
GEMSTONE ENHANCEMENT AND ITS DETECTION IN THE 1980s

By Robert C. Kammerling, John I. Koivula, and Robert E. Kane

The impact that enhancements had on the gem market in the 1980s may be unparalleled in the history of the industry. Heat treatment brought hundreds of thousands of carats of blue and fancy-color sapphires and rubies to the trade, irradiation turned literally millions of carats of white topaz into attractive blue stones, and the filling of surface-reaching separations introduced an entirely new variable to the evaluation of color and clarity in diamonds. One of the key challenges to the gemologist during this decade was the identification of these and other enhancements. This article reviews the enhancements that were introduced or played a major role during the 1980s, focusing on their detection by standard gemological techniques.

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Concern about gemstone enhancements and the search for methods to detect them are not new. Written descriptions of treatment methods date back almost 2,000 years, and reports on enhancement detection were being published as early as 1502 (Nassau, 1984).

However, a number of developments in the last decade have brought greater attention to the subject of gemstone enhancement. These include the proliferation of enhanced stones in the market, the increasing sophistication of the treatment methods used, the need to relay accurate information both within the trade and to the consumer, and government interest in mandatory enhancement disclosure.

Because treatment can significantly affect the value of a gem, this article focuses on the one aspect of enhancement that is the gemologist's primary concern: detection. Through a review of the literature and the authors' own experience, this article examines those enhancements that were new in the 1980s or had a major impact on the gem market during the decade, and describes the detection methods that are now available. The emphasis is on tests that can be performed using standard gemological equipment; reference will be made to tests introduced in the 1980s or new variations on previously known techniques (for more information on the "classic" tests, we recommend that the reader consult Liddicoat, 1989, or Anderson, 1980). However, some discussions will necessarily include techniques that require more advanced instrumentation. We will also consider some enhancements for which there are currently no practical, nondestructive tests. The chart included with this article lists many of the key identifiable treatments currently in the trade and the principal methods available to detect them.

HEAT TREATMENT

Corundum. The 1980s witnessed an extraordinary influx of sapphires and rubies onto the market—over 95% of



Figure 1. Heat treatment brought great numbers of attractive blue and fancy-color sapphires to the gem market. All of the sapphires shown here have been heat enhanced. Courtesy of Gemsource International, Miami, FL; photo © Tino Hammid.

which were believed to have been heat treated by one method or another (Abraham, 1982; figure 1). Nassau (1981) has described no fewer than nine specific treatments used on natural or synthetic corundum.

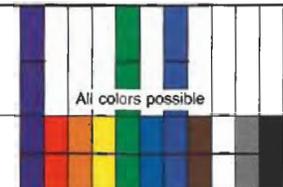
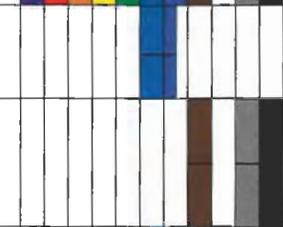
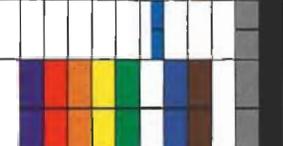
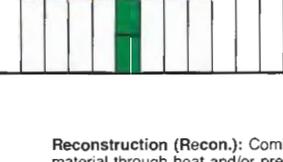
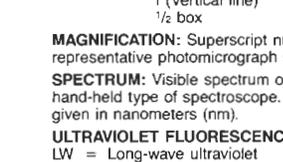
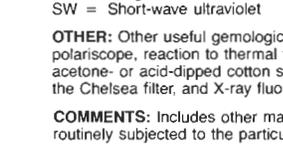
Heat treatment is most widely used to develop blue color in light or near-colorless corundum ("geuda") by means of an intervalence charge transfer involving iron and titanium ions (Moon and Phillips, 1986). Thermal treatment also came into extensive use in the 1980s to induce or enhance yellow to orange to brown colors in corundum. The resulting stones are often of a color that has been described as "rare and somewhat unnatural" (Keller, 1982).

With magnification, the following identifying features may be seen in both blue and yellow (unless otherwise noted) heated sapphires: (1) internal stress fractures (figure 2) or cotton-like

features around inclusions (may have lacy fringes); (2) stubby, partially absorbed (dot-like; figure 3) silk; (3) pockmarked, melted facets, and abnormal multi-plane girdles where part of the original surface was missed in repolishing; (4) blotchy color banding or diffused zoning in blue stones (rarely, with brown color zoning); and (5) colored halos surrounding solid mineral inclusions (Crowningshield, 1980, 1981c, 1982b, 1983b, 1987; Nassau, 1981; Gübelin, 1983; Schmetzer et al., 1983; Scarratt, 1985; Koivula, 1987e; Hurwit, 1987; Koivula and Kammerling, 1988b).

Heat-treated blue sapphires may show a dull, chalky green fluorescence to short-wave U.V. radiation, and may lack both the orange fluorescence to long-wave U.V. and the 450-nm absorption line associated with some natural-color blue sapphires. Heat-treated "golden" sapphires typically show no iron absorption and only weak

Gem Material	Enhancement	Purpose	Color After Enhancement											IDENTIFICATION				Comments						
			C	P	R	O	Y	G	B	V	Br	W	Gr	Bl	Magnification	Spectrum (nm)	Ultraviolet Fluorescence		Other					
AMBER	Heat	Improve clarity, darken color, induce "sun spangles"																	"Sun spangles" discoid fractures.				May also be dyed: look for color concentrations in surface-reaching fractures.	
	Recon.	Produce larger, workable pieces																	"Roiled" structure, flattened elongated gas bubbles, grain boundaries, irregular clear and cloudy areas.	LW: strong, patchy, chalky blue	Polariscope: irregular strain patterns		Also called "pressed" amber.	
BERYL Emerald	Oil	Improve apparent clarity, conceal fractures																	Very low relief of surface-reaching breaks; possibly slight outlining of breaks. Decomposed oils may leave whitish/yellowish dendritic patterns. ¹	LW: chalky yellowish green possible in fractures	Heat may cause oil to "sweat" from fractures. May stain stone papers.		Surface-reaching fractures, pits may be filled with plastic or resin; magnification key to detection. "Hot point" to test emeralds strongly discouraged.	
	Dye	Deepen/induce green color, improve apparent clarity																	Color concentrations in surface-reaching fractures.	630-670 band possible	LW: chalky yellowish green possible in fractures	Oiled stones may have oily odor. Heat may cause oil to "sweat" from fractures. May discolor stone paper, acetone-dipped cotton swab.	Dye concentrations best detected using diffused transmitted light.	
CHALCEDONY	Dye	Alter color																	Rarely, uneven color distribution.	Dyed green: fine lines at 640, 670 Dyed blue: bands at 540, 585, 640		Dyed blue, green: appear red to orangy red through Chelsea color filter.		May be dyed any color. May be selectively or uniformly treated. Treatment often not detectable by routine tests.
CORUNDUM Sapphire Ruby, Sapphire	Heat	Deepen/induce color																	Cotton-like features around inclusions ² ; melted/burst solid inclusions ³ ; ruptured fluid inclusions; "dot-like" silk; diffused color zoning, blotchy color banding (blues); internal color diffusion around solid inclusions; sintered, unpolished surfaces and pockmarked, melted facets.	Blue: very weak or no 450 line Yellow to brown: no iron lines	Blue: LW—lacks orange reaction; SW—dull chalky green, yellowish green Yellow to brown: LW & SW—inert		"Geuda" material usually used to produce blues. Yellow-to-orangy brown stones typically show neither iron absorption of Thai/Australian stones nor strong orangy LWUV reaction of Sri Lankan stones.	
	Heat	Lighten color, remove secondary hues, improve clarity																	Discoid fractures ³ ; melted/burst solid inclusions ³ ; ruptured fluid inclusions; dot-like silk; internal color diffusion around solid inclusions; sintered surfaces.		SW: patchy bluish zones possible in ruby		Treatment typically performed on iron-rich stones like those from Thailand/Australia. Inclusions best seen with darkfield illumination.	
	Diffusion	Induce surface color and/or asterism																	Color diffusion: color concentrations along facet junctions, girdle edge ⁴ ; localization/blotchiness of color; color "bleeding" in pits and surface-reaching fractures. Star diffusion: rutile needles confined to thin surface layer.		SW: chalky yellowish green possible in blue stones; may be patchy	May have unnatural, watery appearance; higher relief of facet junctions, girdle outline in methylene iodide than untreated stones.	Immersion key to detection. Magnification may show other evidence of high-temperature treatment. Repolishing may remove color. Star may be unnaturally sharp.	
	Cavity filling	Improve appearance, add weight, seal voids																	Difference in surface luster, polish, transparency, and/or relative relief (when immersed) between host and filling material ⁵ ; gas bubbles in contact zone.		LW & SW: filling material inert (also to X-rays)	Always reaches surface of stone.	Don't confuse cavity fillings with included crystals/natural glass inclusions breaking surface.	
	Dyeing	Deepen/induce color																	Color concentrations in surface-reaching fractures.					
DIAMOND	Cleavage/fracture filling	Improve apparent clarity																	Filled breaks may exhibit: "flash effects"—orange/blue ⁶ or purple/green; flattened, trapped gas bubbles (may have fingerprint pattern); flow structure; crackled texture; slight yellowish color; no broad-spectrum iridescence or feathery texture.			Treatment may lower apparent color grade; acid, extreme heat may remove filling.	Body color of colored diamonds may mask "flash effect."	
	Laser drilling	Vaporize or bleach dark inclusions																	Laser drill "holes" appear as thin white lines, usually perpendicular to a facet near the inclusion. ⁷			Lasering may be followed by a chemical bleaching of inclusions reached.	Laser drill holes may be filled (see above).	
	Coating	Deepen/induce color																	Dark lines parallel to pavilion main edges; scratches in coating; uneven color.				Repolishing may remove color.	
	Irradiation	Induce/modify color																	Cyclotron-treated stones may exhibit color concentration around culet or outlining facets. ⁸ Possible spotted surface coloration on greens colored by exposure to radioactive salts. Some blue to green electron-irradiated stones show color concentration at culet/keel line. Treated pinks lack characteristic "pink graining" of natural pinks; some show pink and yellow color zoning.	May show 595 line, 496/503 pair Pink may show fluorescent line at 570, absorp. lines at 595, 610, 622, 637	LW: treated pinks fluoresce orange	Some treated stones (typically yellow) show diagnostic features in near-IR; some treated greens may be radioactive; treated blues are electrically nonconductive and have unnatural greenish blue color.	Most of these colors result from annealing after irradiation. Cooling may resolve absorption features; yellows, greens most common colors produced. Origin of color in most green diamonds cannot be determined.	

