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GEMSTONE ENHANCEMENT AND ITS DETECTION IN THE 2000S

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Advances in technology and increased demand for lower-priced gem materials contributed to the proliferation of new treatments throughout the first decade of the 2000s. The developments that made the most difference were the diffusion treatment of corundum with beryllium, diffusion of copper into feldspar, clarity enhancement of ruby and diamond, and heat treatment of diamond, ruby, and sapphire. Gemological laboratories and researchers have done their best to keep up with these treatments, and the jewelry trade has struggled with how to disclose them. This article summarizes these developments and the methods used to identify the various enhancements.

Another decade has passed since we reviewed the events of the 1990s as they pertained to gemstone enhancements and their detection (McClure and Smith, 2000). At that time, we observed that the issue of disclosure (and, especially, the failure to disclose) had caused major upheaval in all areas of the jewelry industry. We ended that retrospective article by stating there would be no end to fresh challenges in treatment identification and disclosure as we entered the new millennium.

The 2000s certainly lived up to our expectations. There were treatments discovered that no one suspected were possible. There were crises of disclosure that resulted in televised exposés and unfavorable publicity for the industry. There were improvements in treatments developed in the ’90s that made them more efficient and often harder to detect.

Detection methods have also become more and more complex. Gemological laboratories have had to invest in more sophisticated instrumentation, sometimes at great expense. For the frontline laboratories, being a good gemologist is no longer good enough. You must also have training in the earth sciences and analytical instrumentation to function effectively in such an environment. Now more than ever, the gemologist in the trade must be able to recognize when a stone requires more advanced testing.

It is important to emphasize that many of these treatments can still be detected with standard gemological equipment, but staying current on the latest developments is absolutely essential. The knowledge base concerning treatments is constantly changing.

Nearly every gem material (e.g., figure 1) is subject to treatments of one form or another. Building on previous reviews (Kammerling et al., 1990a; McClure and Smith, 2000; Smith and McClure, 2002), the aim of this article is to provide an overview of the treatments and identification challenges associated with them that were common during the first decade of the 2000s. The authors strongly recommend that readers familiarize themselves with the original references, as all the pertinent information cannot be presented in a review article.

See end of article for About the Authors and Acknowledgments.
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Figure 1. The 2000s continued to see the widespread use of treatments on a wide variety of gemstones. The gems shown here were enhanced during the 2000s by heat (unless otherwise noted) or other methods known prior to the decade: (1) 8.43 ct aquamarine; (2) 16.86 ct tanzanite; (3) 10.08 ct tourmaline; (4) 4.65 ct Paraíba tourmaline; (5) 3.36 ct red beryl (clarity enhanced); (6) 3.07 ct ruby; (7) 53.54 ct Cu-bearing tourmaline, Mozambique; (8) 13.97 ct zircon; (9) 2.60 ct emerald (clarity enhanced); (10) 6.43 ct zircon; (11) 8.04 ct blue sapphire; (12) 12.15 ct pink sapphire; (13) 13.67 ct yellow sapphire; and (14) 9.07 ct blue topaz (irradiated and heated). Nos. 1, 3, 6, 8, 9, 11, 12, and 13 are courtesy of Evan Caplan (Omi Gems, Los Angeles); 2, 4, 5, 7, and 10 are from Fine Gems International; and 14 is from Tino Hammid. Photo by Tino Hammid, © Robert E. Kane.

NOMENCLATURE AND DISCLOSURE

Although there is no global standard regarding specifically how a seller should disclose gem treatments or enhancements, there is general agreement that they *should* be disclosed. This disclosure should be to all purchasers, at all levels of commerce (from miner to cutter, wholesaler, jewelry manufacturer, retailer, and—ultimately—the consumer). To find the proper protocol in your country or area, contact one of your national or regional colored stone and diamond organizations, such as AGTA (www.agta.org), ICA (www.gemstone.org), CIBJO (download.cibjo.org), or the World Federation of Diamond Bourses (WFDB, www.wfdb.com).

In the early 2000s, a group that came to be known as the Laboratory Manual Harmonisation Committee (LMHC) was formed at the request of leaders of the colored stone industry. Its purpose was to bring together representatives of many of the major gem laboratories and attempt to standardize wording on their reports ("International labs. . . ," 2000). The LMHC is autonomous and has representatives from the U.S., Switzerland, Thailand, Italy, and Japan. If agreement is reached on a given subject, they issue an information sheet with the wording expected to be seen on reports from those labs. To

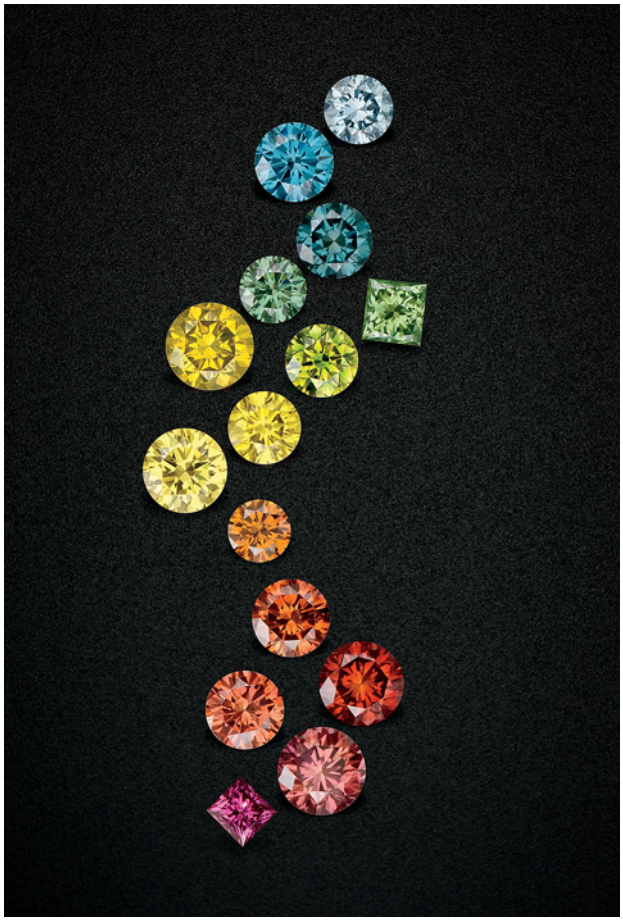


Figure 2. This group of diamonds (0.30–0.74 ct) illustrates the wide array of colors that can be produced by artificial irradiation with subsequent annealing. Courtesy of Lotus Color; photo by Robert and Orasa Weldon.

date, 10 such information sheets have been issued, and the group continues to meet twice a year (to download these standardized nomenclature sheets, go to www.lmhc-gemology.org/index.html).

THERMAL ENHANCEMENT

For a wide variety of gem materials, heat treatment is still the most common enhancement. In some cases, heat treatment can still be identified by routine methods. In others, conclusive identification is possible only with advanced instrumentation and techniques. In still other gems (e.g., aquamarine, citrine, amethyst, and tourmaline), heat treatment remains virtually unidentifiable by any currently known methods. For this last group of stones, which are heated to induce permanent changes to their color, this enhancement may be the rule rather than the exception. One should assume that most of those gem materials have been heated.

High-pressure, high-temperature (HPHT) treatment of diamonds was only introduced commercially in 1999, and much of the first decade of the 2000s was devoted to expanding this high-tech treatment to colored diamonds on the one hand—and detecting it on the other. Research efforts thus far have provided methods to identify not only the lightening of off-color diamonds, but also the production of a wide variety of fancy colors.

Diamond. The last decade bore witness to the greater presence of color-treated diamonds, with the global trade reportedly approaching 25,000 carats per month in the latter half of the decade (3–5% of the total diamond trade; Krawitz, 2007). Although not specifically noted, this figure probably refers mostly to irradiated and annealed diamonds of many different colors. Irradiation, heating, HPHT, or a combination of these treatments can create virtually every hue (figure 2), including black and colorless.

