: 2.199, 2.217, 2.260; Weast, 1980), their presence in the cleavages and fractures of a diamond would make these defects much less noticeable. In addition, such compounds are usually infrared transparent, which is consistent with our results.

Many materials belonging to this class also have low to moderate melting temperatures: $Pb(ClO_3)$ decomposes at 230°C, $PbCl_2$ melts at 501°C, $BiCl_4$ and $BiCl_3$ melt at about 230°C, and BiOCl melts at "red heat" (Weast, 1980). This characteristic would be particularly attractive for a process that requires that the filling material be drawn into the breaks while it is in liquid form.

Finally, some of the compounds mentioned above have an intrinsic yellow to red coloration. This could account for the slightly yellowish additional component (and consequent drop in apparent color grade) observed in some of the test stones after treatment.

DURABILITY AND STABILITY OF THE FILLING

One of the most important issues that surrounds any new enhancement is durability. Therefore, we conducted several tests using one of the six diamonds we had filled (stone 3, table 1) to determine the durability and stability of this treatment under a wide range of conditions to which a diamond might be routinely subjected (see table 2).

The first of these was ultrasonic cleaning,

TABLE 2. Durability and stability of the treatment in a diamond filled by the Yehuda method.[a]

Test	Procedure	Result
Ultrasonic cleaning	Stone placed in solution of commercial jewelry cleaner in ultrasonic cleaner for 30 minutes	No effect on treatment
Steam cleaning	Used two steam cleaners with pressure between 40 and 70 PSI, stone placed one inch from nozzle for 10 minutes	No effect on treatment
Boiling in detergent solution	Stone boiled in solution of water and dish-washing liquid for 30 minutes	No effect on treatment
Boiling in acid[b]	N/A	Filling damaged
Repolishing[c]	N/A	Filling damaged
Low temper-ature and thermal shock	Stone covered with 1/4 inch of liquid nitrogen to cool to −185°C	No effect on treatment
Stress	Stone set in four-prong head, with no prongs touching a filled area	No effect on treatment
Heat from jewelry repair	Prongs retipped with stone in place; 14K gold beads and 18K gold solder, flow point of 810°C, used with MECO midget torch with no. 40 tip; diamond heated a total of nine times	Beads of filling agent sweated out and appeared on surface of gap

[a]*All tests performed by the authors and other GIA staff members used one of the six stones submitted for treatment by the Yehuda method to Dialase Inc., New York.*
[b]*Test not performed by present authors; see Everhart (1987), Rapaport (1987), Koivula (1987), and Weil (1988).*
[c]*Test not performed by present authors; see Maillard (1989a).*

using a solution of commercially available jewelry cleaner. The stone, in the solution, was placed in an ultrasonic cleaner for 30 minutes. Microscopic examination confirmed that the enhancement was completely unaffected by this cleaning procedure.

Jewelers commonly steam clean diamond-set jewelry to remove gold-polishing compound and other debris that has been trapped in the mounting during manufacture or repair. To test the effect of steam cleaning on filled diamonds, GIA's Mark Mann and Jim Lucey used two steam cleaners so that steam pressure could be maintained between 40 and 70 pounds per square inch (PSI). Because the filled areas in the test diamond (the same one used for the ultrasonic cleaning test) were open to the surface on the table and crown, the stone was held in the steam jet in the table-up position approximately one inch from the steamer's nozzle. As the pressure dropped in one steam cleaner to near 40 PSI, the stone was placed under the nozzle of the other unit. Alternating between cleaners, the treated diamond was subjected to a jet of pressurized steam almost continuously for approximately 10 minutes. Again, microscopic examination showed that the treatment was completely unaffected.

Because jewelers will sometimes clean diamonds by boiling them in a solution of water and a detergent, we decided to boil the test diamond for 30 minutes in a solution of 100 ml of distilled water containing 10 drops of commercially available dishwashing liquid. Again, there was no effect on the treated areas.

Occasionally, diamonds are boiled in sulfuric acid to remove dop, prong, and tweezer metal from the surfaces of rough girdles and to clean out laser-drilled inclusions. We did not perform this test, however, because several sources (Koivula, 1987; Everhart, 1987; *Rapaport Diamond Report*, 1987; Weil, 1988) had already reported that acid boiling attacks the treatment (again, see figure 2). Likewise, we did not test the effect of the heat generated during a repolishing procedure, which Maillard (1989a) has already reported will undo the treatment.