JADEITE	Dye	Induce or improve color		Dye concentrations sometimes seen in surface-reaching fractures. ⁹	Diffused band at 630-670 nm in green stones	LW: lavender may fluoresce moderate to strong orange	May discolor acetone- and/or acid-dipped cotton swab. Green may appear red through Chelsea filter. Lavender shows bluish purple X-ray fluorescence.	Magnification key to detection. Nephrite, serpentine, quartzite, and other materials may be dyed green; detection similar. Moderate heat may remove lavender color.
	Coating	Improve apparent luster, polish		Scratches in coating; wax or other applied substance concentrated in surface pits, cavities.				Thermal reaction tester will cause wax, paraffin, oil coatings to melt/flow. Best seen under magnification.
LAPIS LAZULI	Dye	Improve color appearance, conceal white calcite		Dye concentrations in surface-reaching fractures and porous areas. ¹⁰		Inert		Chemical tests may be negative if material has been wax coated: wax must be removed to obtain positive test for dye.
OPAL	"Dye"	Produce dark background for play-of-color		Sugar treatment: peppery, speckled appearance, dark color concentrations in cracks. ¹¹ Smoke treatment: dark brown, mottled appearance with "unnatural" play-of-color.			Smoke treatment: used on porous material, so absorbs liquids, temporarily loses color, and gains weight on wetting. Low R.I. (1.38-1.39), S.G.	Smoke, sugar treatments superficial; true body color seen on fracture surfaces. Black plastic impregnation: unnatural transparency with black, wisplike inclusions; unnaturally low S.G. Surface of smoke-treated opals damages easily.
	Irradiation	Modify color		Saltwater cultured: dark bead nucleus but unaltered light-colored nacre may be seen when drill hole is examined.				Colors in irradiated freshwater pearls unlike those produced in nature.
PEARL (Cultured)	Dye	Produce various body colors		Color concentration around drill hole/surface irregularities ¹² ; possibly extending into veins from hole and/or staining stringing thread.		Dyed black: LW & SW usually inert; may be dull green or chalky white	May discolor weak nitric acid-dipped cotton swab rubbed in drill hole. May show unusually even color distribution.	Dyed black with silver nitrate: may show white (X-ray opaque) conchiolin ring on X-radiograph.
	Dye	Alter color to imitate other gems		Dye concentrations in surface-reaching fractures. In quench-crackled and dyed rock crystal, dyed fractures appear as web-like network.	Dyed green: may exhibit absorption band around 630-670		May discolor stone paper, acetone-dipped cotton swab. Dyed green: may appear red through Chelsea color filter.	Dyed green quartzite used to imitate jade.
QUARTZ	Heat	Produce citrine from amethyst		Dehydration alters goethite inclusions to reddish brown hematite with surrounding pressure-induced cracking.				Heat-treated amethyst-citrine, green quartz ("praseolite") may show similar dehydrated iron-based inclusions.
SPODUMENE	Irradiation	Alter color to imitate hiddenite			No chromium absorption		Color unstable, fades when exposed to light and/or heat.	Irradiated stones lack saturated green color of true hiddenite. Artificial irradiation also used to intensify kunzite color; not detectable by standard tests.

PURPOSE OF CHART

This chart covers only major gems for which there are detection criteria available using standard gemological procedures. It does not include those common enhancements (e.g., irradiated and/or heated topaz, tourmaline, zircon, and tanzanite) for which treatment cannot be confirmed by such techniques; nor does it describe in detail all the tests to which it refers. Rather, it is a working guide to be supplemented with additional references such as those listed in "Gemstone Enhancement and Its Detection in the 1980s" (R. C. Kammerling, J. I. Koivula, and R. E. Kane, *Gems & Gemology*, Vol. 26, No. 1, pp. 32-49).

KEY

ENHANCEMENT (Process Used)

Cavity filling: Filling surface-reaching cavities, pits, other depressions with glass, plastic, or other substance.
Cleavage/fracture filling: Filling surface-reaching breaks with glass, plastic, or other substance (see also Oil, below).
Coating: Treating surface with colorless substance, e.g., wax, paraffin, oil, lacquer.
Diffusion: Using high temperature to diffuse color- and/or asterism-causing compounds into a shallow surface layer.
Dye: Introducing colored substance; includes dyes, colored oils, and impregnations.
Heat: Subjecting material to elevated temperatures. Varies with material/treatment. May be as low as 200°C or up to/exceeding 2,000°C.
Irradiation: Using high-energy particles or electromagnetic waves to alter color.
Oil: Filling surface-reaching breaks with oil or other liquid.

Reconstruction (Recon.): Combining pieces of similar material through heat and/or pressure to produce larger pieces. A binder may or may not be used.

GEM MATERIAL: Listed in alphabetical order by species and, within species, by variety where applicable.

COLOR AFTER ENHANCEMENT: Lists color of end product; no reference is made to color of untreated starting material; red includes pink.

Color Range – (Horizontal line) = Common treated color
1 (Vertical line) = Uncommon treated color
1/2 box = Modifying color

MAGNIFICATION: Superscript number refers to representative photomicrograph shown here.

SPECTRUM: Visible spectrum only; as observable with a hand-held type of spectroscope. All band/line locations given in nanometers (nm).

ULTRAVIOLET FLUORESCENCE:

LW = Long-wave ultraviolet
SW = Short-wave ultraviolet

OTHER: Other useful gemological tests, including polariscope, reaction to thermal tester, reaction to acetone- or acid-dipped cotton swab, appearance through the Chelsea filter, and X-ray fluorescence.

COMMENTS: Includes other materials that are also routinely subjected to the particular enhancement.