HPHT Treatment to Remove Color. HPHT treatment of diamonds to remove or induce color was a central topic of the diamond community throughout the 2000s. In 1999, General Electric Co. and Lazare Kaplan International announced the commercial application of an HPHT process for faceted diamonds (Pegasus Overseas Limited, 1999) that removed color from brown type IIa stones (by annealing out vacancy clusters associated with the brown color in plastically deformed diamonds; Fisher, 2009). Even though scientists had recognized these and other possibilities 30 years earlier (see, e.g., Overton and Shigley, 2008), the results came as a surprise to many in the diamond world—a type IIa brown diamond of any size could be transformed into a colorless stone (see, e.g., Smith et al., 2000). After HPHT treatment, the majority of these diamonds received D through G color grades, and the results were permanent (Moses et al., 1999). Gemological researchers globally mobilized to understand and identify the process (e.g., Chalain et al., 1999, 2000; Schmetzer, 1999; Collins et al., 2000; Fisher and Spits, 2000; Smith et al., 2000).

By late 2000, more than 2,000 decolorized type IIa HPHT-treated diamonds had been seen at the GIA Laboratory (McClure and Smith, 2000). Today, with several treaters in various countries removing color from diamonds with HPHT annealing, this treatment has become almost commonplace.

Determining diamond type is central to the detection of colorless to near-colorless HPHT-treat-

ed diamonds. For a thorough review of how diamond type is determined, see Breeding and Shigley (2009). Nearly 99% of all natural gem diamonds are type Ia. Thus far, all colorless to near-colorless HPHT-treated diamonds reported in the literature have been type IIa. Fortunately, it is easy to determine if a diamond is *not* a type IIa by using the DiamondSure (Welbourn et al., 1996), SSEF Type II Diamond Spotter (Boehm, 2002; Hänni, 2002), or other simple gemological methods (Breeding and Shigley, 2009). At the present time, if a colorless to near-colorless diamond is not type IIa, then it is not HPHT treated.

Visual features related to damage caused by the extreme conditions of the treatment may be seen in some colorless to near-colorless HPHT-treated diamonds. These include a frosted appearance caused by etching or pitting, as well as gray or black graphitization, on naturals or fractures where they come to the surface. Such features are not *commonly* observed in untreated colorless type IIa diamonds, although lightly pitted surfaces and graphitized or graphite inclusions have been seen on rare occasions. Therefore, such features are a good indication of treatment, but they are not proof by themselves (Moses et al., 1999; McClure and Smith, 2000; Gelb and Hall, 2002). Because these heat damage–related features are not always present in a faceted diamond or may be difficult to discern, detection of HPHT treatment in a type IIa diamond generally requires measurement of the absorption and/or photoluminescence (PL) spectra taken with the diamond cooled to a low temperature (see Chalain et al., 1999, 2000; Collins et al., 2000; De Weerdt and Van Royen, 2000; Fisher and Spits, 2000; Hänni et al., 2000; Smith et al., 2000; Collins, 2001, 2003; Novikov et al., 2003; and Newton, 2006).

HPHT Treatment to Produce Color. Refinements to HPHT processing have yielded commercial production of a variety of colors in both type I (orangy yellow, yellow, to yellow green) and type II (pink or blue) diamonds (Shigley, 2008; see, e.g., figure 3).

Identifying HPHT-treated type Ia diamonds requires both IR and low-temperature visible-range spectroscopy, but several gemological properties offer evidence (see Reinitz et al., 2000). The pink and blue HPHT-treated diamonds initially examined by Hall and Moses (2000, 2001b) ranged from Faint and Very Light to Fancy Intense and Fancy Deep. Low-temperature PL spectra identified these products. As discussed below, combining treatments (e.g., HPHT annealing, irradiation, then low-temperature heating) can produce interesting results, such as intense pink-to-red diamonds (Wang et al., 2005b). Smith et al. (2008a,b) contributed useful charts for identifying the natural or treated origin of color in pink and blue diamonds.

Figure 3. This 0.34 ct Fancy Light gray-blue diamond was successfully turned Fancy blue by HPHT treatment. Photos by Elizabeth Schrader.

Heat-Treated Black Diamond. In the late 1990s, it became popular to pavé-set small natural-color black diamonds alongside colorless diamonds in jewelry (Federman, 1999; Gruosi, 1999; Misiorowski, 2000). This design trend continued into the 2000s. As is often the case with successful jewelry lines, less-expensive approaches soon followed. Harris and Vance (1972) had experimented with the production of artificial graphitization in diamond, which Hall and Moses (2001a) confirmed by heating a sample under vacuum for a few minutes to several hours and turning it black; Raman spectra showed a pattern that matched graphite. Notari (2002) discussed several different commercially practiced methods of heating to produce graphitization and black coloration in diamonds.

In many cases, microscopic examination with fiber-optic illumination can provide proof of heat treatment in black diamonds—the black (graphitized) areas are largely confined to surface-reaching cleavages and fractures (Hall and Moses, 2001a). In natural-color black diamonds, the graphitization is randomly dispersed throughout, referred to as a "salt and pepper" effect (Kammerling et al., 1990b). This random orientation is also seen in other color-causing inclusions in natural-color black diamonds, such as magnetite, hematite, and native iron (Titkov et al., 2003). This determination, however, requires a gemologist experienced in examining known samples of both natural-color and heat-treated black diamonds (see, e.g., Smith et al., 2008c).

Ruby and Sapphire. As in the preceding two decades, the heat treatment of corundum to substantially

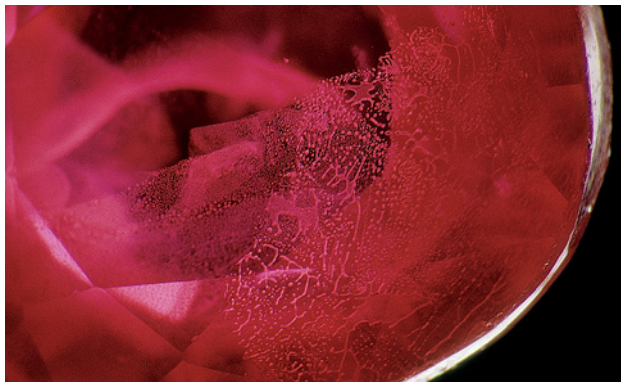


Figure 4. Heat treatment with the stone placed in a flux has largely healed this fracture in a Mong Hsu ruby. However, it has left behind a "fingerprint" that looks similar to those found in synthetic rubies, another challenge for the gemologist. Photomicrograph by S. F. McClure; magnified 40×.

change its color remained a troublesome issue. Heating was applied to the vast majority of rubies and all colors of sapphires during the 2000s.

In some cases, clarity was also affected, as with the flux-assisted healing of fractures (in combination with high-temperature heat treatment) that began in the early 1990s with the discovery of huge quantities of ruby at Mong Hsu, Myanmar (see Peretti et al., 1995; figure 4). The 2000s ushered in a greater understanding of this material—which dominated the ruby market—and cooperation between gemological laboratories to adopt standardized wording to describe heat treatment in corundum and, most importantly, the degree to which fracture "healing" has occurred and the amount of solidified flux "residue" (see e.g., www.lmhc-gemology.org/index.html). Today, there is less production of ruby at Mong Hsu, but this technique is now being used on rubies from Africa (Shor and Weldon, 2009).

There were also new areas of concern, such as beryllium diffusion with high heat (see "Diffusion Treatment" below) and the "Punsiri" high-temperature treatment for blue sapphires. With regard to the latter, concerns arose in late 2003 when some laboratories first observed unusual color concentrations in larger heat-treated blue sapphires (figure 5) immersed in methylene iodide (Scarratt, 2004; Smith et al., 2004). All had one consistent characteristic: a colorless or near-colorless outer rim and a deep blue (or, if color change, purple) interior (figure 6).

