
GEMSTONE ENHANCEMENT AND DETECTION IN THE 1990s

By Shane F. McClure and Christopher P. Smith

Gemstone enhancements and their disclosure became the most important gemological issue for the jewelry trade in the 1990s. Growing public awareness of treatments and the greater use of sophisticated technology to enhance the color and/or apparent clarity of gem materials brought to the forefront the need to maintain (or in some cases regain) the consumer confidence that is so vital to this industry. The treatments with the greatest impact were those that affected the gems that were commercially most important: heat and diffusion treatment of ruby and sapphire, “oiling” of emeralds, and fracture filling of diamonds. At the end of the decade, the decolorization of diamonds by high pressure and high temperature posed one of the greatest identification challenges ever faced by gemologists worldwide. Yet most other gem materials were also subjected to enhancements—ranging from traditional processes as with quench-crackled quartz to novel “impregnation” techniques such as the Zachery treatment of turquoise. This article discusses the treatments that were new or prominent during the ‘90s and suggests methods for their detection.

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At the time the previous retrospective article on gemstone enhancements was published by Kammerling et al. (1990a), enhancement disclosure was a concern of the jewelry industry, but it was still not a major international focal point. Since then, the issue of disclosure has caused a major upheaval throughout the trade, which has extended to all areas of the jewelry business, including diamonds (figure 1). In some cases, treatment disclosure—or the lack of it—has severely damaged the sale of certain gem materials by eroding the confidence of the consuming public in those gems. When consumers feel—rightly or wrongly—that a product is not being represented honestly, they are likely to stop buying that product.

One of the most drastic of these situations in the 1990s concerned emeralds. As a result of several events during the decade, consumers became aware that emeralds are routinely fracture filled, a fact that retailers typically were not disclosing. This new awareness coupled with the general lack of disclosure caused the public to feel that there must be something wrong with emeralds and they stopped buying them, creating a precipitous drop in the sale and value of these stones (see, e.g., Shigley et al., 2000a).

This is just one example of events throughout the ‘90s that made the subject of treatments—what they involve, how they can be identified, and how they should be disclosed—the most discussed gemological issue of the decade. Many questions about treatment disclosure are still being debated industry wide, and the answers are usually very complex. For this reason, this article will not seek to address the many ethical issues that haunt the trade. Rather, we will describe those gem treatments or enhancements that were first reported on or commonly performed during the last decade, and what can be done to detect them.

It is important to recognize that, for some of these enhancements, the detection methods needed have progressed far beyond the ability of most gemologists working in the trade, primarily because the instrumentation required

is often very sophisticated and expensive. We hope that this article will provide sufficient information to help a gemologist recognize when a stone may have been enhanced by such a method, so that he or she can determine whether it should be sent to a laboratory that has the necessary equipment.

THERMAL ENHANCEMENT

Thermal enhancement, or heat treatment, continues to be the most common type of treatment used for gems. Heat-treated stones are stable, and the result is permanent under normal conditions of wear and care. Heat treatment can be identified in some gem materials by routine gemological testing, and in others only by the use of advanced laboratory instrumentation and techniques. In still other gems, heat treatment is not identifiable by any currently known method. By the 1980s, virtually every gem species and variety known had been heated experimentally to determine if its appearance could be favorably altered. Many of the methods used both then and now are crude by modern standards, yet they can be very effective. During the 1990s, applications of, or improvements in, previously known technologies resulted in new commercial treatments. Perhaps the most important of these is the use of high pressure and high temperature (HPHT) to remove color in some brown diamonds and produce a yellow to yellowish green hue in others. These advances had a significant impact on the jewelry industry, some requiring the investment of enormous amounts of time and money to develop identification criteria.

Many gemstones—such as tanzanite, aquamarine, blue zircon, citrine, and the like—have been subjected to heat treatment routinely for several decades. Not only has the treatment of these stones become the rule rather than the exception, but in most cases there is no way to identify conclusively that the gem has been treated. Therefore, heat treatment of these stones will not be discussed here.

Ruby and Sapphire. As was the case in the preceding decade, the heat treatment of corundum remained a serious issue for the colored stone industry around the world. This treatment was applied to the vast majority of rubies and sapphires (figure 2) during the '90s to: (1) remove or generate color, (2) improve transparency by dissolving rutile inclusions, and/or (3) partially "heal" (i.e., close by the recrystallization of corundum) or fill surface-reaching fractures or fill surface cavities.

The primary concern that surrounded this



Figure 1. Gemstone enhancement and its disclosure became a critical issue in the 1990s, affecting not only rubies (here, 6.38 ct), emeralds (4.81 ct), and sapphires (6.70 ct), but colorless diamonds (5.05 ct) as well. Photo by Shane F. McClure.

enhancement was not the heat treatment itself, but the mostly amorphous substances that were left behind by the heating process in rubies (such features rarely have been encountered in sapphires). The disclosure that such substances were present in fractures and surface depressions caused a great deal of controversy in the industry, which contributed to the significant drop in price of heat-treated rubies in the latter half of the decade (see, e.g., Peretti et al., 1995; Shigley et al., 2000a). Many in the industry felt that this material was only a by-product of the heating process (Robinson, 1995), while others felt that it was put there intentionally (Emmett, 1999). Still others maintained that if fractures were being partially healed by this process, they were being healed with synthetic ruby (Chalain, 1995). Back-scattered electron images showed recrystallized corundum on the surface of one heat-treated ruby (Johnson and McClure, 2000). Although the material within the fractures was typically an artificial glass or similar substance, it was also found that natural inclusions could melt during the heat treatment and leave behind similar residual by-products (see, e.g., Emmett, 1999).



Figure 2. The vast majority of rubies and sapphires are now routinely heat-treated. The color or clarity (or both) can be improved in many different types of corundum by this process. Photo by Shane F. McClure.

This debate was fueled primarily by the discovery of large quantities of ruby near the town of Mong Hsu in the upper Shan State of Myanmar (formerly Burma). Virtually all of this material had to be heated to improve its quality, either by removing the blue “cores” that typically occur down the center of the crystals or by filling or partially healing the many fractures (see, e.g., Peretti et al., 1995). The fluxes used during the heat-treatment process melt, flow into surface-reaching fractures and cavities, and subsequently re-solidify on cooling as an amorphous, vitreous solid (i.e., a glass). Because the fluxes can dissolve solid material in the fractures or even part of the corundum itself, the treatment process also may result in the formation of polycrystalline and/or single-crystal material in the fissure (see, e.g., Emmett, 1999). Currently, researchers and other gemologists are investigating the nature of the materials left behind after the heating process in Mong Hsu ruby. It is important to note, however, that heat-treated rubies from any locality (including Mogok) could

contain these materials. In fact, the filling of surface-reaching pits, cavities, and fractures with “glassy” solids was first identified in ruby from Mogok and various deposits in Thailand during the early 1980s (Kane, 1984).

As the decade began, glassy materials were seen less frequently at the surface of heat-treated rubies, where they appeared as areas of lower surface luster in fractures and cavities. Recognizing that this was the evidence many laboratories used to detect such fillings, heat treaters and others in the trade began to routinely immerse the rubies in hydrofluoric acid to remove the surface material (figure 3). Consequently, gemologists had to focus more on the material that was still present in the fractures within the interior of the stone, which is much more difficult (if not impossible) to remove with acids. Note that the amount of residual glassy material left in partially healed or filled fractures is typically minuscule.

At the beginning of this decade, gemological laboratories had vastly different policies (see below)

concerning the nomenclature used to disclose these substances, which included "glass," "glassy," "glass-like," or "a solid foreign substance." In 1995, the Asian Institute of Gemmological Sciences in Bangkok became the first laboratory to introduce a system to denote the amount of this material that was present in a particular ruby. At the same time, they introduced the term *residue* to denote this substance (Johnson, 1996a). Their system described the presence of residue as *minor*, *moderate*, or *significant*. Most internationally recognized laboratories have since adopted similar terminology.

In fact, during this period, discussions took place in the trade and among laboratories specifically to address these nomenclature issues. Nevertheless, it will be very difficult for all international gemological laboratories to reach a consensus on how to present or describe this form of treatment, because different regions of the world have quite differing views on the subject. In the U.S., the trade demands more open disclosure because of legal concerns (see, e.g., Weldon, 1999a). On the opposite side of the spectrum are Southeast Asia and the Far East, where the trade typically wants little disclosure (see, e.g., Hughes and Galibert, 1998). Between these two is Europe, which traditionally follows the rules and regulations set out by CIBJO (International Confederation of Jewellery, Silverware, Diamonds, Pearls and Stones [see Editions 1991 and 1997]).