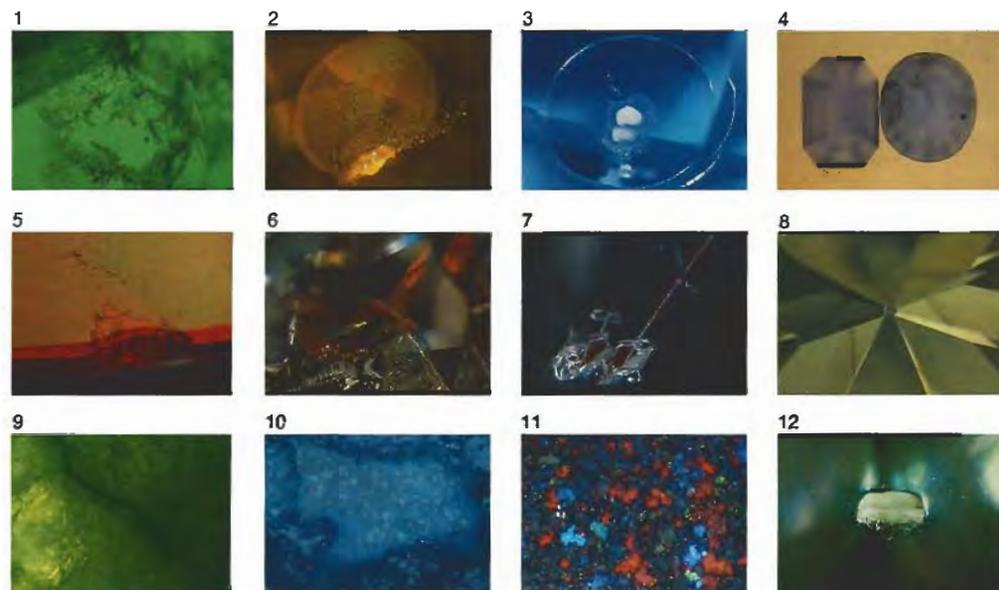




Figure 2. Internal stress fractures, sometimes with lacy fringes, are diagnostic of heat treatment in corundum. Here, they are shown in a "golden" sapphire. Photomicrograph by John I. Koivula; magnified 25 \times .

red-orange or no fluorescence to long-wave U.V. (Crowningshield, 1981c, 1982b, 1984d).

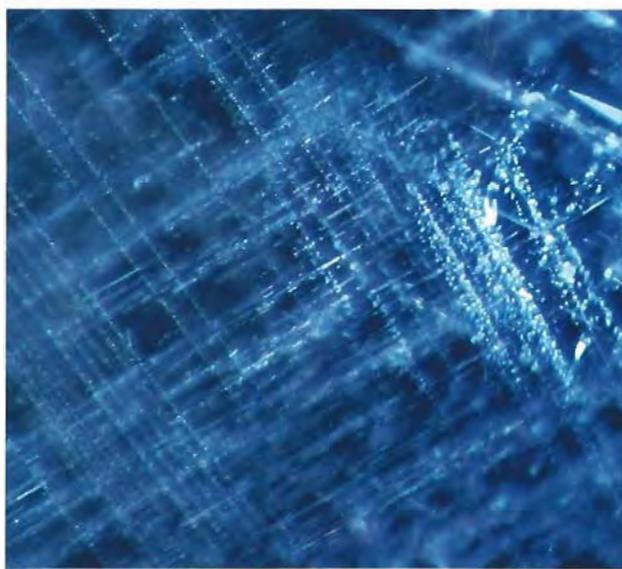
The 1980s also saw other varieties of corundum that apparently owed their color at least in part to heat treatment. These include red-purple and pinkish orange ("padparadscha") sapphires. The presence of strain discs and zones of chalky short-wave U.V. fluorescence helps confirm treatment (Crowningshield, 1984e). When a pinkish orange sapphire was heated to improve the clarity by reducing a dense concentration of tiny (apparently

rutile) particles, the treatment produced an "unnatural-appearing intense orange color" (Kane, 1986a). The stone exhibited a strong, slightly reddish orange fluorescence to both short- and long-wave U.V., as well as chromium-related absorption lines in the red end of the visible spectrum.

Another major application of heat treatment is to partially or totally remove a blue color component from corundum, that is, to lighten the color of dark blue sapphires (commonly those from Australia, as described in Tombs, 1982, and Coldham, 1985) or remove the blue component from purplish stones to produce a purer red ruby (Nassau, 1981; Abraham, 1982; Currie, 1988). Many Sri Lankan pink sapphires also have been heated to drive off any secondary blue overtones (Koivula, 1987c). Identifying characteristics seen with magnification are similar to those listed above for other heat-treated corundum (figure 4). In addition, unusual blue hexagonal color zoning has been observed in blue sapphires heated to reduce the depth of color (Hargett, 1988a; Coldham, 1989). Heat-treated rubies may also exhibit an abnormal reaction to short-wave U.V.: a bluish patchiness and concentric rings (Crowningshield, 1984d).

Often, however, heat treatment to diminish or remove a blue component cannot be detected, as the stones are not heated to the extent that repolishing is necessary. Furthermore, the iron-

Figure 3. The distinctive rutile "silk" in a Sri Lankan sapphire before heat treatment (left) became stubby, partially absorbed, and dot-like (right) after the same stone was submitted to a heating procedure. Photomicrographs by John I. Koivula; magnified 30 \times .



rich blue sapphires that are commonly heat treated to produce a lighter color can display strong iron absorption both before and after treatment (Crowningshield, 1987) and remain inert to U.V.

Research conducted in the 1980s also contributed to the identification of features that would prove that a stone had *not* been heat treated. Specifically, unruptured carbon dioxide fluid inclusions in ruby and sapphire (figure 5) provide conclusive proof that no heat treatment has taken place (Koivula, 1986). Yet the "coarse, well-formed, undisturbed needle-like" or wedge-shaped inclusions once believed to prove that a stone had not been heated (Welch, 1988) have been observed in heat-treated sapphires from Australia, Sri Lanka, and the Kanchanaburi district of Thailand (Coldham, 1989).

One precaution to keep in mind when examining the surface of corundum for heat damage is that similar damage may result when jewelry repair or sizing is performed with the stone still in the mounting. As a final step in such work, the solder joint is often cleaned in a borax-containing hot pickle solution that may produce surface etching (Liddicoat and Fryer, 1980).

Synthetic corundum may also be altered using heat-treatment processes. For example, Chatham flux-grown synthetic dark blue sapphires and dark



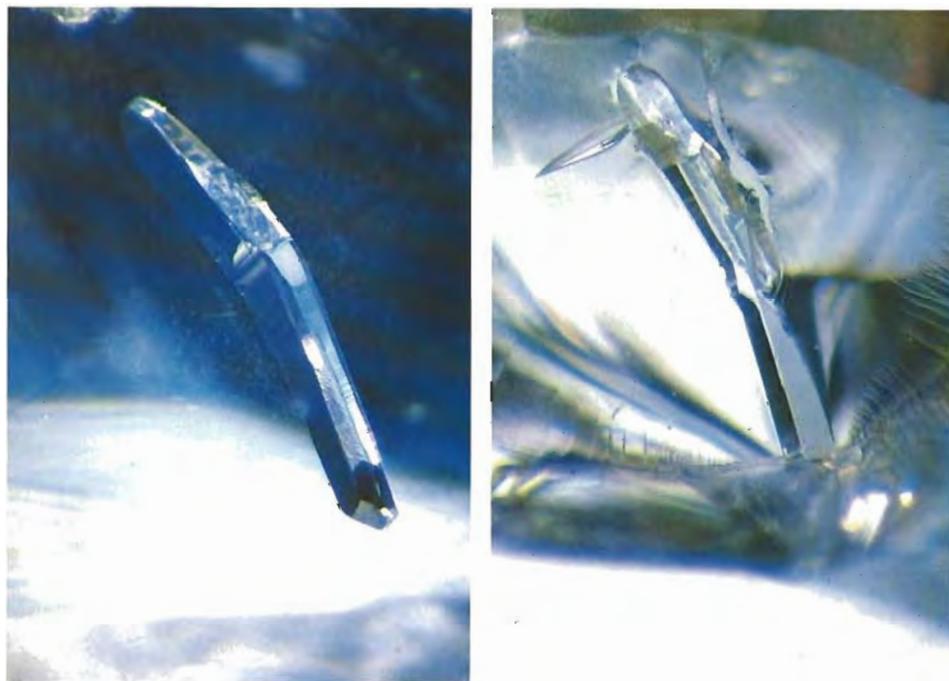
Figure 4. The lacy "fingerprint" surrounding a melted crystal inclusion in this Thai ruby is indicative of heat treatment. Photomicrograph by John I. Koivula; magnified 40 \times .

rubies are reportedly heat treated to lighten the color (Koivula and Kammerling, 1988a).