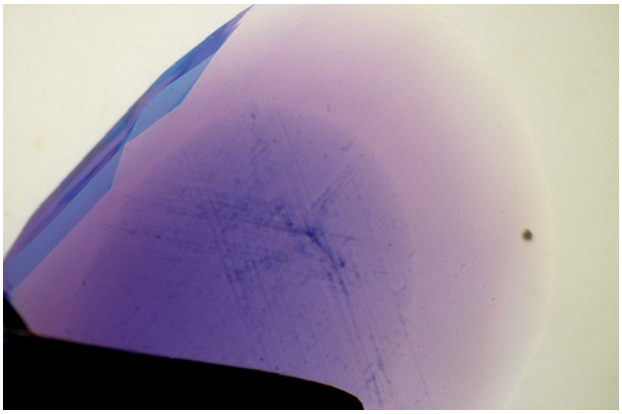
After comprehensive analytical research (and GIA and AGTA observation of the technique as performed by treater Tennakoon Punsiri in Sri Lanka), the SSEF, AGTA, and GIA laboratories all came to the same conclusion: These stones were not diffused with beryllium or any other element (McClure, 2003b; Hänni et al., 2004; "ICA issues its first lab report. . . ," 2004). The major gem labs continue to identify sapphires treated by the "Punsiri" method as natural sapphires that show evidence of heat treatment.

Beginning mid-decade, demand and scarcity significantly drove up prices for colorless or "white" sapphire. As a result, dealers in Sri Lanka reported that lightly colored sapphires had been heated to render them colorless (Robertson, 2008). Ironically, the scarcity of natural white sapphire was caused in part by the large quantities that were being used for Ti blue diffusion and, to a lesser extent, Cr red diffusion.

Since the two previous *G&G* retrospective articles on treatments (Kammerling et al., 1990a;

Figure 5. This matched pair of blue sapphires (3.07 and 3.10 ct) were heat treated by the Punsiri method. Photo by Maha Tannous.

Figure 6. The unusual color zoning in this color-change sapphire is typical of stones treated by the Punsiri method. Photomicrograph by S. F. McClure; magnified 10×.



McClure and Smith, 2000), heat-treatment technology—in the form of electric furnaces with precise temperature and atmospheric controls—has become more sophisticated and accessible. During the 1980s and 1990s, nearly all commercial corundum heat treatment was being conducted in Thailand. While Thailand remains important, Sri Lanka is now a major force, and smaller yet very effective corundum-heating capabilities exist in other producing regions such as Africa, Myanmar, China, and the U.S. (Montana). Nevertheless, some pink sapphires and rubies continue to be heated using simple blowpipe methods at mine sites and trading centers in Vietnam, Sri Lanka, and elsewhere (R. Hughes, pers. comm., 2010).

Heat treatment, particularly at high temperatures, can dramatically alter the internal characteristics and properties of sapphires and rubies. During the past decade, a number of articles addressed heat-treatment techniques and their effects on gem corundum from localities such as Madagascar (Wang et al., 2006a), Montana (Schmetzer and Schwarz, 2007; Kane, 2008), Australia (Maxwell, 2002), Vietnam (Winotai et al., 2004), Myanmar (Kyi et al., 1999), and Malawi (Rankin, 2002; Rankin and Edwards, 2003). Schmetzer and Schwarz (2005) discussed the identification of natural, heated, and Be-diffused yellow to reddish orange sapphires from Sri Lanka, Montana, Madagascar, and Tanzania. David and Fritsch (2001) contributed a valuable study on the use of infrared spectra to distinguish heated rubies and sapphires from 20 different geographic origins.

Proof that a ruby or sapphire has been heat treated is sometimes readily apparent, but in many cases it requires considerable knowledge and observational skills. The criteria for identifying heat treatment in rubies and sapphires using a microscope were set forth during the 1980s and '90s (for a summary, see Kammerling et al., 1990a). Most still apply. They include stress fractures surrounding melted or heat-altered inclusions; spotty coloration in blue stones, best seen with diffused illumination; colored halos surrounding altered solid mineral inclusions; stubby, partially absorbed (dot-like) silk; and pockmarked, resorbed facets.

Relatively low-temperature heating (i.e., 800–1200°C), particularly of purplish pink sapphires (and some purplish red rubies) to remove the blue color component, is still very difficult to detect with standard microscopic testing. The lower the temperature used, the more difficult the detection will be (Krzemnicki, 2010).

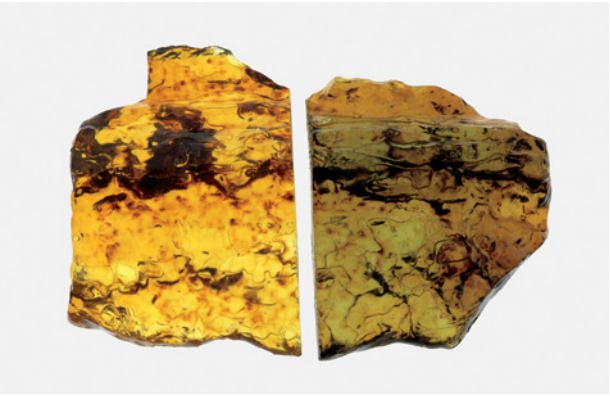


Figure 7. This ~7.5-cm-wide piece of amber was cut in half and the segment on the left treated by the first step of the "greening" process and the half on the right treated by both steps to turn it green. Photo by C. D. Mengason.

Equally important is being able to prove that a ruby or sapphire has *not* been heat treated. The decade yielded rich contributions in this area; see Shor and Weldon (2009) and Shigley et al. (2010) for important literature references. Smith et al. (2008d) and Smith (2010) provided useful charts for identifying the natural or treated state in rubies and sapphires from around the world.

Amber. Amber and copal are still heated to improve clarity, color, and hardness, and to induce "sun spangles" (Kammerling et al., 1990a; O'Donoghue, 2006). In 2009, Abduriyim et al. described a new method to produce a green color in amber and copal (figure 7), some as bright and green as peridot, using a two-stage process of controlled heat and pressure in an autoclave for long durations. Multiple treatments may increase the color saturation, producing an intense, pure green hue that has not been seen in untreated amber. The treatment also reportedly hardens the amber, making it more stable (Abduriyim et al., 2009). While infrared spectroscopy can distinguish amber from copal (Guiliano et al., 2007), this new treatment process "ages" the copal, rendering its properties similar to those of amber and making its identification as copal extremely difficult, even with advanced analytical methods.

The presence of a small absorption around 820 cm^{-1} in the FTIR spectra confirmed the use of multiple treatments on all the commercial "green amber" samples tested by Abduriyim et al. (2009). Although the use of heat treatment on a specific piece can be ascertained, whether or not the original starting material was copal or amber still cannot be routinely identified.

Garnet. Around 2003, members of the trade began reporting that Russian demantoid is routinely subjected to low-temperature heat treatment to remove or reduce the brown color component ("The reds. . . ," 2003; N. Kuznetsov, pers. comm., 2003). Other than the presence of altered inclusions in some stones, no measurable gemological means of detection has yet been reported. The result is that some international laboratories make no determination of whether a demantoid has been heated, whereas others will state if indications of heating are present (Pala International, 2010).

Spinel. As was the case with garnet, it was long believed that spinel was never treated. Beginning in 2005, however, researchers determined that certain pink-to-red spinels from Tanzania were heat treated (Saeseaw et al., 2009a). In 2007, four large (6–54 kg) spinel crystals were faceted into many thousands of fine gems from melee sizes up to 10–50 ct (Pardieu et al., 2008). Again, rumors of heated spinel began to circulate. This prompted researchers to conduct before-and-after heat treatment studies of spinel from various localities. It was concluded that heated and unheated natural spinel could easily be distinguished by the width of the 405 cm^{-1} Raman line, or by examining the width of the Cr^{3+} PL spectrum line in stones containing sufficient chromium (Saeseaw et al., 2009b,c; Kondo et al., 2010).

Tourmaline. The heat treatment of Cu-bearing tourmalines from Paraíba, Brazil, and the enormous demand for both the natural-color and heat-treated material, continued through the decade. An interesting twist occurred when Cu-bearing tourmalines were discovered in Nigeria (Smith et al., 2001; Breeding et al., 2007) and Mozambique (Abduriyim and Kitawaki, 2005; Abduriyim et al., 2006; Laurs et al., 2008).