To determine the effect of extreme cold on the test stone, we placed the diamond in a styrofoam cup and covered it with about one-quarter inch of liquid nitrogen; this almost instantaneously lowered the temperature of the diamond to −185°C (a temperature likely to be reached if the stone was cryogenically cooled for spectrophotometric examination). This not only tested the effects of extreme cold but, in the rapid way the cooling was done, it also subjected both the diamond and the filling agent to extreme thermal shock. Again, though, there were no signs of damage.

A diamond undoubtedly undergoes great stress during setting and, even more so, during the retipping of setting prongs. So, as a final step in durability testing, we turned the same test diamond over to Duane Pebworth and James Morton, of GIA's Jewelry Manufacturing Arts Department, to determine how it would hold up under the pressure generated during setting and the heat required for normal prong retipping.

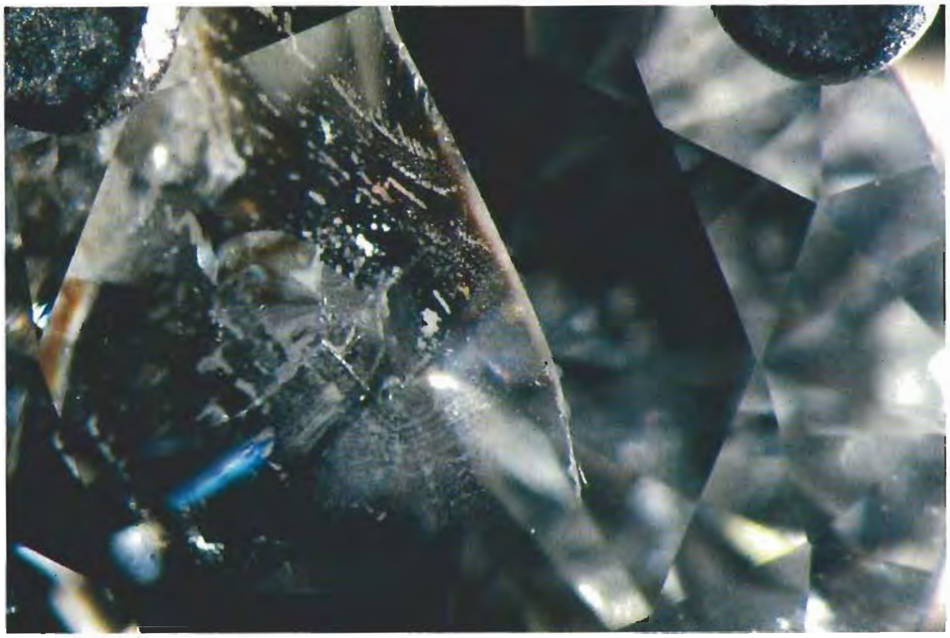


Figure 20. Damage to the filling (the same one illustrated undamaged in figure 13), became evident after the stone was exposed to the heat required in normal prong retipping. Magnified 15×.

The filled diamond, the same one illustrated in figure 13, was first set in a 14K white gold cast four-prong head. Care was taken not to place a prong directly on the filled area. There was no evidence of damage to the stone or the filling from the pressure required for the setting procedure. After ultrasonic and steam cleaning, the prongs were retipped using 14K white gold beads and 18K white gold solder with a flow point of approximately 1490°F (810°C). A MECO Midget torch with no. 40 tip was used with natural gas and oxygen. Following standard practice, the stone was fire coated first and then preheated slowly to bring it up to soldering temperature. During this step, the diamond began to turn a light yellow and, as retipping progressed, it became a dark, bright yellowish green. (This color change was totally unexpected. We do not know if it was caused by the presence of the filling material.) The diamond was heated a total of nine times: once for preheating, four times for tinning the solder, and four times for soldering the gold beads to create new tips on the prongs. As the stone was allowed to air cool, it reverted to its original color.

Examination of the diamond after retipping immediately showed that the filling had been damaged (figure 20); beads of the filling agent had sweated out of the separation and were visible as tiny droplets along the surface edge of the gap (figure 21). Their experience with this stone led Messrs. Pebworth and Morton to conclude that although most filled diamonds could probably withstand the pressure of setting (provided the work was done by an experienced artisan who was

Figure 21. Beads of the filling agent can be seen as tiny droplets along the surface edge of the gap shown in figure 20. They sweated out during the retipping procedure. Magnified 50×.

aware that the stone had been filled), such treated stones should NEVER be subjected to the heat of a jeweler's torch.