All of these factors served to confuse people in the trade and consumers alike. They not only contributed to a dramatic decrease in the price of heated rubies, but they also created greater demand for non-heated rubies and sapphires by the end of the decade.

Besides the continuation of the heating practices described in the previous retrospective article (Kammerling et al., 1990a), there were some significant new developments during the 1990s relating to the heat treatment of sapphire as well as ruby. First, equipment became increasingly more advanced. In

addition to the more sophisticated control of temperature and atmosphere, some electric furnaces were also equipped for elevated pressure (Karl Schmetzer, pers. comm., 2000). Such advanced techniques led to the successful heat treatment of blue sapphires from Mogok, which was previously not commonplace (Kenneth Siu, pers. comm., 1997).

Heat treatment alters many of the properties and internal characteristics of rubies and sapphires. For those laboratories that provide locality-of-origin determinations, such modifications—coupled with the greater number of corundum sources found during the decade—only added to the complexity of determining the geographic origin of a ruby or sapphire (see, e.g., Schwarz et al., 1996). However, a number of articles did address the techniques used and the effects of heat treatment on sapphires from localities such as Kashmir (Schwieger, 1990), Sri Lanka (Ediriweera and Perera, 1991; Pemadasa and Danapala, 1994), Montana (Emmett and Douthit, 1993), Australia (Themelis, 1995), and Mogok (Kyi et al., 1999).

Proving that a stone has *not* been heat treated is often no simple matter, and it may require a significant amount of experience. Little new information was published in the '90s concerning the identification of this treatment in corundum. The criteria of the '80s, most of which require the use of a microscope, still apply. These include spotty coloration, cottonball-like inclusions, broken or altered rutile silk, internal stress fractures around solid inclusions, altered mineral inclusions, and chalky bluish to greenish white fluorescence to short-wave ultraviolet radiation (see, e.g., Kammerling et al., 1990a). It has been suggested that enhancement can be effected in some rubies and sapphires by heating them at lower temperatures, which might not produce the evidence normally seen in heat-treated corundum (John Emmett, pers. comm., 2000). This would make identification of the treatment even more difficult.

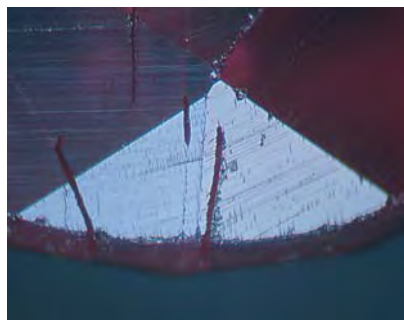
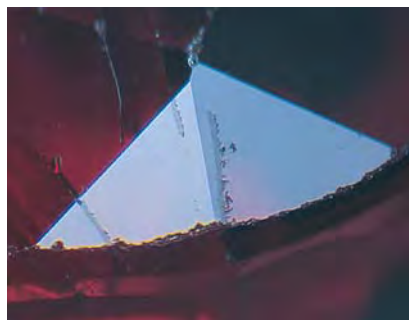


Figure 3. Rubies that have been fracture filled with a glassy substance can be detected by the lower luster in reflected light of the glassy material within the fractures (left). In the mid-'90s it became common for treaters or dealers to immerse these stones in hydrofluoric acid to remove this surface evidence (right). Photomicrographs by Shane F. McClure; magnified 40 \times .

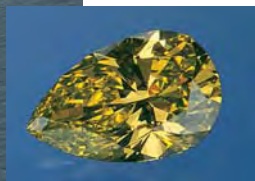


Figure 4. The color enhancement of diamond moved to the forefront late in the decade, when it was learned that new high pressure/high temperature techniques had been developed that could turn brown type IIa diamonds colorless and turn brown type Ia diamonds, similar to the rough diamonds shown here, yellow-green (inset, 4.45 ct). Photo by Shane F. McClure; inset photo by Maha Tannous.

Diamond. The close of the decade witnessed a dramatic new development in thermal enhancement. Beginning in approximately 1996, intense yellow to greenish yellow to yellowish green type Ia diamonds began to enter the international diamond market (see, e.g., Reinitz and Moses, 1997b). Soon thereafter it became known that the color in these diamonds, which were primarily thought to have originated in

Russia, had been produced in type Ia diamonds by high pressure/high temperature annealing techniques (figure 4).

On March 1, 1999, Lazare Kaplan International subsidiary Pegasus Overseas Limited announced that they planned to market diamonds that the General Electric Company (GE) had enhanced by a proprietary new process (Rapnet, 1999). GE scientists soon confirmed that they were using HPHT annealing to *remove* color from type IIa diamonds (figure 5; see box A of Moses et al., 1999). This development sent a shockwave throughout the international diamond industry (see, e.g., Barnard, 1999; Weldon, 1999b,c). Gemological and research laboratories around the world soon began the task of developing a means to detect these HPHT-enhanced diamonds (see, e.g., Moses et al., 1999; Chalain et al., 1999, 2000). Currently, several characteristics have been identified that may indicate if a diamond has been exposed to HPHT conditions. Unfortunately, most are not within the scope of techniques available to the average gemologist, because they depend heavily on absorption and/or photoluminescence spectral features present at low temperatures (see, e.g., Fisher and Spits, 2000; Reinitz et al., 2000b; Smith et al., 2000). Nevertheless, the process may produce some indications that are visible with a microscope. These relate primarily to damage caused by the extreme conditions of the treatment, such as etched or frosted naturals, or fractures that are partially frosted or graphitized where they come to the surface (figure 6). It must be emphasized that these are indications only, and they may be difficult to recognize for all but the most experienced observers.

By the end of the decade, a number of different groups in various countries were modifying the color of diamonds by exposure to HPHT conditions (see, e.g., Moses and Reinitz, 1999). The majority of these stones are the yellow to yellowish green type Ia diamonds, but more than 2,000 “decolorized” type IIa diamonds had been seen in the GIA Gem



Figure 5. This 0.84 ct piece of type IIa diamond rough was HPHT annealed by General Electric for GIA researchers. The original dark brown material (left) was changed to approximately “G” color (right) after being subjected to the process. Photos by Elizabeth Schrader.

Trade Laboratory by the end of 2000. Most recently, HPHT-processed pink type IIa diamonds and even a limited number of blue type IIb diamonds have appeared (Hall and Moses, 2000).

Tourmaline. The 1989 discovery of elbaite tourmaline in Brazil's Paraíba State revealed colors that had never before been seen in this gem species. It was soon determined that exposure to high temperatures could produce a vivid blue or green hue in some crystals from this deposit; the "emerald" green was not known to occur naturally (Fritsch et al., 1990). The heat treatment of these tourmalines (commonly referred to as "Paraíba" tourmaline) continued throughout the 1990s, even though finds of this material declined as the decade progressed. Other types of tourmaline from various countries also continued to be heat treated during the 1990s. However, as with the Paraíba material, such treatment cannot be identified in these tourmalines by standard gemological methods.

Topaz. Pink topaz continues to be produced by exposing brownish yellow to orange "Imperial" topaz from Brazil to elevated temperatures (figure 7). This color does occur naturally in topaz from a number of localities, including Brazil.

The most recent report on the mining and heat treatment of Imperial topaz was done by Sauer et al. (1996). This article described a possible new test for detecting heat treatment in topaz. The limited number of heated stones in this study showed a distinct change in short-wave UV fluorescence from a very weak to moderate chalky yellow-green in the untreated stones to a generally stronger yellowish or greenish white in the treated stones. As the authors noted, more research is needed to determine the reliability of this test.

Zoisite. Most people in the trade are now familiar with the fact that the color of the vast majority of tanzanite in the market is the result of the heat treatment of brown zoisite. In 1991, however, transparent green zoisite was discovered. Although the finds to date have been relatively small and sporadic at best, limited experimentation showed that only a small percentage of this material responded to heat treatment, changing from the original bluish green through brownish green to a greenish blue. Barot and Boehm (1992) suggested that green zoisite was not routinely being heated because of the rarity of this material.