These tourmalines were commonly heated (e.g., figure 8) to create a wide range of attractive colors similar to many of those found in Paraíba. With the exception of obviously heat-altered inclusions, standard testing cannot identify heat treatment in these tourmalines.

For several decades, heat has been known to reduce saturation in overdark red tourmalines. However, many cutters resist heating these stones because tiny fluid inclusions tend to burst during heating and cause breakage (B. Barker, pers. comm., 2008).

Figure 8. This copper-bearing tourmaline from Mozambique was cut in half, and the piece on the right (8 × 20 mm) was then heat treated. Photo by C. D. Mengason.

Zircon. Faceted orangy, pinkish, and yellowish brown zircons from Tanzania, known by trade names such as "cinnamon" zircon, were plentiful in the market (see figure 1, no. 10). To lighten overdark tones, nearly all such stones in the market have been heated—often in a test tube with low heat (R. Shah, pers. comm., 2010). Since there is no means of identifying whether these gems—like blue zircon—have been heated, we recommend that all zircon of this color range be considered as heated.

Cultured Pearls. Although not widely recognized, heat is sometimes used to alter the appearance of cultured pearls. Heat alone usually produces more saturated yellow colors, and other effects can result when heat is used in combination with other methods ("Better techniques improve brown pearls," 2006) such as bleaching. In all cases, detecting heat treatment can be challenging. There are no obvious thermally enhanced inclusions as in some gems, and the only useful methods determined to date usually involve UV fluorescence reactions and UV-Vis-NIR spectroscopy (Elen, 2001; Wade, 2002).

DIFFUSION TREATMENT

Diffusion treatment was more problematic for colored stones than any other enhancement in the 2000s. Beryllium diffusion, in particular, "upped the bar" on the sophistication of equipment and level of knowledge needed by gem laboratories.

Corundum. Titanium diffusion of sapphire continued throughout the decade, with one instance reported of these stones sold in Australia as heat-treated Ceylon sapphire ("Fusion treated sapphire alert," 2001). Little changed with this method, and its identification remains the same—color concentration along facet junctions, facet-related color, high relief in immersion, and the like (Kane et al., 1990).

Chromium diffusion of corundum has been debated as being more of a chemical reaction at the surface of the stone than true diffusion. It was actually shown on some stones to be a synthetic ruby overgrowth (Smith, 2002). This treatment is very difficult to perform, and to the authors' knowledge is not currently being used.

The diffusion of corundum using cobalt was also reported in the last decade (Kennedy, 2001; McClure, 2002b), but this material was easily identified with magnification and diffused light by a very shallow color layer that showed spotty coloration, as well as observation of a cobalt spectrum with a desk-model spectroscope.

The first serious diffusion challenge started in 2001, when large numbers of pinkish orange ("padparadscha") sapphires showed up in certain markets (Genis, 2003). The color was attributed to a new form of heat treatment done in Thailand. Some labs in Japan are said to have issued over 25,000 reports stating just that (Genis, 2003; Weldon, 2003). In early 2002, however, examination with the stones immersed in methylene iodide revealed that they had a surface conformal layer of orange color surrounding a pink core (Weldon, 2002; figure 9). With this discovery, the illusion that the color was caused by "standard" heat treatment began to crumble ("Orange crush," 2002).

The story is well documented by Emmett et al. (2003). At first, the reason for the orange surface-related color zone could not be determined. The standard equipment available in gemological laboratories detected nothing unusual (McClure et al., 2002). At the February 2002 Tucson shows, however, it was announced that the culprit was beryllium ("GIA-GTL suspects beryllium causes orange colour in treated pink sapphires," 2002; Hughes, 2002; Genis, 2003). Unfortunately, beryllium was almost unknown in corundum, with very little information available in the literature.

There were two major differences between Ti and Be diffusion. First, beryllium, being a very small atom, was capable of diffusing all the way through even large sapphires. Titanium could not do this, even with heating times lasting several weeks. Second, titanium is only capable of creating blue color in sapphire. Beryllium, however, can affect virtually every color of corundum in some way when combined with Fe (figure 10). Colorless, light yellow, or light blue can be turned to intense yellow (see, e.g., figure 11); pink can be altered to orange or padparadscha color; dark brownish red to bright red; and dark inky blue to lighter blue—just to name some of the possibilities (Coldham, 2002; Henricus, 2002; Moses et al., 2002).

Figure 9. A shallow surface-related orange rim around a pink core is diagnostic of some Be-diffused sapphires. Photomicrograph by S. F. McClure; magnified 10×.

Identifying this treatment turned out to be complicated. Severely heat-damaged inclusions were found in many of these treated stones (Roskin, 2003a; Schmetzer and Schwarz, 2005), but they only indicate that the stone was treated at extreme temperatures—they do not prove the presence of Be (Emmett et al., 2003). After a time, we started to see Be-diffused blue sapphires treated by an even newer method that showed no surface-related characteristics and created unusual inclusions (figure 12; Choudhary, 2006; Kitawaki and Abduriyim, 2006; Roskin, 2006; DuToit et al., 2009). These inclusions also did not prove Be treatment, but they strongly indicated that further testing was needed.

Areas of synthetic corundum overgrowth were commonly seen on Be-diffused faceted stones, but Be was not necessary for this to happen (McClure, 2002a). UV fluorescence was helpful in some situations, but not all (Fritsch et al., 2003). Even chemical analysis was a problem, as the standard instruments used at gemological laboratories and most universities (EDXRF and electron microprobe) cannot detect light elements such as beryllium. Detecting Be meant using instrumentation such as mass spectrometers. At that time, no gemological laboratory

Figure 10. Almost every color of corundum can be affected in some way by beryllium diffusion, as illustrated by this large group of Be-diffused rough and faceted stones (1.03–8.53 ct). Photo by Robert Weldon.

possessed this capability, so testing had to be done at commercial laboratories, which is very expensive. Today, several gem labs have this equipment in-house and offer Be testing as a service.

Feldspar. In 2002, a transparent red feldspar colored by copper debuted on the market, reportedly originating from the Congo. This did not raise suspicion initially, as natural red feldspar colored by copper was already well known (from Oregon). Over time, however, the supposed location of this feldspar mine kept changing—to "China," "Inner Mongolia," and then "Tibet." Although most of the feldspar was red, some green material also entered the market (e.g., figure 13).

The first question raised about this material had nothing to do with treatment, but focused on nomenclature (Krzemnicki, 2004a): Was it labradorite or could it be called andesine? Andesine was rare in gem quality, so this could be very valuable to marketing efforts. Although much of the material was indeed andesine, in time this became a secondary issue. Large amounts of this feldspar were being sold as all-natural, untreated material. In July 2008, however, Masashi Furuya of the Japan Germany Gemmological Laboratory reported that he had direct evidence (from experiments done in Thailand) that this feldspar was being diffusion treated by a three-step process that took months to complete (Furuya, 2008). He also mentioned the same type of material being diffused in China by an unknown process.

Other reports suggested that the unusual color zoning found in this material indicated diffusion treatment (Fritsch et al., 2008). Subsequent studies conflicted with this idea, showing natural Oregon material with very similar zoning (McClure, 2009).

To address the controversy, systematic experiments were undertaken to diffusion treat plagioclase. They showed it was surprisingly easy to duplicate the Cu-diffusion process in only a few days (Roskin, 2008; Emmett and Douthit, 2009). Also, gemologists visited a mine in China's Inner Mongolia that produced andesine-labradorite, but

Figure 11. As these before-and-after images illustrate, beryllium diffusion can produce profound color alterations in corundum, here turning 1–2 ct light blue sapphires to intense yellow. Photos by Maha Tannous.