GIA GEM TRADE LABORATORY POLICY ON GRADING FILLED DIAMONDS

At the GIA Gem Trade Laboratory, diamonds determined to have cleavages and/or fractures that have been filled are considered in the same category as coated diamonds. Just as one cannot grade a coated diamond accurately without first removing the coating, one cannot grade a filled diamond accurately without first removing the filling material from the cleavages and fractures. In neither case can the true color of the stone be determined while the enhancement is present. Unlike the situation with most coated diamonds, however, there is no practical way to remove all of the filling material.

Laser drilling is a permanent, stable (unless, of course, the drill hole has been filled), and irreversible procedure that attempts to enhance the clarity of a diamond. The filling procedure, however, is not stable, especially when exposed to high temperatures, and cannot be considered a permanent change in clarity. More importantly for grading purposes, laser drilling alone does not affect the color of a diamond, while the filling procedure may produce a significant change (see table 1). The long-range stability of this color change has not been determined. It is, therefore, the present policy of the GIA Gem Trade Laboratory *not* to issue grading reports on any diamonds that contain any form of filling. The only GTL document given on such a stone is an identification report stating that the stone in question is a diamond and that it has been filled.

CONCLUSION

This relatively new process is designed to enhance the clarity of a diamond by filling breaks that reach the surface of the stone with a high R.I. material that, in effect, helps disguise these inclusions. It can improve the apparent clarity the equivalent of one full grade in many, though not all, appropriate stones. The presence of the filling material in a diamond is readily identifiable with a standard darkfield-equipped gemological microscope. The orange and blue "flash effects," flow structures, and flattened, trapped gas bubbles evident in the filled areas of these diamonds are easily detected by the properly trained diamond dealer or gemologist. In some cases, where the filled areas are particularly large, a standard 10× loupe may be adequate. Although not all of these characteristics were evident in all of the diamonds studied, at least one was present in each. In addition, the opacity to X-rays of the filling agent in most of the stones examined offers another useful test, provided it is applied in conjunction with microscopy.

The combined results of infrared spectroscopy, chemical analysis, scanning electron microscopy, and X-ray diffraction analysis show that in all probability the filling agent used is a lead-containing glass-like material.

This enhancement should in no way be confused with a repair process, although it has been referred to as such incorrectly, as evidenced by the title of an article in a recent trade publication: "Diamond Treatment that Removes Flaws Hits U.S. Market" (Everhart, 1989). The most recent advertising used to promote this enhancement process also uses the word *removed* in reference to "white gletzes" in its clarity improvement claims. This suggests that surface-reaching cleavages and fractures present before treatment are "removed" from the stone by the procedure and are no longer present after treatment. In fact, like the oiled fractures in emeralds, the cleavages and fractures in diamonds subjected to this form of filling still exist; they are just more difficult to see, thereby improving the apparent clarity of the stone.

There is nothing inherently wrong with this or any other enhancement process provided it is *always* disclosed at every level of sale. This filling process does improve the appearance of diamonds that, before treatment, were perhaps not marketable as jewelry-quality stones, although the GIA Gem Trade Laboratory has seen evidence of fillings in stones that might have graded in the VS range before treatment. In effect, this treatment also acts as a sealant, preventing dirt or other unsightly debris from entering laser drill holes and other open fissures in a stone.

The process does, however, appear to have a negative impact on the overall color of the stone. In addition, it is not durable in all normal manufacturing and repair situations. The filled breaks in these diamonds cannot take the heat required to repolish the stones or to retip the prongs on the mountings in which they are set. It therefore follows that filled diamonds that are sold without full disclosure may in time be discovered by the diamond cutter or bench jeweler who must work with them.

There has been continuous speculation that there is more than one vendor offering this type of filling service. One such rumor states that a similar procedure, using a molten, lead-based glass, is now being routinely done in India on low-quality goods. The slight variation in chemical composition noticed during scientific analysis supports the claim that there could be more than one filling material in the trade, or at least variability in the material used by the Yehuda operations. In addition, many of the filled diamonds seen recently in the GIA Gem Trade Laboratory show evidence of a filling procedure that is far less sophisticated (i.e., fractures are not completely filled, or have not been thoroughly cleaned before enhancement and thus show considerable dirt) than the one to which our 18 core test stones had been subjected.

It appears that the filling of diamonds is becoming more prevalent. Members of the trade have informed GIA that they have seen large parcels of filled diamonds. The more than 50 such stones examined at the West Coast GIA Gem Trade Laboratory in late May and early June lends support to these reports. Although most of these stones were from a single client, almost all of the several clients who have submitted diamonds found to be filled thus far indicated that they did not know their stones had been filled. With careful examination, however, most diamonds enhanced in this manner should be readily identifiable.

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