Figure 6. This fracture in an HPHT-annealed yellow-green diamond has partially graphitized, which is an indication that the stone has been subjected to high pressure/high temperature conditions. However, the presence of graphitization should not be construed as proof of treatment. Photomicrograph by Shane F. McClure; magnified 31 \times .

As is the case with many other materials, at this time heat treatment in zoisite is not detectable in most cases.

Amber. Several reports in the '90s described a kind of surface-enhanced amber, where a dark brown layer of color is generated at a shallow depth by exposing the amber to controlled heating, up to

Figure 7. Heat treatment of brownish yellow to orange Imperial topaz from Brazil changes the color of the material to pink, such as the piece shown here on the lower left. The larger crystal weighs 115.0 ct. Photo by Maha Tannous.





Figure 8. This heat-treated amber bead has been ground down on opposite sides to show that the color imparted by the treatment is confined to a thin surface layer. The dark brown hue fades on prolonged exposure to light. Photomicrograph by Shane F. McClure; magnified 10 \times .

approximately 220°C (Crowningshield, 1993; Hutchins and Brown, 1996; Safar and Sturman, 1998). In many cases, the interior of this material is left almost colorless (figure 8). With prolonged exposure to light, however, the dark surface layer proved to be unstable, fading to a much lighter tone.

This treated amber can be recognized by a dull, chalky green fluorescence to long-wave UV, rather than the stronger orange fluorescence of untreated material, as well as by the presence of numerous tiny gas bubbles in swirling clouds just below the surface of the stone.

Other Gem Materials. It seems that people in our trade have a fascination with exposing gemstones to heat, just to see what happens. Some examples of this reported during the past decade include changing the color of blue benitoite to orange (Laurs et al., 1997), yellow chalcedony to carnelian (Brown et al., 1991), and rhodolite garnet to a more brownish color with a metallic oxide coating (Johnson and Koivula, 1997a).

DIFFUSION TREATMENT

Corundum. At the beginning of this decade, the trade witnessed a dramatic resurgence in diffusion-treated blue sapphire (e.g., Kane et al., 1990; Hargett, 1991). This resurgence was attributed to a new technique that allowed for a much deeper penetration of the diffused color, which came to be known as “deep diffusion” in the trade. For a time, these stones seemed to have a certain degree of trade acceptance,

and large diffusion-treated sapphires—some exceeding 20 ct—were produced (e.g., Koivula and Kammerling, 1991b). By the mid-’90s, however, interest in this material had declined dramatically (Koivula et al., 1994). We believe that, for the most part, these stones were being marketed and disclosed properly, although there were several incidents of diffusion-treated sapphires being “salted” in parcels of natural-color blue sapphires (Brown and Beattie, 1991; Koivula et al., 1992d).

Identification of this material is best accomplished by immersing it in methylene iodide. Diffusion treatment in sapphires is characterized by color concentrations along facet junctions, patchy surface coloration, and higher relief in immersion when compared to an untreated stone (Kane et al., 1990). It was recently reported that some diffused sapphires do not show the characteristic concentrations along facet junctions, which are caused by the stones being repolished after treatment (Emmett, 1999). This was attributed to the possible use of a molten titanium-bearing flux instead of a powder, which could eliminate the need for repolishing. Such stones can still be identified by the “bleeding” of color into surface-reaching features such as “fingerprints,” fractures, and cavities, or by their characteristic higher relief in immersion.

The most significant new development in diffusion treatment during the decade was the introduction of red diffusion-treated sapphire (often called diffusion-treated ruby), as described by McClure et al. (1993). This type of diffusion treatment never seemed to gain wide usage, probably because of the difficulties inherent in diffusing chromium into the surface of corundum. These difficulties result in a very shallow surface layer of color, as well as in some unwanted colors such as purple and orange (Koivula and Kammerling, 1991f,g; McClure et al., 1993; Hurwit, 1998). One of the authors (CPS) was informed that when this material first came out, several prominent ruby dealers in Bangkok paid very high prices for diffusion-treated “rubies” that were represented as heated only.

Diffusion-treated “rubies” can be identified readily by their patchy or uneven surface coloration, color concentrations along facet junctions, relatively high relief in immersion (figure 9), very high surface concentrations of chromium, very high refractive index, patchy bluish white to yellowish white short-wave UV fluorescence, and atypical dichroism (see, e.g., McClure et al., 1993).

Figure 9. Immersion in methylene iodide reveals the patchy surface coloration, color concentrations along facet junctions, and high relief (when compared to untreated stones) of these red diffusion-treated sapphires. Photo by Shane F. McClure.



Occasionally encountered were corundums that owed their asterism, as well as their coloration, to diffusion treatment (e.g., Crowningshield, 1991, 1995c; Johnson and Koivula, 1996c, 1997c). Even colorless synthetic corundum was diffusion treated (Koivula et al., 1994; Crowningshield, 1995b; Johnson and Koivula, 1998a).

Topaz. We first encountered what was being represented as “diffusion treated” topaz in 1997 (Johnson and Koivula, 1998d). However, it is still not clear if the cobalt-rich powders employed during the enhancement process actually diffuse into the lattice of the topaz. Nevertheless, the green-to-blue colors of this material (figure 10) are quite different from the orange, pink, or red hues we have seen in topaz colored by a surface coating (Johnson and Koivula, 1998d; Hodgkinson, 1998; Underwood and Hughes, 1999). The “diffusion treated” material is easily identified by its spotty surface coloration. The colored layer is as hard as topaz and is so thin that no depth was visible in a prepared cross-section, even at 210× magnification (Johnson and Koivula, 1998d).

IRRADIATION

In the 1980s, experimental and commercial irradiation played a significant role in the arena of gemstone treatment (Kammerling et al., 1990a). During the following decade, however, the role of irradiation diminished considerably when compared to other forms of enhancement. In the 1990s, very few new

types of artificially irradiated gems appeared on the market, although there were a number of changes or improvements made to the methods used with some already well-known irradiated gems, such as blue topaz (Fournier, 1988; Skold et al., 1995). During this decade, gemologists and gem laboratories continued to see irradiated gem materials, but very little of what they saw was actually new.

Figure 10. “Diffusion treated” green-to-blue topaz (here, 4.50–5.86 ct) was first seen in the late 1990s. While it has not yet been proved adequately that the color is actually diffused into these stones, the extremely shallow color layer is as hard as topaz. Photo by Maha Tannous.





Figure 11. The red color in this 0.55 ct synthetic diamond was produced by irradiation and subsequent annealing. Photo by Robert Weldon.

Likewise, there was little progress in detection methods. For many gems, no test or series of tests, destructive or nondestructive, is currently available to establish whether they have been subjected to irradiation. Unless the technique used produces a visually distinctive pattern in a treated stone, such as the “umbrella effect” seen around the culet of a cyclotron-treated diamond, the use of irradiation to improve a gemstone’s color still can be difficult or impossible to detect gemologically. For example, although treaters have used irradiation to produce intense pink-to-red colors in near-colorless to light pink tourmaline for many years, this well-known form of color enhancement is still not detectable. This is also the case for blue topaz, as well as for many other gem materials that are routinely irradiated.

Yet another factor to consider in the detection of any suspected means of treatment, including irradiation, is economics. While it may be economically feasible and even imperative to attempt to detect irradiation-induced color enhancement in a fashioned green diamond, the same is usually not the case with respect to smoky quartz or blue topaz. The low value of the starting materials, and the limited potential gain in value of those materials after color enhancement, does not warrant a significant expenditure in laboratory time to attempt to detect the treatment.

Radioactivity is a word that stirs particularly strong emotions in the public at large. This is primarily due to a widespread lack of understanding concerning the various forms of irradiation and their short-lived or long-lasting effects. If there is anything that generates press quickly, it’s the detection of residual radioactivity in an irradiated gem and the potential threat to health it suggests. Diamond, ruby, chrysoberyl, spodumene, and topaz are a few of the gem materials that have shown residual radioactivity after color enhancement by irradiation.

Diamond. Irradiation to improve or induce color in diamond is generally performed on faceted stones, because usually the need for color improvement can be determined accurately only after a stone has been cut. However, rough diamonds are also occasionally irradiated. A 13.12 ct treated-color yellow rough diamond was reported late in the decade (Reinitz, 1999). Treatment of rough is a highly questionable practice, since such material is often misrepresented. The fact that some treatment methods produce only a shallow layer of color that can be removed easily on faceting strongly suggests that the treatment is only done to deceive.