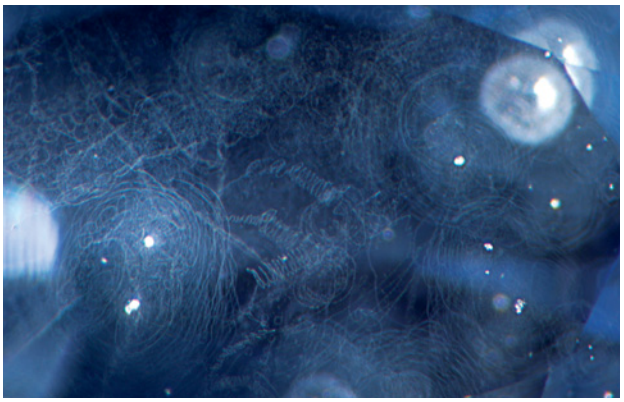


Figure 12. Later developments in Be diffusion of blue sapphires created some very unusual inclusions. Photomicrograph by S. F. McClure; field of view 2.0 mm.

only with a pale yellow color (Abduriyim, 2008). The material could not be simply heated to red or green because it contained virtually no copper (Thirangoon, 2009). This fact left diffusion as the only possible treatment method for these stones.

Claims of a mine in Tibet began in 2005, but their credibility was questionable. In 2008, a team visited a mine in Tibet, collected samples, witnessed mining, and documented red andesine that appeared to be *in situ* (Abduriyim, 2008). However, the samples collected proved to be virtually identical to the diffused Inner Mongolian red andesine, calling the mine into question again. Its authenticity is still not resolved.

With the controversy surrounding this material, identification of this feldspar as treated is still problematic using standard gemological techniques, primarily because the issue of the Tibet mine is not resolved. Color zoning may be useful. A complete "bull's-eye" color zoning with red-inside-green usually means the stone is natural, while green-inside-red may indicate treatment (McClure, 2009). However, if you have a partial "bull's-eye" or merely zoned areas, this criterion is unreliable. To date, larger platelets of copper have been found only in the natural Oregon material (McClure, 2009; Rossman, 2009, 2010). However, separation of Oregon and Chinese feldspar in a gemological laboratory is not difficult as they are all distinct chemically.

Topaz. Blue-to-green topaz surface-treated with cobalt was marketed in the 2000s as an alternative to irradiated blue topaz (Federman, 2007a), a tactic that took advantage of the public's fear of radiation. This material has long been marked as "diffusion treated," even though this claim was never truly substantiated. Gabasch et al. (2008) determined that the layer of coloration was "diffusion induced," creating new phases at the surface. This is not so different from the opinions put forth in the late '90s that the treatment was more of a chemical reaction than diffusion.

Several companies announced lines of "diffusion treated topaz" in new colors of red and pink to "champagne" and bicolors (Roskin, 2003b; "Diffused topaz from India," 2003), but questions still exist as to whether they are from a diffusion or coating process.

Identification of this material is fairly easy. With magnification, the color has a spotty appearance and, due to the extremely thin nature of the color layer, any small chips or abrasions will show the colorless nature of the base topaz.

Other Materials. The discovery that diffusion treatment of feldspar was possible generated claims that many other gems—such as Cu-bearing tourmaline from Mozambique, Imperial topaz, and tsavorite (Federman, 2009)—were also being diffusion treated. To date, no significant scientific data have been presented to support these claims, though experiments have begun to explore some of these possibilities (Saeseaw et al., 2009a).

There was one report of tanzanite possibly being diffused, but examination of the suspect stones showed no evidence of diffusion (Wang, 2003).

CLARITY ENHANCEMENT

During the 1990s, clarity enhancement was one of the jewelry trade's most formidable challenges. Its use

Figure 13. Represented as untreated plagioclase from Tibet, these feldspars weigh 0.45–15.51 ct. Photo by Robert Weldon.

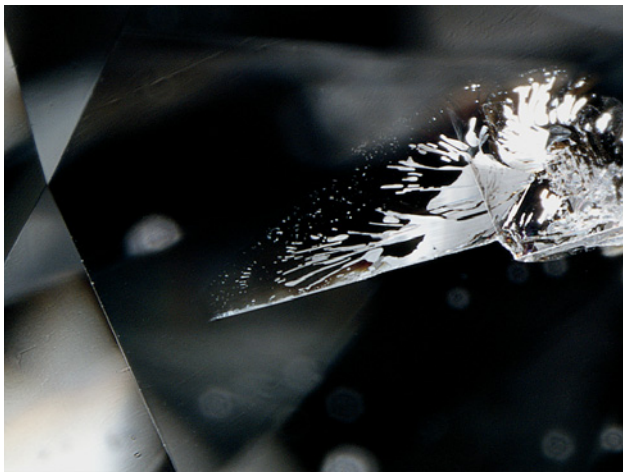


Figure 14. The filler in most clarity-enhanced diamonds is easily damaged by heat, which created the voids seen in this stone. Photomicrograph by S. F. McClure; field of view 2.1 mm.

with emeralds—in what had basically amounted to a trade secret—was touted publicly and almost caused the crash of the emerald market. In addition, the clarity enhancement of diamonds spread rapidly, with improper disclosure causing the ruin of some businesses. Clarity enhancement has remained a serious issue in the 2000s. However, the methods changed and the focus has been on different gem materials.

Diamonds. The biggest concern about the clarity enhancement of diamonds via fracture filling revolved around the durability of the glass filler. Damage due to heat (figure 14) from standard jewelry repair procedures, such as retipping, inevitably caused problems at the retail level. One of the major treaters (Oved) announced in 2000 that they had developed a filler that could withstand such heat (Bates, 2000;"Oved announces. . . ," 2000). However, testing showed that although the new Oved filler material seemed to withstand higher temperatures, it still could be damaged by some jewelry repair procedures, even when performed by a master jeweler (Shigley et al., 2000). Oved instituted a policy of laser inscribing their company name on a bezel facet of all the diamonds they treated so the filled stones could not be misrepresented (Gallagher, 2000).

The practice of laser drilling diamonds to create an opening through which acid could be introduced to remove a dark inclusion had remained unchanged for many years, until a new version was introduced that took advantage of advances in laser technology. Developed in Israel and referred to as the "KM treatment" (short for *kiduah meyuhad*, or "special drill" in Hebrew; Horikawa, 2001), this method did not actually drill a hole into the stones. Instead, it used lasers to create a small fracture from the inclusion to the surface so that the inclusion could be bleached without leaving a tell-tale hole at the surface (figure 15; McClure et al., 2000a). Unfortunately, this treatment entered the market undisclosed, and its fraudulent nature caused the Israel Diamond Bourse to outlaw its use. It continues to be encountered, and is often referred to as "internal laser drilling."

Identification is done with magnification. The laser leaves behind lines or dots of irregular squiggles, with feathers leading from an inclusion to the surface (figure 16). These marks tend to look black in transmitted light, and are usually confined to a feather (McClure et al., 2000a; Cracco and Kaban, 2002; McClure, 2003a). They may be tiny and difficult to find even with a microscope—or large and numerous, easily seen with a loupe.

Other observations were posted in the literature periodically. Among them were changes in flash-effect colors (Cracco and Johnson, 2008), filled fractures in treated-color diamonds (Song et al., 2009; Gelb, 2005), difficult-to-identify damaged fillers (Gelb and Hall, 2005), and fracture filling associated with a pink dye (Yeung and Gelb, 2004).

Ruby. The first report of faceted rubies showing a flash effect similar to that seen in clarity-enhanced diamonds was in 2004 ("Lead-glass impregnated ruby. . . ," 2004). Chemical analysis revealed that these rubies were filled with a high-lead-content glass. Soon other labs reported this treatment ("New treatment on unheated rubies. . . ," 2004; Rockwell and Breeding, 2004; Milisenda et al., 2005).

It became apparent that this treatment was going to be very significant to the industry (Roskin, 2004). The starting material was very low quality, translucent-to-opaque, non-gem rough from Madagascar (Pardieu, 2005). By a process that involved low-temperature heating, cleaning in an acid bath, and then filling with a high-lead-content glass, this non-gem corundum was transformed into transparent, facetable material (figure 17). This made available huge amounts of treated rubies that were usually sold at very low prices.