Quartz. For decades now, most citrine on the market has been produced by heat treating amethyst. While this treatment is generally not detectable, the presence of altered iron-based inclusions may provide proof in some cases. Experimentation has shown that when amethyst containing brownish yellow goethite inclusions is heated, the

Figure 5. The presence of an unruptured CO₂-containing negative crystal (left) in this Sri Lankan sapphire proves that the stone has not been heat treated. When heated to only 270°C, the same inclusion burst (right), producing a large fracture.

Photomicrographs by John I. Koivula; magnified 45 \times .



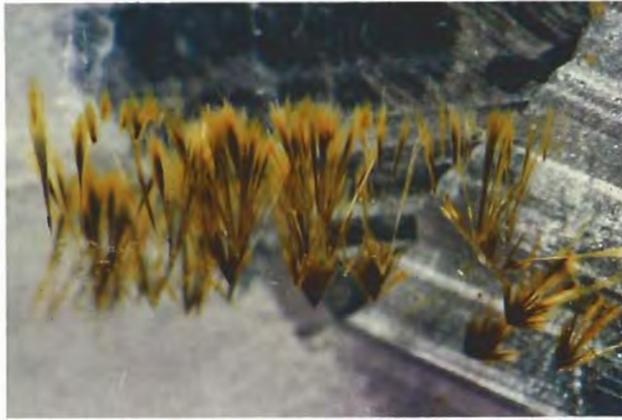


Figure 6. Unaltered yellowish goethite (left) is observed in amethyst that has not been heat treated. Heat treatment to lighten the color of the amethyst or to convert it to citrine (right) dehydrates the goethite, altering it to hematite. Photomicrographs by John I. Koivula; magnified 25 \times .

goethite dehydrates to reddish brown hematite, often with the accompanying formation of stress cracks (figure 6). Therefore, the presence of reddish brown "brush-like" radiating groups of hematite fibers in citrine is a strong indication that the color was produced by heat treating amethyst (Koivula, 1987d).

Zircon. The heat treatment of various natural colors of zircon to produce blue, as well as some "golden," red, and colorless gems, has been practiced for many years (Rupasinghe and Senaratne, 1986). Recently, however, heat treatment has been used to produce cat's-eye stones. While natural chatoyant zircons do occur rarely in nature, heat-treated stones contain diagnostic inclusions in the form of parallel oriented stress fractures around inclusions of thermally decomposed apatite crystals (Hänni and Weibel, 1988).

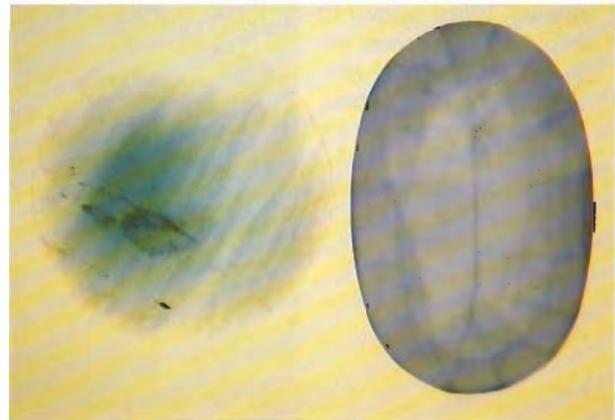
Other Gem Materials. Heat treatment continues to be used extensively to alter the appearance of a number of other gem materials. For example, heat treatment produces: a pink color in chromium-bearing orangy brown topaz, a purer blue hue in greenish blue aquamarine, lighter colors in some dark blue to green tourmaline, and the blue-violet colors of tanzanite from greenish brown zoisite. It was only recently reported (see Koivula and Kammerling, 1990b) that heat treatment was being used to produce unusual, "electric" blue and green colors in tourmaline from the new locality in Paraíba, Brazil. In general, however, these alterations require lower temperatures, so the physical evidence, especially damage or alteration of inclusions associated with heat treatment of corundum,

is generally lacking in these materials (the color produced usually is the only indication of treatment).

DIFFUSION TREATMENT

Another heat-related enhancement of corundum that came to the fore in the 1980s was the diffusion of color-causing agents into the surface layer of an already preformed or fashioned stone. The use of different chromophores produces different colors, including blue, red, yellow, and pinkish orange, although only the blue has been seen in the trade

Figure 7. Immersion in methylene iodide reveals the high relief characteristic of a diffusion-treated sapphire (right), as exemplified by a blue outlining of facet junctions and the girdle edge. The heat-treated blue sapphire (left) does not show any outline facet junctions except near the girdle where some areas are slightly abraded. Photomicrograph by Robert E. Kane; magnified 10 \times .



with any frequency. One of the first identifying features noted in some of these stones was a patchy fluorescence to short-wave U.V. where the diffused color had been removed during repolishing (Crowningshield, 1981b; Hänni, 1982). Diffusion-treated blue sapphires may also show an unnatural "watery" appearance, that is, decreased transparency that is not due to inclusions. Examination of such stones using diffused illumination and immersion in methylene iodide also reveals the areas where color has been removed during repolishing, as well as a higher relief than stones that have not been diffusion treated (as evidenced by a dark outlining of facet junctions) and a blotchiness of color from one facet to another (figure 7). Another characteristic, noted with magnification, is a "bleeding" of color around pits and surface-reaching fractures. Also seen are stress fractures around inclusions, pockmarked facets, and abnormal girdles (Nassau, 1981), although these are a result of the high temperatures used and are not specific to the diffusion process.

Diffusion treatment may be more difficult to detect in cabochons than in their faceted counterparts because cabochons lack the many sharp junctions on which color irregularities can occur. Such treatment is best detected on cabochons at the girdle (i.e., setting) edge (Crowningshield, 1982a).

Diffusion treatment may also be used to create asterism by adding titanium oxide to the surface layer of a stone. Such treatment is identified by: (1) an unnaturally sharp star; (2) uneven color; and (3) bleeding of color around pits, cavities, and fractures—as well as by other evidence of high-temperature treatment as described above. Because the treatment usually affects only a relatively thin surface layer, repolishing can remove the star (Crowningshield, 1985).

Diffusion treatment has also been identified in a synthetic blue sapphire. Immersion in methylene iodide revealed the curved color zoning and tiny gas bubbles typical of Verneuil synthetics, as well as the uneven color from one facet to another that is characteristic of diffusion treatment (Hurwit, 1982).

IRRADIATION

One of the most significant developments during the 1980s was the widespread use of laboratory irradiation to induce or alter color in gemstones. Today, nondestructive detection (i.e., without re-

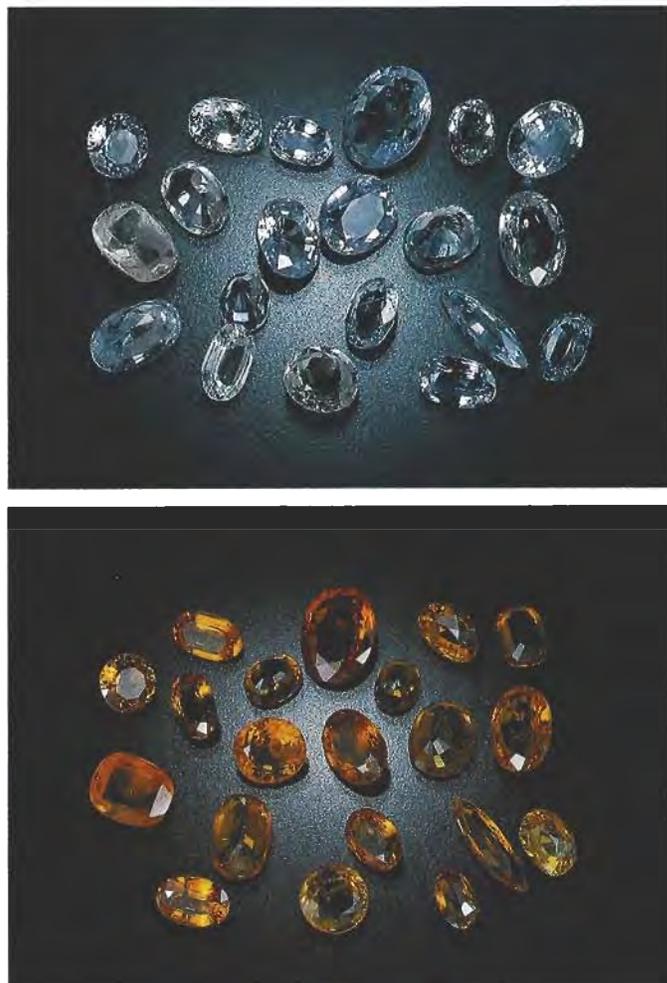


Figure 8. During the 1980s, irradiation was used to change pale-colored sapphire (top) to a golden yellow (bottom). The color, however, is not stable and will fade on exposure to light. Photos by Shane F. McClure.

moving the color or damaging the stone) of such irradiation is one of the gemologist's greatest challenges. Also of concern is the stability of the color obtained to normal conditions of light and heat.