Unfortunately, most radiation-induced color patterns in faceted diamonds, such as those produced by electron bombardment, are subtle and difficult to detect. Careful inspection with a gemological microscope, however, may show color zoning that is directly related to the facet shape of the diamond (Fritsch and Shigley, 1989). Artificially irradiated diamonds that show subtle but diagnostic forms of color zoning in blue to green (Hargett, 1990; Hurwit, 1993; Moses and Gelb, 1998) and reddish purple (Reinitz and Moses, 1998) were encountered regularly throughout the 1990s. Diffuse transmitted light is useful in the detection of treatment in these stones if the light can be directed through the diamond. To facilitate light transmission, total or partial immersion of the diamond in methylene iodide is often helpful.

A number of treated pink to purplish pink diamonds encountered in the ‘90s (Crowningshield and Reinitz, 1995; King et al., 1996) did not show color zoning that could be related to irradiation. In such cases, both the diamond’s reaction to UV radiation (bright, chalky orange to both long- and short-wave) and its spectrum (sharp absorption lines at 595, 617, and 658 nm) are distinctive of treatment. Although irradiation-produced pink in diamonds was previously rare and usually accidental

(Kammerling et al., 1990a), significant quantities of laboratory-irradiated pink diamonds (typically melee-size) appeared on the market in the latter half of the decade.

Also encountered in the 1990s were diamonds irradiated to such a dark green that they appeared essentially opaque and black in all jewelry applications. These diamonds are identified by the fact that they are dark green instead of the dark gray of natural black diamonds, which is caused by inclusions (Kammerling et al., 1990b). Some of these “black” stones are treated in a nuclear reactor, which can result in residual radioactivity. One such treated diamond examined in the GIA laboratory was sufficiently radioactive to render it unlawful to sell for almost 37 years (Reinitz and Ashbaugh, 1992). Another report on “black” irradiated diamonds stated that the residual radioactivity was related to metallic polishing residues in surface-reaching cracks that became radioactive when the stones were irradiated. Prolonged boiling in acid removed the radioactive residues and rendered these treated diamonds safe (Koivula et al., 1992h).

Irradiation and annealing also can change synthetic diamonds from yellow and brownish yellow to red (figure 11—Moses et al., 1993; Kammerling and McClure, 1995c). These treated synthetic stones do not present significant identification problems because they have distinctive spectra (the same as for treated pink diamonds mentioned above) and all the internal characteristics expected of synthetic diamonds. The short-wave UV fluorescence is particularly distinctive, as these treated-color red synthetic diamonds almost always show a bright green “cross” in the middle of the table with orange throughout the rest of the stone (figure 12; Moses et al., 1993).

Ruby. Radioactive rubies were new to the gem trade in the 1990s. These stones first appeared on the market in Jakarta, Indonesia, and were reported in the trade press in mid-1998 (“Indonesia: Irradiated ruby...,” 1998). Two of these stones were examined by Ken Scarratt at the AGTA Gemological Testing Center, who subsequently loaned them to GIA for photography and further study (Johnson and Koivula, 1998c). The slightly brownish red stones closely resembled rubies from East Africa. Both showed clear evidence of heat treatment and were partially coated with a black crust of unknown origin that appeared dark brown along thin edges.

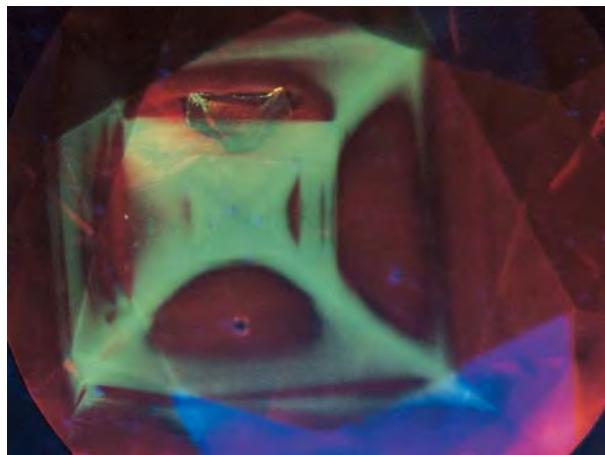


Figure 12. A characteristic property of irradiated red synthetic diamonds is their short-wave UV fluorescence, which typically shows a strong green “cross” in the center of the stone surrounded by weak orange. Photomicrograph by John I. Koivula; magnified 15 \times .

The isotopes responsible for the residual radioactivity in these stones were not determined, so we do not know just how long the stones would remain radioactive. To date, no information has become available as to the precise source of these rubies and their original starting color.

These radioactive rubies cannot be recognized by any standard gemological means. The only indications are their brownish color and the black crust. However, these indications are unreliable. Fortunately, we know of no further reports of these stones in the marketplace.

Chrysoberyl. Yet another form of radioactive gem material appeared in the 1990s. Hundreds of carats of cat’s-eye chrysoberyl of an unusual dark brown color were sold at gem markets around the world. These cat’s-eyes showed a dangerous level of radioactivity—50 times greater than that which is legally acceptable in the United States—and were thought to have been treated in a nuclear facility in Indonesia (perhaps the same source as for the radioactive rubies described above). The original starting material is believed to have come from Orissa, India (Weldon, 1998b). All dark brown cat’s-eye chrysoberyls are suspect until they are tested for radioactivity by a properly equipped gemological laboratory.



Figure 13. These two “Ocean Green” irradiated topazes (3.00 and 3.13 ct) were originally the same color, but after being taped to a south-facing window for one day, the stone on the left lost almost all of its green component. Photo by Maha Tannous.

Topaz. Large amounts of irradiated blue topaz continued to be seen in the international gem market. Irradiated green topaz with unstable color (figure 13) was reported (see, e.g., Koivula et al., 1992f; Ashbaugh and Shigley, 1993). It was marketed under the trade name Ocean Green Topaz. Because the color is produced by irradiation in a nuclear reactor, like other reactor-treated gems this green topaz has the potential to be radioactive. The color ranges from light to medium tones of yellowish and brownish green through a more saturated green to blue-green. On exposure to one day (or less) of sunlight, the green component fades, leaving a typical blue topaz color. The relative tone and saturation remain the same.

The original starting material is said to have come from Sri Lanka. Green topaz has been reported to occur in nature, but it is very rare. With this in mind, any green topaz should be suspected of some kind of treatment.

Quartz. Pale gray cat’s-eye quartz was being irradiated to a dark brown to enhance the appearance of the chatoyancy by having the bright, reflective, inclusion-caused “eye” appear against a dark background (Koivula et al., 1993a). Also reported was the gamma irradiation (followed by heat treatment) of colorless quartz to produce colors ranging from green through yellow and orange to brown (Pinheiro et al., 1999). All of the colors were stable to light.

No tests are presently available to detect the treatment in these stones.

Beryl. Maxixe beryl, the dark blue beryl that owes its color to natural or (usually) artificial irradiation, appeared again in the 1990s, in at least one instance as a substitute for tanzanite (Reinitz and Moses, 1997a). Another report reviewed its susceptibility to fading (in most cases, dark blue is an unstable color in beryl) and the gemological properties used to recognize this type of beryl (Wentzell and Reinitz, 1998).

DYEING

Although dyeing is one of the oldest treatments known, the 1990s witnessed a number of apparently new variations on beryl, corundum, jade, and opal, among other gem materials. Especially convincing were dyed quartz and quartzite imitations of gems such as amethyst and jadeite. At the same time, the proliferation of inexpensive cultured pearls brought with it a multitude of colors produced by dyes.

Beryl. In addition to the standard dyeing techniques used to enhance pale green beryls to an emerald color or colorless beryl to aquamarine (e.g., quench crackling, or drill holes coated with dye; Koivula et al., 1992b), the market saw the continued use of green oils and the introduction of green Opticon as fracture fillers (Koivula and Kammerling, 1991a). Using a combination of heat (to increase porosity and thus color penetration) and dye, Dominique Robert of Switzerland turned massive beryl with intergrown quartz into imitations of ornamental materials such as charoite and sugilite, as well as turquoise and coral (Koivula et al., 1992e). As is the case with most dyed stones, the treatment was readily identifiable by the presence of dye concentrations in the fractures.