Identification of these filled rubies was not difficult. Most had so many filled fractures that the flash effect was easy to see with magnification, although the red color of the ruby sometimes partially masked the orange flash (McClure et al., 2006; figure 18). Flattened gas bubbles and high-relief unfilled areas within the fractures were also readily visible with

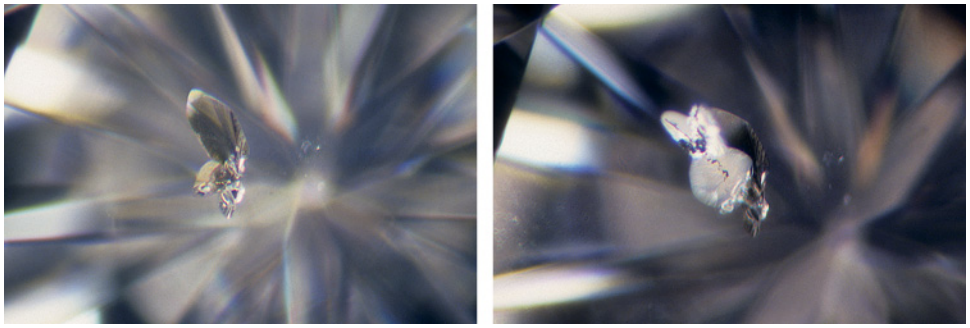


Figure 15. Internal laser drilling can create a feather from a black inclusion to the surface of the diamond, providing a conduit for the acid that will then bleach the inclusion, as shown in these before (left) and after photos. Photomicrograph by S. F. McClure; magnified 40×.

Figure 16. Internal laser drilling can be identified by the characteristic inclusions—such as dots, squiggles, and disk-like marks—it leaves behind. Photomicrographs by S. F. McClure; magnified 25× (left) and 40×.

magnification. However, the use of reflected light to look for differences in surface luster was not very helpful in this case. The luster of this glass was very similar to that of ruby, sometimes even higher (Smith et al., 2005), so it was much more difficult to see than the more typical silica glass fillers.

The filler proved relatively durable to heat (up to ~600ºC), but it was easily etched by even mild acids such as pickling solution (McClure et al, 2006). This etching turned the filler white near the surface, rendering it quite visible.

Also of concern was the decreasing quality of the starting material. We began to see stones where the flash was everywhere, and internal filled cavities containing large spherical gas bubbles were common (Scarratt, 2009).

The nomenclature for this treatment soon became an issue. The early material was referred to as *clarity enhanced* because even though the treatment was fairly extensive, the rubies were mostly solid material that would be expected to stay together even without the treatment. However, some of the later material contained so much glass that it appeared the glass was actually holding the pieces of ruby together. Soaking such stones in hydrofluoric acid to remove the glass resulted in their falling apart along fractures or being reduced to tiny pieces (Scarratt, 2009). Accordingly, GIA developed a three-tiered system, keeping *clarity enhanced* for more solid material, specifying *ruby with glass* for stones that needed the glass to stay together, and using *ruby/glass composite* for those composed of unrelated pieces of ruby floating in glass (Scarratt, 2009; figure 19). The other labs of the LMHC adopted these criteria. American Gemological Laboratories (AGL) and the International Colored Gemstone Association (ICA) have chosen to call all these stones *composite ruby*.

The real problem, however, is the large amount of this material that is being sold without any disclosure. So far, this treatment has even appeared in ruby

Figure 17. Filling ruby with a lead-based glass can change non-gem corundum (on left) to transparent, facetable rubies such as those on the right. The samples (2.13–39.17 ct) are courtesy of Hussain Rezayee and GIA; photo by Robert Weldon.

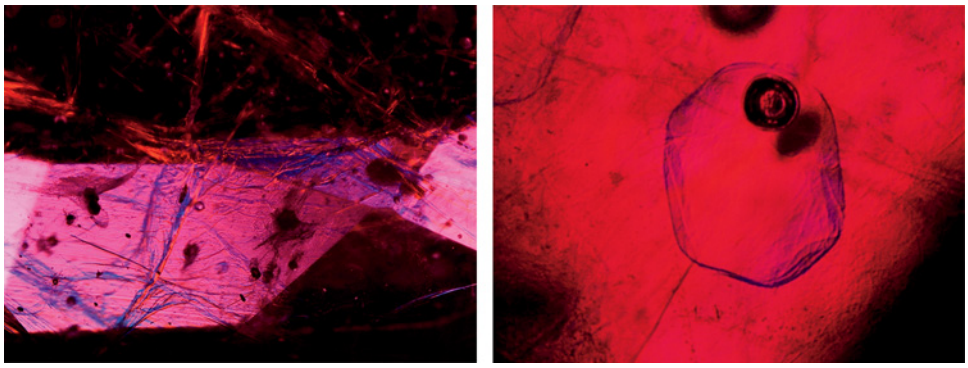


Figure 18. Blue and orange flash colors are the best indication of lead-glass filling, whether they are seen in fractures (left) or in internal cavities that often also have spherical gas bubbles (right, with only the blue flash color visible). Photomicrographs by S. F. McClure; fields of view 2.4 and 1.4 mm respectively.

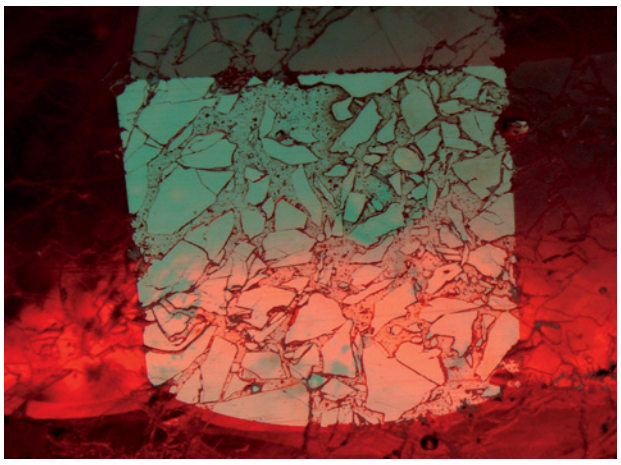
beads (Hänni, 2006a), color-change sapphires (Choudhary, 2008), hollowed-out rubies set in closed-back mountings (Krzemnicki, 2007), and estate jewelry (Quinn Darenius, 2010).

Emerald. The damage caused to the emerald market in the 1990s from lack of disclosure of clarity enhancement slowly began to fade in the 2000s (Gomelsky, 2003). However, the debate over the use of oil versus polymers as filler material continues, and a significant study was done on the durability of fillers (Johnson, 2007).

To address the possibility that a highly fractured stone was masquerading as a much finer one simply because of the treatment, labs started to state the degree of enhancement on their reports (e.g., McClure et al., 2000b). Different systems were developed with anywhere from three to nine categories (Gomelsky, 2001a,b); the most common were three- or four-tiered. Today, degree-of-enhancement calls have become standard procedure for emerald reports from all the major laboratories.

Near the end of the decade, it was reported that some emerald rough was being "stabilized" with hardened polymers, so larger stones could be cut (Roskin, 2007; Federman, 2008). In effect, though, the polymer glues the pieces of emerald together at the fractures (e.g., figure 20), so its removal would result in the stone falling apart (Federman, 2007b). This situation is very similar to that of the lead glass–filled rubies, making disclosure even more important.

Figure 19. Some lead glass–filled rubies have so much glass that they are actually pieces of ruby floating in glass. Such material is more properly called a ruby/glass composite. Photomicrograph by S. F. McClure; field of view 4.1 mm.



Other Materials. Laboratories have reported on many other filled gems. Some of those mentioned in the 2000s include: aquamarine and tourmaline (Wang and Yang, 2008; Deng et al., 2009), andalusite (Fernandes and Choudhary, 2009), fuchsite quartzite (Juchem et al., 2006), hackmanite (Wehr et al., 2009), and iolite (McClure, 2001).