Corundum. Irradiation has been used to produce an unstable golden yellow color in corundum (Rossman, 1981; figure 8). Although some dealers recommended detection by placing the stone in a flame for about one minute or in direct sunlight for several hours, either of which would cause an irradiated sapphire to fade (Keller, 1982), the heating test has since been discouraged. Nassau and



Figure 9. The detection of treatment in diamonds that had been irradiated to produce fancy colors was a major problem during the decade of the '80s. These diamonds, 0.65 to 1.45 ct, have all had their color enhanced by laboratory irradiation. Courtesy of Theodore and Irwin Moed; photo © Tino Hammid.

Valente (1987) recommend only exposure to light, "typically two days of bright sunlight or longer for less intense (natural or artificial) illumination."

Diamond. A major issue of the '80s was the identification of radiation-induced color in diamonds (figure 9). Although detection of some color-treated diamonds has become relatively straightforward, other stones still cause considerable problems.

Cyclotron-treated diamonds, which continued to have a presence on the market during this decade, can still be readily identified by the shallow color confined to a layer near the surface and, in some stones, a concentration of intense color paralleling the edges of facets on the crown or

around the culet or girdle (as illustrated in Kane, 1981a; Scarratt, 1982).

The use of radium or other radioactive salts to produce green color in diamond dates back to the turn of the century. These stones, too, are readily identifiable, because they retain residual radioactivity, which can be detected with a Geiger counter or by the autoradiograph produced when they are placed in contact with photographic film. Historically, these stones have also been known to exhibit mottled dark green stains on facet surfaces. Given the known high radioactivity of diamonds treated by this method, it was surprising to see in the 1980s the release of diamonds color-enhanced by the radioactive salt americium. These stones do not show the mottled, or "mossy," green stains described above (Moses, 1989).

The 1980s witnessed a proliferation of diamonds irradiated by electrons and neutrons, followed by annealing. Many diamonds treated by these methods—producing such colors as yellow, brown, and orange—can only be identified by spectral analysis. Often, a hand-held type of spectroscope in conjunction with an aerosol refrigerant for cooling the diamond is sufficient to identify treatment in these stones; during the 1980s, most major gemological laboratories also had access to a recording spectrophotometer equipped with a liquid-nitrogen cooling unit. In the great majority of diamonds, treatment is evidenced by the presence of a strong line at 595 nm (historically cited by gemological researchers dealing in Angstrom units as the "5920"), accompanied by a pair of bands at 496 and 503 nm (previously known to gemologists as the "4980/5040 pair"). During the 1980s, it was determined that the 595-nm line could be removed by further annealing at much higher ($\geq 1000^{\circ}\text{C}$) temperatures (Collins, 1982). Yet even in these rare cases, two characteristic absorption bands have been observed in the near infrared—at 1936 nm and 2026 nm, designated H1c and H1b, respectively (Woods, 1984; Woods and Collins, 1986; Collins et al., 1986). Unfortunately, the sophisticated instrumentation required to detect these bands is not readily available to the jeweler/gemologist.

Another development of the 1980s was the intensification of color in natural fancy-color diamonds by laboratory irradiation. One natural fancy light yellow diamond examined in the GIA Gem Trade Laboratory had its depth of color increased to the equivalent of a fancy intense

yellow. When the stone was resubmitted to GIA-GTL after treatment, spectroscopic examination revealed both a strong 595-nm line, proving artificial irradiation, and a moderate Cape spectrum (Kane, 1983b).

In rare instances, the treatment process inadvertently produces pink stones (L. Perlman, M. Fuchs, and I. Moed, pers. comm., 1986). Such treated pink stones are readily identified with a hand-held spectroscope by—in all stones—a fluorescent line slightly above 570 nm and a 595-nm absorption line, plus—in most stones—610-, 622-, and 637-nm absorption lines. Treated pink diamonds also typically fluoresce orange to long-wave U.V. radiation. It was first reported in the 1980s that some treated pink diamonds with the above spectral pattern also showed distinctive yellow and pink color zoning, which appeared to be related to a zoned long-wave U.V. (Hargett, 1988b).

Determining origin of color in most green diamonds is one of the greatest challenges in gem identification. Small green to brown radiation stains on the surface of light green diamonds used to be considered a strong indication (but not proof) of natural color, as such stains have not been reported to have been produced artificially. However, the possibility exists that near-colorless and light green or yellow diamonds with these stains may be irradiated to induce or intensify (to light, medium, or dark) a green color. In fact, the authors examined several diamonds with brown radiation stains both before and after treatment. Even though the originally faint green and near-colorless stones developed a dark green color with irradiation, there was no change in the radiation stains.

It was also long believed that color was a good indication of enhancement, with treated green diamonds commonly having an unnatural brownish, grayish, or bluish hue in dark tones. Today, though, the fact that more lighter-toned green stones are seen in the market suggests that color is no longer a reliable indicator (Fritsch et al., 1988).

However, the decade did produce some criteria that can be used to separate treated green diamonds from their natural-color counterparts. Most important was the observation of distinct color zoning in the pavilion or culet of an irradiated diamond. With magnification and diffused lighting, this characteristic is readily visible in some electron-treated blue and green diamonds (Fritsch and Shigley, 1989; figure 10).

Unfortunately, the origin of color still cannot be determined with certainty in the vast majority of green diamonds. Detection of this treatment will be one of the greatest challenges of the 1990s.

Pearl (Cultured). In the 1980s, irradiation was commonly used on off-color saltwater cultured pearls to produce bluish gray colors. Gamma rays darken the freshwater bead nucleus of the relatively thin nacre Akoya cultured pearls to influence the overall body color. Detection requires examining the pearl through the drill hole, where the darker bead nucleus may be seen to contrast with the lighter (unchanged) nacre.

Irradiation of tissue-nucleated cultured freshwater pearls produces a range of colors from blue-gray to black. This treatment is readily identifiable because the colors produced are rarely seen in such pearls naturally.

Quartz. A number of colors have been produced in quartz using artificial irradiation, most notably "smoky" and amethystine. To date, however, we know of no method by which natural-color "smoky" quartz and amethyst can be separated from their artificially irradiated counterparts.

Figure 10. The distinct blue zone at the culet of this bluish green diamond—visible with magnification and diffused lighting—proves that it was treated. Photomicrograph by John I. Koivula.





Figure 11. This 57.3-ct topaz derives its color from irradiation (in a linear electron accelerator) and subsequent heat treatment. It originally resembled one of the white topaz pre-forms on which it sits. Stones courtesy of P. Flusser, Overland Gems, Los Angeles, CA. Photo © Tino Hammid.

Spodumene. Kunzite, the best-known gem variety of spodumene, ranges from light to medium pink to lavender. Hiddenite is the rare, chromium-colored green variety. Laboratory irradiation has been used to intensify the color of kunzite (Ashbaugh, 1988). However, it can also turn kunzite deep green to mimic hiddenite. A simple (but color destructive) fade test will separate chromium-colored hiddenite from the unstable irradiated material, as the latter will commonly bleach out in a matter of one to a few hours in sunlight.

In 1982, Rossman and Qiu reported that neutron irradiation of spodumene had produced an unusual range of colors, from brownish orange through orange to brownish yellow, orange-yellow, and even greenish yellow. This treatment can be identified both by the unnatural colors of the stones and, with a Geiger counter, by their detectable radioactivity.