Corundum. Although the red staining of quartz that has been heated and quenched (“crackled”) to induce fissures dates back hundreds of years, for the first time gemologists identified corundum in which fractures had been induced and the pale sapphires then dyed a purplish red. These stones were recognized by the irregular color distribution and the presence of a yellow fluorescence confined to the stained fractures; they also lacked the red fluorescence and Cr lines in the spectroscope that are characteristic of ruby (Schmetzer et al., 1992). A similar process was also seen in dyed natural star corundum (Schmetzer and Schupp, 1994). Dyed red beads examined in the

GIA Gem Trade Laboratory responded to a simple acetone test; removal of the dye from one bead revealed that it originally was a pale green sapphire (Crowningshield and Reinitz, 1992).

Jadeite. Colored substances have been used to fill cavities in bleached and impregnated jadeite (Johnson and McClure, 1997b). These fillers are readily visible with a microscope.

Of particular concern was the identification of dye in a green jadeite bangle that did not show the typical dye band with the handheld spectroscope (Johnson et al., 1997). This piece first aroused suspicion when the expected absorption bands for chromium were not seen in the spectroscope. The bangle was of sufficient color that these bands should have been present if the color was natural, so the piece was examined very carefully with a microscope. Fortunately, in this case the dye was evident as color concentrations along grain boundaries (figure 14).

Opal. Because of its porosity, opal has long been subjected to enhancements such as the "sugar" treatment commonly used on Andamooka material to darken the background so the play-of-color is more prominent (see, e.g., Brown, 1991). During the 1990s, however, we also saw opal darkened by silver nitrate (similar to the treatment used to produce black in pearls). As with the sugar-treated material, the silver nitrate treatment is evidenced by the presence of dark irregular specks (Koivula et al., 1992i). In still another process, opal-cemented sandstone is soaked in an organic solution and then carbonized at temperatures over 500°C to produce an attractive carving material (Keeling and Townsend, 1996). Particularly interesting was the introduction of dark blue enhanced opal, produced by soaking a highly porous chalky white hydrophane opal in a mixture of potassium ferrocyanide and ferric sulfate (Koivula et al., 1992c). This material looks black to the unaided eye, but strong transmitted light reveals its unnatural dark blue body color.

Pearls. Numerous examples of black cultured saltwater pearls that had been dyed with a silver nitrate solution (figure 15) were seen during the '90s, including some mixed with natural black pearls in a fine necklace (DelRe, 1991). The treated pearls were first spotted by the lower contrast on the X-ray film between the shell bead and the nacre; their chalky green appearance to long-wave UV confirmed that

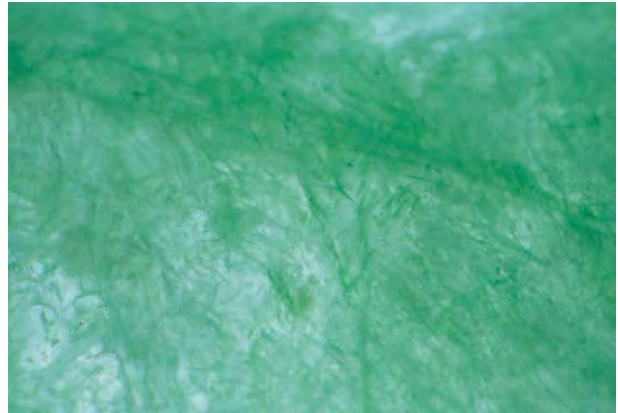


Figure 14. Careful microscopic examination revealed dye concentrations in this piece of jadeite, which did not show the dye spectrum typical of this type of material. Photomicrograph by Shane F. McClure; magnified 34 \times .

they were dyed. Other indications of silver nitrate staining include damage to the nacre layers or, occasionally, a dimpled surface (Moses, 1994). Of particular concern toward the end of the decade was the prevalence of dyed "golden" South Sea cultured pearls. Unfortunately, the natural or treated origin of these pearls often cannot be determined ("Pearl treatments..." 1998). Whereas the colors of dyed saltwater pearls are usually fairly limited (black, brown, dark green, and "golden"), freshwater cultured pearls have been dyed in a wide array of hues, including "silver," "bronze," and bright "pistachio"

Figure 15. This cultured pearl was turned black with a metallic oxide, most likely by the use of a silver nitrate solution. Photo by Jennifer Vaccaro.





Figure 16. Quartzite dyed green to imitate jadeite, as illustrated by these 8 mm beads, was commonly seen in the 1990s. Photo by Maha Tannous.

green (Johnson and Koivula, 1999). In many cases these dyed pearls can be identified by their unnatural color alone, or by the presence of dye concentrations around drill holes or just under the surface of the pearls.

Quartz. For literally thousands of years, quartz has been quench-crackled and dyed to imitate more valuable gem materials such as ruby and emerald. During the last decade, we observed for the first time quartz beads that had been quench-crackled and dyed to imitate amethyst (Reinitz, 1997b). In at least one sample, green dye had been mixed with an epoxy resin such as Opticon before it was introduced into the quench-crackled stone (Koivula et al., 1992j).

Of particular interest were unusual dyed quartzites (a metamorphic rock composed primarily of quartz grains) in colors such as purple (to imitate sugilite; Reinitz and Johnson, 1998). One of the most convincing of such imitations was quartzite dyed to imitate jadeite, both lavender (Koivula and Kammerling, 1991c) and green (figure 16; Kammerling, 1995a). As with most dyed gems, though, dye concentrations in the fractures and between grains provided a strong indication of treatment.

CLARITY ENHANCEMENT

The previous retrospective article titled this section "Oiling/Fracture Filling." Since that time, it has become commonplace to refer to such treatments as clarity enhancement, because that is the objective. As mentioned in the introduction to this article, the issue of disclosure of clarity enhancement had some damaging effects on the trade during the 1990s. In

fact, many of these issues have continued into the new millennium.

Diamond. Clarity enhancement of diamonds by fracture filling began in the late 1980s, with the first comprehensive article on the subject published by Koivula et al. (1989). This first article focused on the product from Yehuda Diamond Corp., the only company performing this treatment at the time. Five years later, another comprehensive article (Kammerling et al., 1994b) dealt not only with the then-current Yehuda product, but also with filled diamonds from newer players in this field, especially Koss and Goldman-Oved (figure 17).

Clarity enhancement of diamonds became a serious issue when the lack of disclosure by certain U.S. retailers led to devastating exposés in the national media (see, e.g., "Everyone's best friend," 1993). In particular, two St. Louis jewelers were accused of selling filled diamonds without disclosing the treatment ("Five on your side," 1993), which eventually led to the destruction of their business and even the tragic death of one of them ("Rick Chotin..." 1994).

The key identifying feature for fracture-filled diamonds remains the flash effect: the different colors seen when the fracture is viewed at an angle nearly parallel to its length, first in darkfield and then in brightfield. Colors seen perpendicular to the fracture are not flash colors and are due to diffraction within feathers that most often contain only air. The 1994 article by Kammerling et al. showed that while the identifying features of filled diamonds from the three manufacturers were similar in many respects, there were differences in the intensity and hue of the flash colors from one product to another; however, no flash effect was sufficiently unique to identify a particular manufacturer. This was also the case with other microscopic features typical of filled stones, such as trapped gas bubbles, areas of incomplete filling (particularly at the surface), and cloudy fillings.

A number of other studies concerning clarity enhancement of diamonds were published during the first half of the 1990s (Scarratt, 1992; Nelson, 1993, 1994; Sechos, 1994; McClure and Kammerling, 1995). All were aimed at disseminating the identification criteria for this treatment to as many people in the trade as possible.

Also noteworthy was the discovery that *rough* diamonds were being filled and then shipped to Africa to be sold (Even-Zohar, 1992). This obvious



Figure 17. Clarity enhancement of diamonds can be very effective, as illustrated by these before (left) and after (right) views of a 0.20 ct diamond that was treated by the Goldman-Oved Company. Photomicrographs by Shane F. McClure.

attempt to defraud buyers was quickly condemned by the diamond industry, and a resolution was eventually passed by the combined leadership of the International Diamond Manufacturers Association and the World Federation of Diamond Bourses that prohibited the filling of rough or the selling of filled rough (Even-Zohar, 1994).

Filled fractures were observed in several colors of fancy diamonds, including yellow (McClure and Kammerling, 1995), pink (Reinitz, 1997a), and brown (Sechos, 1995). The yellow-to-orange flash effect normally seen in darkfield illumination was almost not visible in the yellow diamond, although the dark blue brightfield flash color stood out quite nicely on the yellow background. The color appearance of the pink diamond improved as the numerous large fractures in the stone were made transparent by the treatment.