IRRADIATION AND COMBINED TREATMENTS

Intense colors can be induced in many gems by exposing them to various forms of radiation, such as electrons, gamma rays, or neutrons. To remove unwanted color overtones, some irradiated stones are subsequently heated. While the 1980s saw significant experimentation and development in the area of gemstone irradiation, very few new types of irradiated gems appeared on the market during the 1990s and 2000s. Likewise, little progress was made in detection methods.

For many gems, there is no definitive test or series of tests to establish whether they have been irradiated. Even though irradiation has been used for many years to produce intense colors in yellow beryl, pink-to-red tourmaline, and kunzite, these enhancements remain undetectable. The same is true for blue topaz and many other routinely irradiated gem materials.

Blue irradiated (and annealed) topaz generates

more than $1 billion annually in retail sales (Robertson, 2007). The low cost of irradiated blue topaz (typically a few dollars per carat at wholesale) leaves the trade little economic incentive to determine whether or not the gem has been treated. As a result, all blue topaz is assumed to have been irradiated. The same is true for smoky quartz and dark yellow beryl.

Diamond. With the staggering prices realized at auction for some fancy-color diamonds during the last decade (e.g., more than $1 million per carat for some natural-color blue and green diamonds), there is huge incentive to determine whether a diamond's color is natural or irradiated. Large quantities of diamonds continued to be irradiated (often followed by low-temperature annealing at atmospheric pressures) to produce a wide variety of colors—red, orange, yellow, green, blue, violet, and purple—in saturations from light to very dark (see Overton and Shigley, 2008; Shigley, 2008). Many treaters produced small faceted irradiated (and annealed) colored diamonds for use in jewelry.

The most significant developments in diamond irradiation since 2000 were in combination treatments. Both natural and synthetic diamonds are now color enhanced by a process that involves first HPHT annealing, then irradiation, followed by low-temperature heating (likely in that order), to produce several colors, including red, pink, orange, and green (Schmetzer, 2004; Shigley et al., 2004; Wang et al., 2005a; Wang and Johnson, 2010b; Wang et al., 2010). Identification generally requires measurement in a laboratory of the absorption and/or photoluminescence spectral features present with the diamond cooled to a low temperature, although in some cases standard gemological testing can also offer clues (see e.g., Shigley, 2008). Other combinations also exist, such as irradiated and glass-filled diamonds (Gelb, 2005; Gelb and Hall, 2005).

Topaz. The potential enforcement of Nuclear Regulatory Commission (NRC) guidelines on irradiated gems (American Gem Trade Association, 2007) caused great concern in the first decade of the 2000s. Since 1986, NRC regulations have stated that any neutron-irradiated gemstone produced in or imported into the U.S. must be tested for residual radiation by an NRC-licensed testing facility (Nuclear Regulatory Commission, 1986; Ashbaugh, 1988). Whereas considerable amounts of blue topaz were once treated in the U.S.—and then properly tested for radioactivity and held until the radioactivity subsided—nearly all treated blue topaz entering the market since the latter half of the decade has been irradiated and annealed in other countries, some of which may not restrict the export of "hot" material.

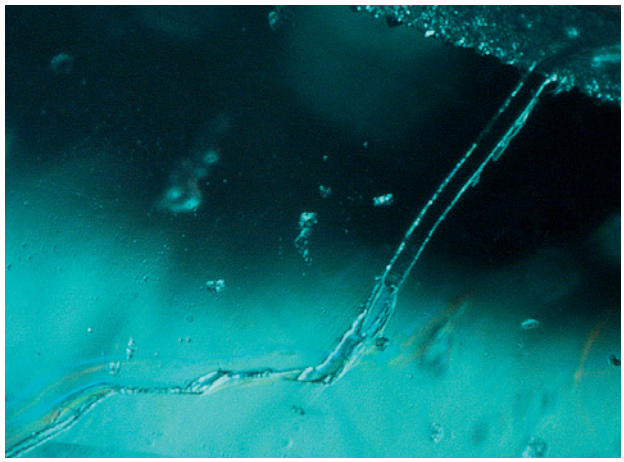


Figure 20. In some emeralds, as with some rubies, the filler material may actually be holding the stone together. Note in this emerald that the resin is binding material that otherwise would be broken by the large, wide fractures.

Amid the confusion generated by this issue, several major retail chains and department stores stopped selling blue topaz. After receiving numerous trade and public inquiries regarding blue topaz, the NRC issued a fact sheet on irradiated gemstones (United States Nuclear Regulatory Commission, 2008). To further address the issue, the Jewelers Vigilance Committee (JVC) and American Gem Trade Association (AGTA) published a 2008 brochure titled "The Essential Guide to the U.S. Trade in Irradiated Gemstones."

To our knowledge, the NRC has still not enforced its regulations, and neutron-irradiated blue topaz continues to be imported and sold in the U.S. However, no blue topaz containing residual radioactivity has been reported recently in the trade.

Earlier—around 2000—Europe faced similar concerns that irradiated blue topaz exhibiting residual radioactivity had made its way into several different countries (Kennedy et al., 2000).

U.S. Postal Service Irradiation. During the anthrax scare of late 2001, the USPS irradiated envelopes and packages to kill potential biological agents. The company that the postal service contracted with to perform the test, SureBeam, used a linear accelerator to create a beam of high-energy electrons. The potential impact of this exposure was immediately recognized, since the same ionizing radiation is routinely used to change the color in several types of

gems. McClure et al. (2001) showed alarming evidence of several gems that had their color changed dramatically after being exposed in SureBeam's facility to the same dosage as was used for the mail. The USPS subsequently abandoned these procedures, after determining that the time and money needed to sanitize all mail would be prohibitive.

Green Quartz. In the latter part of the decade, an unusual amount of faceted green quartz suddenly appeared on the world market. Nearly all these gems—which originated from Rio Grande do Sul, Brazil—began as colorless to light yellow quartz that was subsequently irradiated to produce the green color (Kitawaki, 2006; Schultz-Güttler et al., 2008). Natural green quartz does exist but is extremely rare, and "greened quartz" (also known as prasiolite) is produced by heating certain types of amethyst. Irradiated green quartz shows a broad spectral absorption at 592–620 nm, while prasiolite exhibits a broad band centered at 720 nm. When examined under a Chelsea filter with incandescent light, irradiated green quartz appears red and prasiolite appears green (Schultz-Güttler et al., 2008; Henn and Schultz-Güttler, 2009).

Beryl. In addition to the huge quantities of irradiated yellow beryl, which remains undetectable, irradiated yellowish green beryls were seen. Milisenda (2007a) reported absorption lines between 500 and 750 nm for the ordinary ray, which are also typically seen in artificially irradiated "Maxixe-type" beryls. Milisenda (2007b) reported a beryl with "Maxixe-type" spectra that was offered for sale as a cat's-eye scapolite but proved to be a blue irradiated cat's-eye beryl.

Hiddenite. Milisenda (2005a) reported on a parcel of intense green faceted spodumenes from Pakistan, offered for sale in Idar-Oberstein as hiddenite, that were artificially irradiated. The stones revealed a broad absorption band centered at 635 nm. As expected for this material, the color faded to the original pale pink within a few days.

Pearls. The irradiation of pearls has been known for decades, and little has changed since 2000. The treatment is almost always associated with freshwater pearls or nuclei, since the radiation appears to alter the state of the trace element manganese found in these materials. Gray, silvery gray, and black colors have all been produced. In fact, pearls were one of the gems significantly altered by the U.S. postal service irradiation mentioned above. Detection remains a challenge in some cases, and research has continued on its identification (Liping and Zhonghui, 2002).

SURFACE COATING

As it has been for centuries, applying surface coatings to change the color of gems continues to be a common practice. Not only do gemologists need to be aware of high-tech coatings, we must also remember to look for older, simpler alterations.

Diamonds. Just as Miles (1964) described decades ago, in 2003 Sheby reported seeing two slightly yellow diamonds that were coated with a blue material to improve the apparent color. Also as a recent reminder, Eaton-Magaña (2010) described a 1.5 ct diamond with a color equivalent to Fancy pink that revealed a nearly imperceptible trace of reddish material on a natural when viewed with the microscope. After cleaning, the diamond was graded Faint pink.