Topaz. Probably no treated material had as broad a presence in the gem and jewelry market of the 1980s as irradiated topaz (figure 11). Millions of carats of irradiated blue topaz were processed and sold in the course of the decade. Almost any colorless or light yellow topaz can be altered to a range of colors from yellow to dark brown by

gamma, high-energy electron, or neutron irradiation. None of these colors is stable to heat or light (Nassau, 1985). However, heating these stones to approximately 200° to 300°C for as little as one and a half hours will turn some of them blue. While these blue colors appear to be stable, the decade of the '80s produced no definitive gemological test to separate all of the irradiated stones from natural blue topaz.

We do know that the irradiated colors are usually darker and more intense than those seen in nature; no natural-color blue topazes of a medium dark or darker tone have been observed in jewelry-size stones. In addition, some artificially irradiated stones contain residual radioactivity. As early as 1981, Crowningshield (1981a) reported on the presence of radioactive blue topaz in the trade and suggested routinely testing parcels of stones with a Geiger counter. By the end of the decade, a variety of more sophisticated, and more sensitive, radiation detectors were also available (Ashbaugh, 1988). In France, the Centre d'Etudes Nucléaires de Grenoble has developed an instrument that can measure the residual radioactivity of large quantities of gemstones (Robert, 1987).

Research into the application of more advanced technology to identify irradiated topaz continued throughout the 1980s, but with little success. A test based on the property of thermoluminescence, that is, light caused by heat, showed some promise. Unfortunately, the heat required for the measurement bleaches the blue color from the specimen (Rossman, 1981); it is also possible that the effect could be negated by a selective preliminary heating (Nassau, 1985).

Tourmaline. Also during this decade, reports emerged about the artificial irradiation of colorless to light pink tourmaline to produce dark pink to red material (Ashbaugh, 1988). Currently, we know of no method by which this treatment can be detected.

DYEING

Throughout the decade, dyeing of a variety of materials was observed. Perhaps most prevalent was the dyeing of a number of porous minerals, such as calcite, dolomite, and magnesite, for use in beads to imitate more expensive (e.g., turquoise, lapis lazuli) materials (figure 12). Dye in such materials can usually be identified by the use of acetone and, in some cases, a 10% hydrochloric acid solution on a cotton swab. Dyes were also

detected in a number of other gem materials, as described below.

Beryl. Emerald or colorless beryl may be treated with a green oil or dye to enhance or induce a green color. With magnification and diffused transmitted light or immersion, such treatment is detected as color concentrations in fractures (Brown and Snow, 1985; figure 13). However, dark-colored reflections in untreated fractures in emerald should not be confused with dye colors in treated stones. Note that changing the angle of observation should negate any reflection effect, whereas true color concentrations in fractures should be visible at any angle (Ringsrud, 1983).

Jade. Jadeite may be dyed any number of colors, but, not surprisingly, green and lavender are the most common. The detection of dyed green jadeite is relatively straightforward, as discussed in the various identification texts. Detecting dyed lavender jadeite has proved more of a challenge. In a 1982 study, however, specimens believed to be colored with an organic dye fluoresced strong to very strong orange to long-wave U.V. radiation (figure 14); and weak brownish orange to brownish red to short-wave U.V.; had a moderate bluish purple X-ray fluorescence, and showed evidence of color concentrations in microscopic surface cracks. In addition, the color bleached when the specimens were exposed to heat exceeding 220°C for no more than half an hour (Koivula, 1982).

Although much less common, dyed green nephrite has also been seen. This was first reported in "potentially commercial quantity" in 1984. The dyed material shows a broad absorption band in the 660- to 700-nm region and, with magnification, green color concentrations in fine surface-reaching cracks. Under the Chelsea color filter, such dyed nephrite may appear a faint brown (Hurwit, 1984; Crowningshield, 1984c).

Lapis Lazuli. A great deal of lapis, much of it dyed, was seen on the gem market in the 1980s. Although commonly dyed to impart color to whitish (calcite) areas, some lapis is so heavily dyed that it virtually owes its color to the treatment. With magnification, evidence of blue dye may be seen in fractures and some porous areas. Heavily dyed material may show unnatural color in patches not associated with such areas. On beads, dye may concentrate in drill holes. Heavily dyed material

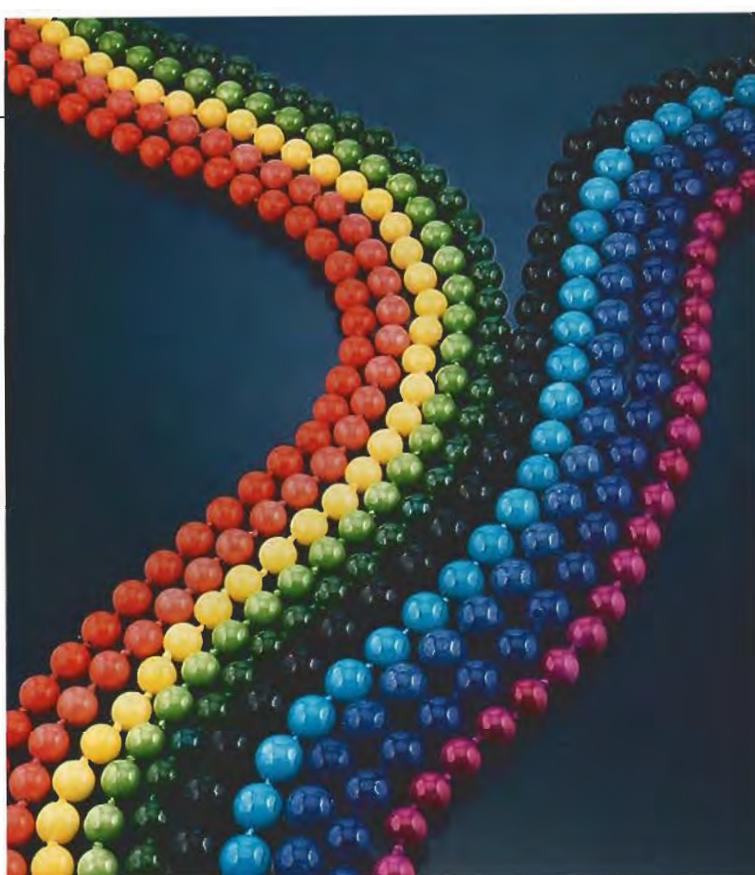


Figure 12. The beads in these strands are composed of a variety of materials, such as dolomite and magnesite, that have been dyed to alter their color. Photo by Shane F. McClure.

may not exhibit the chalky green fluorescence to short-wave U.V. associated with lapis lazuli, and may appear an unnaturally bright brownish red when viewed through the Chelsea color filter (Crowningshield, 1986).

Often the dye can be detected when color appears on an acetone-dipped cotton swab that has

Figure 13. Colored oils, like that shown here in an emerald, may be detected as color concentrations in surface-reaching fractures. Photomicrograph by Robert E. Kane; magnified 25×.





Figure 14. The strong orange fluorescence to long-wave U.V. radiation of this lavender jadeite indicates that the color is a result of dye treatment. Photo by Tino Hammid.

been rubbed on an inconspicuous spot. Some dyes used on lapis lazuli, however, do not yield a positive test to acetone but do produce a blue discoloration when tested with a 10% hydrochloric acid solution (Kane, 1986c) or denatured alcohol (Borgardt, 1986). Some lapis lazuli was treated to conceal the dye from chemical tests and improve apparent luster. With magnification, however, the wax coating may be detectable by its unevenness and the presence of areas of calcite or pyrite where the wax cannot penetrate. After the wax has been removed from a small area with a needle probe, the stone can be retested for dye (Kane, 1981b).

OILING/FRACTURE FILLING

This general treatment category refers to the introduction of any substance into surface-reaching fractures in gems for the primary purpose of lowering the relief of the fractures, thereby making them less noticeable. The substances used may be colorless or slightly tinted. Their primary purpose, however, is to improve apparent clarity and appearance, not to add color, and in some instances they may even have a negative effect on apparent color.