Variations in the flash effect were reported occasionally. One diamond showed a vivid blue flash color that resembled a dark "navy" blue ink splotch (Hargett, 1992a). In some filled diamonds, the flash colors are so subtle as to be easily overlooked; in such cases, the use of fiber-optic illumination is invaluable (Kammerling and McClure, 1993a). Conversely, another note reported flash colors that were so strong as to appear pleochroic in polarized light (Johnson, 1996b).

Johnson et al. (1995) reported a filling material with an unusual chemical composition: It contained thallium in addition to the more typical trace elements found in fillers, Pb and Br. They speculated that this might have been one of the earlier filled diamonds, as there were rumors that some of the first fillers contained thallium.

Even though much has been published about the inability of diamond filler materials to withstand heat, gemological laboratories commonly see filled diamonds that were damaged during jewelry repair procedures. In almost all cases seen to date, the jew-

eler was not told that the stone had been clarity enhanced and did not take the time to look at the diamond with magnification for the telltale signs. Such were the circumstances with a 3.02 ct diamond that was eye clean before the jeweler started repair work on the ring in which it was mounted (Hargett, 1992b). The large, eye-visible fractures that appeared in the center of the stone when the mounting was heated created a difficult situation for the jeweler. This scenario has been played out many times since then. A later report described filler material that actually boiled out of the fractures and deposited on the surface of the diamond in small droplets (Johnson and McClure, 1997a).

Emerald. There has never been a better example of the impact that a gem treatment can have on the jewelry business than what occurred with emeralds during the last decade. Even though emeralds have undergone some sort of clarity enhancement for centuries (figure 18), not until the 1990s did this treatment and its disclosure become a critical issue for the trade. A series of unfortunate events created a loss of consumer confidence, particularly in the United States, that had a devastating effect on the emerald market. Bad press in the form of high-profile lawsuits, and local and national television exposés on programs such as *Dateline NBC* ("Romancing the stone," 1997), contributed to this problem, but they were certainly not the only cause. One noted emerald dealer pointed out that this lack of consumer confidence started in 1989, when a synthetic resin called "palm oil" or "palma" became prevalent for fracture filling in Colombia (Ringsrud, 1998). He attributed the problem to the fact that this substance, which has an R.I. of 1.57, hides fractures too efficiently and is notoriously unstable. He estimated that in approximately 20% of the stones treated with "palm oil," the filler would turn white and become translucent in only a



Figure 18. Clarity enhancement of emeralds has been done for centuries, but it became a significant issue for the trade in the 1990s. Many saw the dramatic effect this treatment can have on an emerald for the first time with the publication of photos that showed stones before enhancement (left) and after (right). Photos by Maha Tannous.

few months, so that fractures that had been virtually invisible became obvious to the unaided eye. One can only speculate as to the potential impact of such deterioration on the consumer, who probably was not told the emerald had been filled at the time of purchase.

These and other aspects of the issue were heavily debated in the trade press (see, e.g., Bergman, 1997; Federman, 1998; Schorr, 1998). Three major concerns surfaced: (1) what types of fillers were being used, (2) how permanent or durable each filler was, and (3) how much filler was present in any given stone.

The types of fillers being used for clarity enhancement of emeralds have expanded dramatically during the last decade. Kammerling et al. (1991) noted that in addition to traditional fillers such as cedarwood oil, treaters had started to use epoxy resins, the most popular of these being Opticon. This article also mentioned that proprietary filling substances were being developed by several other companies (Zvi Yehuda Ltd. of Israel, CRI Laboratories of Michigan, and the Kiregawa Gemological Laboratory of Japan).

Since that time, many other fillers have been introduced, and the infamous "palm oil" was identified as probably being the liquid epoxy resin Araldite 6010 (Johnson et al., 1999). Treaters also started to use hardened epoxy resins, with the idea that they would be more durable than the liquid materials, which tended to leak out over time. The formulas for these resins are considered proprietary and carry names such as Gematrat, Permasafe, and Super Tres.

The durability of the individual fillers remains the subject of ongoing research. There is little debate as to the nonpermanence of "palm oil" or cedarwood oil (see, e.g., Kammerling et al., 1991; Federman, 1998; Kiefert et al., 1999). However, those who use other fillers have made various

claims regarding their ability to hold up under normal conditions of wear and care. In fact, this feature has been the focus of marketing efforts by several of the treaters who offer hardened resins (Johnson and Koivula, 1997b; Weldon, 1998a; "New type of epoxy resin," 1998; Fritsch et al., 1999a; Roskin, 1999).

An interesting development during this debate came when many in the industry claimed that a desirable feature of a filler would be the ability to remove it. Because some of these resins decompose and turn white or cloudy with time, dealers recognized that they eventually would need to be removed so that the stones could be retreated. This was a valid concern, as attempts to remove these unstable fillers often have been unsuccessful (Themelis, 1997; Hänni, 1998).

Also during this decade, a number of laboratories maintained that they could comfortably make the distinction between specific types of fillers and began to offer such a service (see, e.g., Hänni et al., 1996; Weldon, 1998c; Hänni, 1998, 1999; Kiefert et al., 1999). Others believe that while these fillers may be separated into broad categories, it can be difficult or even impossible to identify mixed fillers or stones that have been treated multiple times with different fillers (Johnson et al., 1999).

In light of this debate, many have suggested that the amount of filler in a given emerald is perhaps more important than the kind of filler used (Johnson and Koivula, 1998b; Drucker, 1999). Thus, many laboratories offer a service that classifies the degree of enhancement. In most cases, the system uses four or more classifications, such as *none*, *minor*, *moderate*, and *significant* (see, e.g., McClure et al., 1999).

The criteria used to detect fillers in emeralds have been described at length by various researchers (see, e.g., Johnson and Koivula, 1998b; Hänni, 1999). These criteria primarily consist of

flash effects (figure 19), incomplete areas of filling or gas bubbles (figure 20), and whitish or deteriorated filler within the fractures—all of which can be seen with magnification.

Additional information about emerald fillers was published throughout the decade. Hughes Associates, the manufacturer of Opticon, reported that the refractive index of Opticon can range from 1.545 to 1.560, depending on the amount of hardener added (Koivula et al., 1993b). The chemistry of fillers was closely examined to determine if it could be an aid in identification (Johnson and Muhlmeister, 1999). Unfortunately, the answer was *no*. The new hardened filler Permasafe was characterized by Fritsch et al. (1999a). Early on, two Brazilian dealers reported that some treaters were adding a green coloring agent to Opticon (Koivula and Kammerling, 1991a), a practice that is not acceptable in the trade.

Other Gem Materials. Of course, it was inevitable that clarity enhancement would find its way into other gem species. We know of two reported incidences in the '90s: one in alexandrite (Kammerling and McClure, 1995a), and the other in a pyrope-almandine garnet (Kammerling and McClure, 1993b).

IMPREGNATION

Impregnation of porous gem materials with different kinds of polymers to improve their appearance or durability has been widespread for many years. The 1990s saw major developments concerning the use of this treatment technique on a number of important gem materials.

Jadeite. The most significant gem material affected by impregnation during the last decade was jadeite. The treatment process, which is often referred to as "bleaching," caused such an uproar in the jade industry that jadeite sales in Japan fell as much as 50% over a three-month period in the beginning of the decade ("New filler threatens jadeite sales in Japan," 1991).

"Bleaching" actually involves a two-step process. First the jadeite is immersed in an acid to remove the brown iron oxide staining that is so common in this material. This staining gives the stone a brown coloration that is less desirable and therefore detrimental to its value. The result after "bleaching" is a color such as pure green or green and white. Unfortunately, this process leaves



Figure 19. The flash effect is one feature that can be used to identify if an emerald has been filled. The two most common colors, orange and blue, are seen in this stone in a fracture that is otherwise almost invisible. Photomicrograph by Maha Tannous; magnified 15 \times .

behind voids in the structure of the jadeite, which make the grain boundaries of the aggregate material readily visible, and many fractures may appear. Not only do these voids and fractures adversely affect the translucency of the gem material, but they also can affect the durability of the jadeite, so that it is more susceptible to breakage (Fritsch et al., 1992).