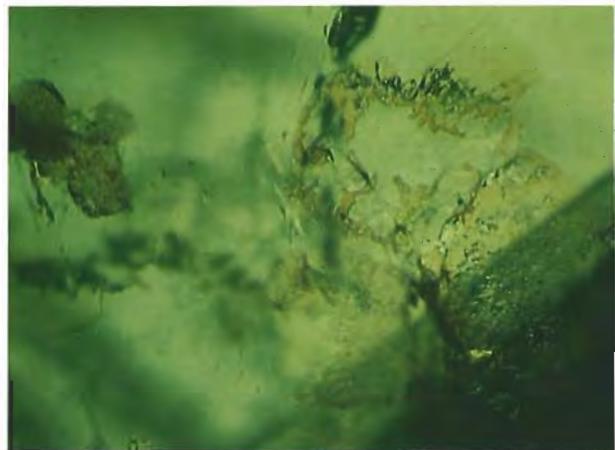
Beryl. To most gemologists, the term *oiling* is automatically associated with emerald. Reports published during this decade have helped fine-tune some of the tests traditionally used to detect oiling in emerald.

The two most commonly used substances for emerald treatment in Colombia are cedarwood oil and Canada balsam (figure 15). The latter fluo-

resces dull yellow to long-wave U.V. radiation, which is used as an indication of oiling. When examined from the side with pinpoint illumination, some oiled emeralds reveal dull areas indicating fracture filling. If the refractive index of the oil differs significantly from that of the stone, careful manipulation of the light source under magnification may produce an iridescent effect. If the oil has not completely filled the fracture, gaps may be seen in the filling material (Ringsrud, 1983). White dendritic deposits may be noted in fractures after oiled stones have been "dried out" by ultrasonic cleaning or exposure to high temperatures (Crowningshield, 1984a).

In the 1980s, reports began to appear of emeralds that had surface-reaching fractures filled with a plastic substance, possibly introduced in liquid form and then hardened by exposure to light or U.V. radiation (Koivula, 1987b). One commercially available prepolymer plastic resin, Opticon, is hardened using a chemical catalyst (Jones, 1986). An early observation relevant to the separation of plastic-filled from oiled emeralds was that oiled stones tend to "sweat" oil when exposed to even slight temperature increases, such as those produced by a microscope lamp, whereas plastic-filled stones would not be expected to "sweat." Such plastic-treated fractures might be detectable by a characteristic fluorescent reaction and diagnostic infrared spectrum (Koivula, 1987b).

Figure 15. The dendritic patterns and yellowish color seen in this surface-reaching fracture are typical of emeralds treated with Canada balsam in which the treatment material has partially decomposed. Photomicrograph by John I. Koivula; magnified 35 \times .



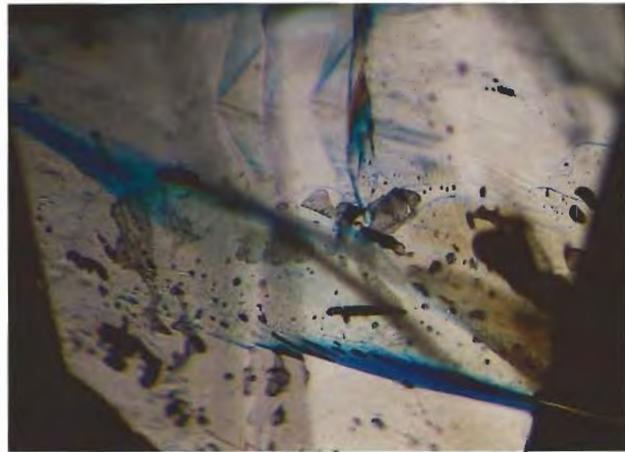


Figure 16. A "flash effect" is one of the most important visual features of filled separations in a diamond. In many fracture-filled diamonds examined to date, this effect is a characteristic yellowish orange in darkfield illumination (left); it changes to a distinctive blue when the stone is rotated slightly to a position where the background becomes bright (right). Photomicrographs by John I. Koivula; magnified 20 \times .

Diamond. One of the most significant enhancements introduced in the '80s is the filling of surface-reaching separations in diamonds (commonly called the Yehuda treatment). This was first reported from Japan in 1987, on diamonds imported from Israel. Later that year, all three locations of the GIA Gem Trade Laboratory had examined stones with treated surface-reaching breaks. One had a subsurface white cruciform pattern where some of the treatment material had been removed when the stone was boiled in concentrated sulfuric acid (Koivula, 1987a).

A comprehensive investigation found that this treatment did improve apparent clarity in many stones, but often at the expense of the overall color (Koivula et al., 1989). These authors subsequently identified a number of diagnostic features. Some treated stones have a slightly greasy or oily appearance with a very slight yellowish overtone. With the microscope, at least one of the following was noted in all of the filled diamonds studied: (1) an interference "flash effect" that appears in most stones as yellowish orange in darkfield illumination but changes to a vivid "electric" blue when the stone is rotated slightly (figure 16; as reported in Koivula and Kammerling, 1990a, some filled diamonds may show a pinkish purple/yellowish green pair of "flash effect" colors); (2) a flow structure of the filling material; (3) flattened gas bubbles trapped in the filling material; (4) a crackled or web-like texture in the filling material; (5) a light

brown to brownish or orangy yellow appearance where the filling-material is unusually thick; and (6) a cloudy white surface residue. In some stones, the filled areas were opaque to X-rays. The treatment material is not stable to extremely high temperatures (Koivula et al., 1989).

CAVITY FILLING

Beryl. Although cavity fillings in corundum were first noted in the mid-1980s (see below), recently an emerald was found to have a cavity filled with a plastic-like substance. The filling material contained gas bubbles, fluoresced a strong bluish white, and was easily indented with a pin (Hurwit, 1989).

Corundum. The last decade saw the introduction of the filling of surface pits and cavities with a glass-like substance to seal open voids, add weight, and improve the overall appearance of rubies and sapphires (Hughes, 1984a, b; Scarratt and Harding, 1984; Kane, 1984; Scarratt et al., 1986). Because of the difference in refractive index and hardness between the host corundum and the filling material (R.I. 1.52, hardness 6 $\frac{1}{2}$), large filled cavities may be detected with the unaided eye by a difference in surface luster (figure 17). The effect is best seen with magnification using surface-reflected light from a fluorescent overhead illuminator. The filling material was found to be inert to both U.V. and X-radiation (Kane, 1984).

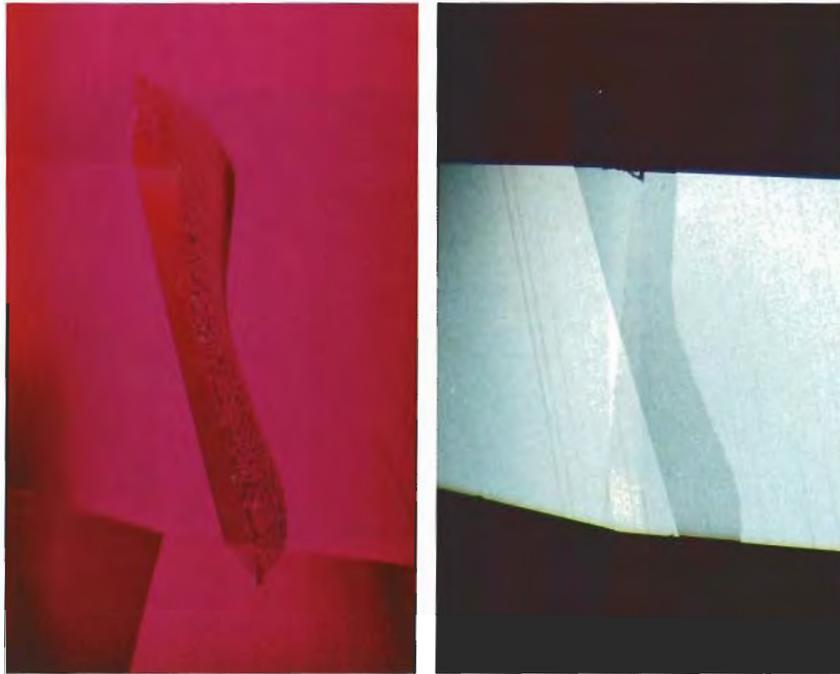


Figure 17. These two photomicrographs of a 2.8×0.4 -mm glass-filled cavity in a 7.01-ct Burmese ruby illustrate how the glass filling appears when different illumination techniques are used. On the left, darkfield illumination shows the difference in transparency between the glass and the ruby, but there is no apparent difference in luster. On the right, reflected illumination from a fiber-optic light source reveals the great contrast in surface luster. Photomicrographs by Robert E. Kane; magnified $60\times$.