It is because of these adverse effects that the sec-

Figure 20. Another feature that can help determine whether an emerald has been filled is the presence of gas bubbles or unfilled areas within a very low relief fracture. Photomicrograph by Shane F. McClure; magnified 22 \times .

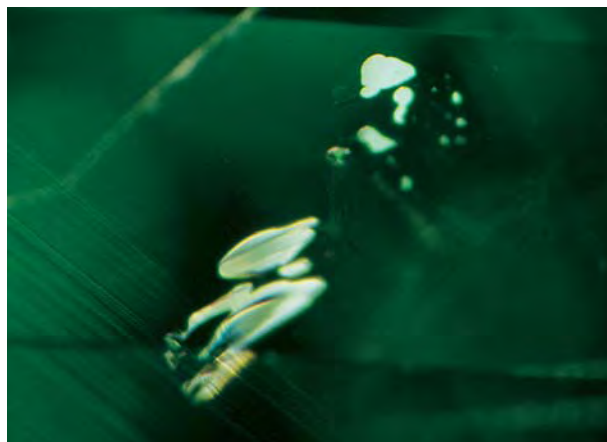
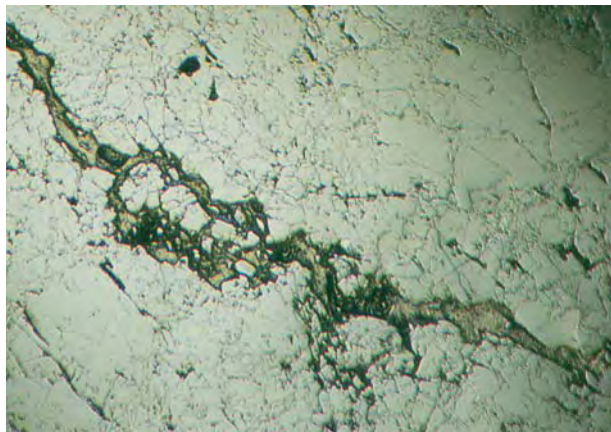




Figure 21. All of these jadeite cabochons have been bleached and subsequently impregnated with a polymer to improve their appearance. The overall result is usually quite effective. Photo by Maha Tannous.

ond step of the process is necessary: The “bleached” jadeite is impregnated with a polymer (usually an epoxy resin) to fill the voids and return the stone to an acceptable translucency (figure 21). This addition of a foreign material created the need for a new classification of jadeite. The bleached and polymer-impregnated material came to be known as “B jade.” “A jade” refers to jadeite that has not been treated at all, and “C jade” is used for dyed jadeite.

Figure 22. Structural damage caused by the bleaching process is clearly seen in this treated jadeite. Also visible is a large fracture filled with the impregnating polymer. Photomicrograph by Shane F. McClure; magnified 30×.



The origins of this treatment lie somewhere in the mid-1980s, and an early report was given by Hurwit (1989). The beginning of the '90s saw an explosion of bleached jadeite on the market. Since no in-depth studies had been done on the material at that time, there were no procedures in place to identify it. Once this became widely known, and all jadeite became suspect, the price of jadeite plummeted.

The first comprehensive study on the identification of bleached and impregnated jadeite was published by Fritsch et al. (1992). These researchers found that the only conclusive way to detect if a piece had been treated was to examine its infrared spectrum for the telltale “signature” of the polymer filler. Subsequently, a number of other identification methods were described, such as the use of X-ray photoelectron spectroscopy (Tan et al., 1995) and diffuse reflectance Fourier-transform infrared (FTIR) spectroscopy (Quek and Tan, 1997), as well as the use of a simple drop of acid (described in Fritsch et al., 1992, and elaborated in Hodgkinson, 1993). Some of these methods were even used to identify polystyrene as one of the polymers used (Quek and Tan, 1998). However, infrared spectroscopy remains the easiest test to perform, provided one has the necessary equipment. By the end of the decade, several jadeite dealers had purchased an FTIR spectrophotometer so they could personally test all the jadeite they handle.

It was also noted early on that the structural damage caused by the bleaching process could be seen in reflected light with a microscope (figure 22—Ou-Yang, 1993; Moses and Reinitz, 1994; Johnson and DeGhionno, 1995). This surface texture has been referred to as having an etched or honeycomb-like appearance, which is a manifestation of the gaps or voids left between the individual grains in the jadeite structure. Articles were published on the use of a scanning electron microscope to study and document this phenomenon so that it might be used as an aid in identification (Tay et al., 1993, 1996).

Tests conducted on the durability of this material found that long-term exposure to detergents could damage or remove some of the filler. Also, heating at 250°C can turn the treated jadeite brown (Johnson and Koivula, 1996b).

Some particularly unusual examples were reported: a bangle bracelet with internal gas bubbles generated by the filling of cavities that were created when the acid etched out carbonates within the

Figure 23. These turquoise cabochons were treated by the Zachery process, which decreases the porosity of the material, making it less likely to discolor with time and wear. Photo by Maha Tannous.



jadeite (Koivula, 1999), a necklace that had a mixture of treated and untreated jadeite beads (Kammerling, 1995b), and the first reported instance of a bleached and polymer-impregnated lavender jadeite (Kammerling et al., 1994a).

The most important thing for the gemologist to remember about this treatment is that it can be identified conclusively only by sophisticated means such as infrared spectroscopy. There may be some indications, such as a yellow fluorescence, low specific gravity, or coarse surface texture, but these do not prove that a piece of jadeite has been treated.

Turquoise. Turquoise is notorious for being impregnated. Because its inherent porosity makes it subject to discoloration from wear, treatment is very common. As one might expect, impregnation of turquoise with plastics (Kammerling, 1994a,b) and oils (Koivula et al., 1992g, 1993c) was still prevalent in the 1990s.

The most significant turquoise treatment that came to light in the '90s may not be an impregnation at all. Called Zachery treatment after the man who developed it, this process actually was introduced in the late 1980s, although the first major study did not appear until 1999 (Fritsch et al., 1999b). During this decade, millions of carats of Zachery-treated turquoise entered the trade (figure 23).

The process is still a closely guarded secret, so exactly how it effects the change in turquoise is not completely understood. We do know that Zachery treatment reduces the porosity of turquoise, but

there is no evidence that it adds any polymers or other foreign material. The end result is turquoise that does not absorb oils or other liquids during wear and therefore does not discolor, as most natural turquoise does in time. The turquoise can be treated without changing its original color, or the color can be darkened, depending on the wishes of the client.

Regardless of the actual enhancement mechanism, the only way to prove conclusively that an individual piece of turquoise has been treated by this process is through chemical analysis, since Zachery-treated turquoise usually has an elevated potassium content. Visual indications of this treatment include a slightly unnatural color, a very high polish, and blue color concentrations along surface-reaching fractures (figure 24).

Opal. While there were no new advances in the impregnation or "stabilization" of some kinds of matrix opal, which has been a common practice for many years, there were a few other notable developments with regard to opal.

The hydrophane opal mentioned in the Dyeing section, which was treated to resemble Australian black opal, was also impregnated with a plasticized liquid to seal the porosity and improve the transparency after the dyeing process (Koivula et al., 1992c).

Impregnated synthetic opal appeared on the market during this decade. It is not difficult to identify, because the specific gravity (around 1.80–1.90) is



Figure 24. Although Zachery treatment can be proved only through chemical analysis, the presence of color concentrations along fractures in the turquoise is a good indication. Photomicrograph by Shane F. McClure; magnified 10 \times .

too low for untreated material (Kammerling and McClure, 1995b; Kammerling et al., 1995; Fritsch, 1999). However, controversy arose when some in the trade objected to the use of the term *synthetic* in association with this material, because it is impregnated with plastic. This nomenclature issue is still being investigated and discussed.

SURFACE COATINGS

Changing the color of gem materials by the use of colored surface coatings was a very popular treatment throughout the 1990s, as it has been for centuries. We continue to see different kinds of coatings on various gems, sometimes to imitate more valuable stones and sometimes to create a unique look not associated with a natural material.

Plastic remained a popular coating substance. To improve transparency and luster, treaters used both plastic and wax to coat **jadeite** (Koivula and Kammerling, 1990b, 1991i). Plastic also provided stability to fossilized **ammonite** that was unstable due to natural frost shattering in surface deposits (Koivula and Kammerling, 1991h). A transparent colored plastic coating was used to impart an emerald-like appearance to beads fashioned from light **green beryl** (Crowningshield, 1995a). The presence of air bubbles and abnormal surface irregularities visible with magnification, as well as reaction to a “hot point,” are the best means to identify this type of coating.

The surface coating of colorless **topaz** was widespread, with different processes being used by the end of the decade. Orange, pink, and red material (see, e.g., figure 25) showed a spotty surface coloration (detected with low magnification) that was easily scratched by a sharp object. Although the process was originally represented as diffusion treatment, these colors (unlike the green-to-blue surface-treated topaz described in the earlier Diffusion Treatment section) were probably produced by sputter coating (Johnson and Koivula, 1998d).

Thin metallic coatings remained popular for treating both **quartz** and **topaz**, as crystals and as faceted stones (figure 26). Microscopic examination of gold-coated blue to greenish blue “Aqua Aura”-treated samples, which made their debut in the late 1980s, revealed unnatural coloration at facet junctions and an irregular color distribution on some facets (figure 27—Koivula and Kammerling, 1990a; Kammerling and Koivula, 1992). Durability testing of these gemstones showed that even though the coating is relatively hard and chemically inert (Koivula and Kammerling, 1990a), care must be taken to avoid damage during jewelry manufacturing or repair (Koivula and Kammerling, 1991d). New colors and effects were created in coated quartz by using different combinations of metallic elements. These included purple, yellow, green, and red hues created by Au, Bi, Pb, Cr, Ti, and other elements (Johnson and Koivula, 1996a), as well as a “rainbow” iridescence that was reportedly caused

Figure 25. These topazes were originally represented as being diffusion treated, but they actually were coated with a color layer that was easily scratched off. The pink stone is 3.19 ct and the red one, 3.29 ct. Photo by Maha Tannous.



by an Ag/Pt coating (Koivula and Kammerling, 1990e). A colorless sapphire with a yellowish orange coating that was seen in the early 1990s also might have been treated by such a process (Moses and Reinitz, 1991).

The demand for certain colors of **sapphire** led to the resurfacing of some “old tricks” in Sri Lanka that used organic compounds to create surface coatings (Koivula and Kammerling, 1991b). Pale or colorless rough was turned yellow by boiling in water (sometimes with wax added) that contained the branches or bark of a local tree. Some Sri Lankans took a similar “low-tech” approach to imitate pink sapphire rough: The treater placed the pale or colorless sapphire in his mouth along with a local berry, chewed the berry to create the pink coating, and followed this by smoking a cigarette (which reportedly improves the durability of the coating). These treatments may seem unimportant, but to the gem buyer alone in a remote area of Sri Lanka, knowing about them could mean the difference between a successful trip and a disaster.

Coated **diamonds** were still encountered in the laboratory during the 1990s, although less frequently. One such stone showed a brownish purple-pink color that rarely occurs naturally in diamonds (Crowningshield and Moses, 1998). Although the exact nature of the coating substance could not be identified, its speckled appearance over the entire stone suggested a sputtering process. **Diamond-like carbon** (DLC)—an amorphous brown material with a hardness between that of diamond and corundum—was used experimentally at the beginning of the decade to coat several gemstones, which resulted in greater durability (Koivula and Kammerling, 1991e). More recently, DLC was identified on a treated-color “black” diamond by Raman analysis; researchers used the same method to tentatively identify a carbide compound on a treated-color green diamond (Reinitz et al., 2000a).

New **pearl** coatings presented some significant identification challenges in the 1990s. A strand of lustrous black circled cultured pearls was found to be coated with a form of silicone called polydimethyl siloxane (Hurwit, 1999). A peculiar smoothness, sticky feel, and slight anomalous reaction to a thermal reaction tester were the only clues to the presence of the coating; advanced techniques were needed to identify it. Mabe assembled blister pearls also were coated, but Hurwit (1991) reported that the lacquer coating was applied to the plastic dome *under* the layer of nacre. The effect

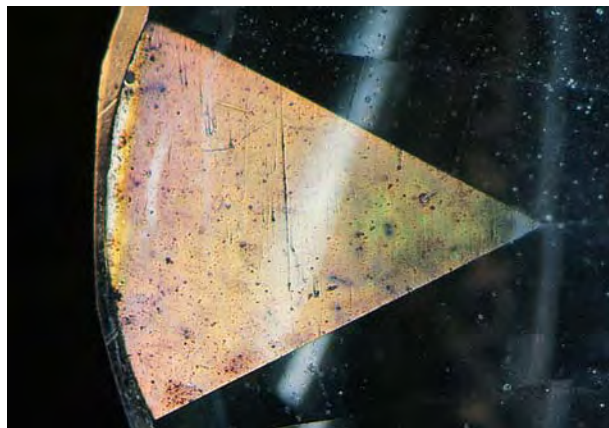


Figure 26. Aqua Aura treatment was still used extensively on quartz (the two inside stones) and topaz (the two outside stones) throughout the '90s. Photo by Robert Weldon.

was to improve the luster and overtone of the white mabe pearls. A spotty, uneven color distribution suggested the presence of an enhancement, but only by disassembling a sample could the coating be confirmed.

To produce a dark background and thus bring out the play-of-color, **opal** was subjected to several coatings, including: (1) black paint on the base of diaphanous opal from Australia (Brown et al., 1991),

Figure 27. Aqua Aura treatment is easily detected by the presence of unnatural surface coloration on the facets of a stone. Photomicrograph by John I. Koivula; magnified 12 \times .



(2) a dark plastic-like material on portions of a Mexican opal (Koivula and Kammerling, 1990c), and (3) sugar-treated opal that appeared to be further coated with a plastic-like substance (Koivula and Kammerling, 1990d). All of these coatings were readily apparent with microscopic examination.

Two other relatively isolated occurrences of coatings deserve mention. A brittle glass-like coating is responsible for the dark violet-blue color of some drilled **quartz** beads (Kammerling and McClure, 1994). This coating, possibly applied by an enameling process, was identified through a combination of microscopic examination of the drill holes, hardness testing, and advanced techniques. Koivula et al. (1992a) noted that acrylic spray could be used to enhance the luster of massive gem materials such as **lapis lazuli** and **jadeite**. Such a coating is easily identified: With magnification, slight concentrations are seen in surface irregularities, and the acrylic can be easily removed if it is rubbed with a cotton swab that has been dipped in acetone.

It is interesting to note that the use of coatings has spread to some laboratory-grown materials. A company in northern California trademarked the name Tavalite (Johnson and Koivula, 1996d) for **cubic zirconia** that had been treated with an optical coating. The process created six different colors that had a different appearance in reflected and transmitted light. This product was very easy to identify, in that it does not resemble any other material.

CONCLUSION

It can safely be said that events of the 1990s changed the attitude of the entire industry toward treatments and disclosure, which today constitute the single most important issue facing the trade. Identification of some of the significant treatments—such as glass-filled rubies, HPHT-processed diamonds, and a variety of irradiated gem materi-

als—continues to challenge many gemologists. Within the last year, we have already seen significant new developments in the laser drilling of diamonds (McClure et al., 2000), as well as in the material used to fill fractures in diamonds (Shigley et al., 2000b). In addition, there has been recent talk of new filling processes that will bring true clarity enhancement to higher-refractive-index colored stones such as ruby, sapphire, and alexandrite (Arthur Groom, pers. comm., 2001). Also, the technology being used to create the “diffusion treated” blue-green topaz can be applied to other gem materials, and it is likely that some of these will reach the market in the future. One of the authors (SFM) has already seen colorless quartz turned pink by this process. All of these developments will undoubtedly create more identification challenges.

Some of the issues regarding disclosure may not have solutions that will be agreeable to everyone in the industry. However, there were a number of meetings in the latter half of the ‘90s at which leaders of prominent gemological laboratories and trade organizations worldwide met to establish better communications and greater consistency in reporting terminology. These meetings illustrate the determination of the jewelry industry to address these issues and find solutions that will benefit members of the trade and consumers alike.

The 1980s retrospective article asked the question, “What new treatments might face us in the not-too-distant future?” Yet technological advances in the last 10 years have produced treatments, such as removing the color from brown diamonds, that most of us would not have thought possible at the beginning of the decade. Without a doubt, technology will continue to advance at an even faster rate during the next decade. The only thing we can guarantee is that there will be no end to fresh challenges in treatment identification and disclosure as we enter the new millennium.

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