Microscopic examination provided several additional clues. With darkfield illumination, a difference in transparency between the glass and the host corundum may be seen. With immersion and diffused transmitted light, the filled areas often stand out in high relief; at some viewing angles the glass may appear colorless. Magnification may also reveal gas bubbles in the filling material. Some filled cavities have uneven voids of trapped gas or a "heat-wave" appearance at the junction of the filling and the host material (Hughes, 1984a, b; Koivula, 1984c; Kane, 1984).

Care must be taken not to mistake included crystals or glassy two-phase inclusions that break a stone's surface for such treated areas. Natural glassy inclusions tend to be no longer than 1 mm and have very irregular shapes (Koivula, 1984c; Kane, 1984).

Opal. Cavity fillings have also been seen in opal. A black opal cabochon examined in the GIA Gem Trade Laboratory had what appeared to be matrix on the base. However, this material was soft, with a waxy luster, and magnification revealed several hemispherical cavities. A scraping of the material flowed easily when a hot point was applied, proving that it was a wax-like substance (Kane, 1981c).

Tourmaline. Historically, shellac has been used to seal the growth tubes in cat's-eye tourmaline. A stone examined in 1984, however, had tubes filled with a plastic. The treatment was detected by the

presence of gas bubbles in the filling and by the odor produced when a hot point was applied (Koivula, 1984b).

COLORLESS IMPREGNATIONS

Colorless impregnations have been used to enhance color and/or improve stability of porous aggregate materials to a greater extent in the 1980s than ever before. Although plastic impregnation of turquoise per se is not new to the 1980s, the treatment of higher-quality material is. Here the purpose is to improve both color and stability. Research has shown that plastic-impregnated turquoise can be identified by X-ray diffraction (which shows the presence of an AlPO_4 phase with berlinite structure) and by infrared spectroscopy (which reveals absorption due to the plastic impregnating material; Lind et al., 1983).

SURFACE COATINGS

A number of gem materials have had their color altered or induced through the use of colored surface coatings. These include faceted natural beryl that has been coated with a green substance to imitate emerald (Kane, 1982); corundum beads that have had their drill holes coated with a red substance (Koivula, 1984a); and faceted quartz with a red plastic coating, white jadeite with a thin layer of dark green plastic, and near-colorless star sapphire with a red plastic coating (Hughes, 1987). Black dye was put in the paraffin used to coat a turquoise carving, and applied selectively to simu-

late matrix (Kane, 1986b); a similar treatment has been used on magnesite dyed blue to imitate spiderweb turquoise (Koivula and Misiorowski, 1986a). Coated diamonds have plagued the gem market for decades, with old stones continuing to reappear in the 1980s. Apparently, coating gemstones continues to be a common practice in India (Tatiwala, 1985).

Such surface coatings are usually detectable with magnification, by the presence of near-colorless areas where the coating has partially worn off (figure 18). Such coatings may also be scratched with a brush probe; they will often leave a discoloration on an acetone-dipped cotton swab (Kane, 1982, 1983a). Note on diamonds that only some surface areas may be coated (e.g., only the pavilion facets) or only a thin band on one or both sides of the girdle (Crowningshield, 1983a, 1984b).

Rough gem crystals, especially beryl, have also been surface coated to alter their color. The coatings are generally detectable using the same methods described above. In addition, the coatings on rough materials may be seen to concentrate in surface fractures, cavities, and depressions (Kane, 1982).

In 1988, a new enhanced gem material known as "Aqua Aura" quartz appeared on the market. Natural colorless quartz crystals (and, more recently, faceted stones) are coated with a very thin, transparent film of gold, so that they exhibit a blue to bluish green color with a superficial iridescence. The coating is durable and does not affect other gemological properties; in addition to the fact that such material does not occur in nature, the coating can be detected by some of the same methods described above (Kammerling and Koivula, 1989a).

INDUCED/ALTERED INCLUSIONS

The 1980s also saw examples of inclusions that were artificially induced or altered. One such process that seemed to resurface during this decade was the use of staining and electrolysis on whitish chalcedony to induce both a blue-green color and dendritic inclusions. The material is identified by the combination of its color, inclusions (Koivula and Misiorowski, 1986b), and the presence of precipitated copper on the surface, extending from the otherwise internal mass (Koivula and Kammerling, 1989).

Heat treatment has also been used to induce "fingerprints" into surface-reaching fractures in Verneuil synthetics (Koivula, 1983). Typical identi-

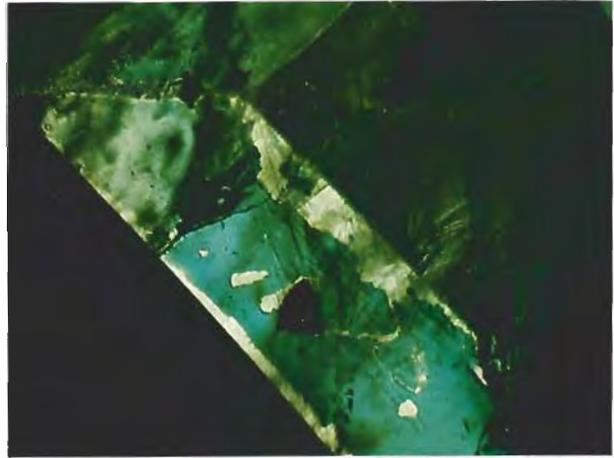


Figure 18. Magnification reveals surface areas of this coated beryl where the green coating has been removed. Photomicrograph © GIA.

fying features of such materials are the simultaneous presence of "fingerprints," curved but diffused Verneuil banding, and occasional gas bubbles (Crowningshield, 1980; Nassau, 1981).

Topaz with limonite-stained etched dislocation channels to make the inclusions darker and more noticeable was heated. In the process, the limonite was altered to hematite. The presence of red-brown hematite-stained etch channels is sufficient evidence that the stone has been heated (Kammerling and Koivula, 1989b).

CONCLUSION

The above discussion focused on a number of the gemstone enhancements that challenged gemologists in the 1980s and continue to challenge them today. What new treatments might face us in the not-too-distant future? A number of recent developments indicate some areas that must be monitored closely.

Plasma-enhanced chemical vapor deposition has been used to produce thin films of polycrystalline synthetic diamond and diamond-like carbon. While such materials appear to be relatively easy to detect, the possibility exists that a transparent layer of colorless monocrystalline synthetic diamond might in the future be grown on various gem materials (Fritsch et al., 1989). One obvious application would be to produce a highly durable surface layer on a relatively soft gem material. Such technology might also be used to produce a layer of synthetic diamond on cubic zirconia in an attempt to "fool" a thermal reaction

tester, or on a near-colorless (or off-color) diamond to alter the apparent body color. Note that, with diffusion treatment, only a thin layer of color is required to drastically alter the appearance of otherwise pale corundum.

Recently it was reported in the trade press that the firm offering commercial fracture filling of diamonds was beginning to offer a fracture filling treatment for emeralds (Everhart, 1989; "Yehuda launches 'permanent' treatment for emeralds," 1989). Although the filling materials are undoubtedly different, a 14+ -ct emerald described in the Gem Trade Lab Notes section of this issue exhibited a number of features visually identical to those seen in filled diamonds: an orange and blue "flash effect," a crackled texture, and trapped bubbles in the filling material. As an extension of this, might we reasonably expect to see a series of new, perhaps more durable, fracture-filling treatments in other gem materials as well?

Artificially irradiated blue topaz has become a

staple of the colored stone market. Yet concern over possible health risks associated with such enhanced gems has caused some to look for alternative methods of producing this now-popular blue color in topaz. In this regard the authors have recently seen faceted topazes that have been treated with the "Aqua Aura" process, that is, coated with an ultra-thin layer of gold, resulting in an attractive greenish blue color with overlying iridescence. Whether or not this enhancement will have any real commercial significance has yet to be seen.

Certainly, detection will continue to face many challenges in the coming decade. Even if no new enhancements were to emerge, the need continues for definitive tests to identify heat-treated rubies and sapphires, artificially irradiated diamonds and tourmaline, and topaz that has been heated or artificially irradiated and annealed. Important strides were made in the 1980s; important strides must be made in the years to come.

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