Sputter-coated optical thin films were originally developed in the 1940s to improve the optical performance of lenses. We continued to see similar coating technology used on diamonds in the 2000s. Evans et al. (2005) and Wang et al. (2006b) reported on faceted diamonds that were colored pink by sputter-coated thin films. A potentially new kind of diamond coating was described by Epelboym et al. (2006)—rather than using the fluoride coatings previously known, pink and orange-treated diamonds were suspected of being coated with a silica film doped with gold.

Shen et al. (2007) reported that the trade was submitting greater numbers of pink diamonds coated by calcium fluoride (CaF_2) to the GIA Laboratory for grading and origin reports. They also described Serenity Technologies' use of multiple micro-thin coatings of various compositions to produce a variety of colors on diamonds, including intense blue, green, yellow, and orange to pink to purple-pink (figure 21).

We continue to see crude yet effective colored coatings applied to the girdle facets of diamonds with permanent markers and solutions made from colored art pencils.

Diamond-Like Carbon Thin Films. Super-hard coatings, such as diamond-like carbon (DLC) films, are becoming increasingly popular for a variety of mechanical, scientific, and technological applications, such as cutting tools, razor blades, and the like. This technology is also making its way into the gem industry. Several companies, including Serenity Technologies and Zirconmania, market DLC-coated cubic zirconia. Eaton-Magaña and Chadwick (2009) reported that these products were easily separated from diamond.

Serenity Technologies also offers a "patent pending nanocrystalline diamond coating process" named "Diamond Rx" which they apply to a variety of gems, including emerald, apatite, chrome diopside, zircon, peridot, tourmaline, kunzite, tanzanite and aquamarine (Serenity Technologies, 2010). They maintain that such coatings are extremely durable. However, it is very difficult (and sometimes impossible) to identify whether these DLC coatings are in fact even present on a gemstone.

Tanzanite. In April 2008, a Los Angeles gem dealer encountered two parcels comprising a few hundred color-coated tanzanites (E. Caplan, pers. comm., 2010; figure 22). Research concluded that the smaller stones (4.5 mm) could be identified on the basis of unusually intense color for their size, by areas of wear seen with microscopic examination, and by unusual surface iridescence ("American Gemological Laboratories identifies. . . ," 2008; McClure and Shen, 2008). Larger stones (e.g., 3+ ct) were much more difficult to identify with magnification, but EDXRF and LA-ICP-MS analyses revealed Co, Zn, Sn, and Pb in the coating (McClure and Shen, 2008). Since their initial sighting, coated tanzanites have all but disappeared from the market.

Topaz. In the late 1990s, we began to see different colors of topaz (blue-to-green, orange, pink, and red) that were being represented as "diffused" (Fenelle, 1999; McClure and Smith, 2000). Schmetzer (2006, 2008) reviewed the patent literature and concluded that the various mechanisms and treatment methods were not diffusion and should all be described as "surface coated." However, Gabasch et al. (2008) showed that certain colors were due to coatings, whereas others were diffusion-induced. For more details, see the section on topaz under "Diffusion Treatment" above.

Coral. Typically, gem coatings are ultra-thin. However, Hänni (2004) described black coral (also known as horn coral) that was coated with several relatively thick layers of artificial resin.

Pearls. Any gem can be coated to alter its color, provide a degree of protection, improve the luster, or mask some imperfection. Pearls usually fall into the latter three categories. Porous by nature, pearls may be coated for protection from harmful chemicals. Or they may have luster and/or surface imperfections that a coating can hide. In this decade, a number of coatings were applied to natural and cultured pearls (Moses and Reinitz, 2000; Hurwit, 2002; Krzemnicki, 2005a; Shor, 2007). One development in particular that should be carefully monitored by the pearl industry in the future is the application of DLC coatings (Drucker, 2008) to improve durability.

Figure 21. New coatings can turn diamonds virtually any color, as illustrated by these ~0.40 ct coated diamonds. Courtesy of Serenity Technologies; photo by C. D. Mengason.

DYEING

Although it dates back to the time of Pliny (23–79 AD), dyeing continues to be seen in nearly every gem material that is porous or has surface-reaching fractures. Careful microscopic examination will frequently reveal the presence of dye in cracks and around grain boundaries. In a number of porous materials, rubbing the surface with a cotton swab soaked in acetone or a 10% hydrochloric acid solution can identify the presence of dye. In others, absorption spectra can provide proof of dyeing.

Pearls. Dye continues to be used to improve the appearance of lower-quality natural and cultured

Figure 22. Tanzanite coated with a cobalt-colored material was of great concern for a brief time. All of these tanzanites (0.50–3.01 ct) were coated, but the one on the lower left was repolished to give an idea of the original (paler) color. Courtesy of Fine Gems International; photo by Roger Mathis, ©Robert E. Kane.

Figure 23. Dyed "golden" pearls, such as the 11.4–14.0 mm cultured pearls shown here, were only one of the identification challenges related to pearls in the last decade. Photo by Robert Weldon.

pearls (Hurwit, 2001; Overton and Elen, 2004; Wentzell, 2005). While the majority of dyed pearls are nacreous, dye may also be used to make non-nacreous pearl imitations more convincing, such as those mimicking Melo pearls (Wentzell, 2006). Of ongoing concern since the late 1990s is the detection of dyed "golden" cultured pearls (figure 23; "Concerns raised. . . ," 2003; Liu and Liping, 2007). Some samples present identification challenges, requiring the use of chemical analysis to detect trace elements such as iodine. Other developments involve the use of additional whitening compounds in freshwater non-beaded cultured pearls (Shouguo and Lingyun, 2001) and the use of metallic dyes injected into pearl sacs ("Pre-harvest colour-treated Akoya unveiled," 2008; Coeroli, 2010). A form of dyeing marketed as "lasering" has also been reported. This is said to produce dark "peacock" green or dark purple colors (Liping, 2002).

Other Gem Materials. Several other dyed gem materials were encountered during the decade. Blue and green diamond crystals were found to owe their color to dyeing (Van der Bogert, 2005). Quartzite was dyed red to imitate ruby (Mayerson, 2003a), whereas green dye was found in quartzite to resemble emerald (Milisenda, 2003). Mayerson (2003b) described an effective simulant for high-quality jadeite: a tricolored (lavender, green, and orange) dyed and polymer-impregnated quartzite bangle bracelet. Tan et al. (2006) used light-induced autofluorescence spectroscopy to identify dyed polymer-impregnated jadeite. Of particular interest was dyed jadeite found to resemble nephrite jade (Mayerson, 2004).

Low-quality red and blue corundum were found to have been dyed (Milisenda, 2004). A parcel of faceted "rubies" purchased in Afghanistan was identified by Milisenda (2005b) as dyed sillimanite. Dyed blue carbonate minerals, such as magnesite and dolomite, were sold as turquoise ("Some dyed minerals. . . ," 2000). To imitate common opal from the Peruvian Andes, marble was dyed pink and fashioned into beads (Milisenda, 2006). Raman and IR spectra identified dyed black chalcedony in an attractive pendant set with diamonds and pearls (DeGhionno and Owens, 2003). A copper-based dye was detected with UV-Vis-NIR spectroscopy in a natural-appearing chalcedony bead (Inns, 2007a).

BLEACHING

Bleaching is a process that uses agents such as acids or hydrogen peroxide to remove unwanted color from a gem. Only a limited number of materials will respond to such treatment.

Jadeite. Jadeite with brown staining caused by natural iron compounds is often bleached with acid. This treatment started in the 1990s and was categorized in the impregnation section of McClure and Smith (2000). This is because jadeite treated in this manner must be impregnated with polymers, as the acid damages the structure, making it very susceptible to breakage.

This treatment has become commonplace in the jadeite market. However, the bleaching itself typically cannot be detected, only the polymers used for impregnation (Sun, 2001; Fan et al., 2007). The treatment is now being used on nephrite jade as well (Jianjun, 2005).

Pearls. Bleaching is considered an "acceptable" pearl treatment due to the difficulty of proving a pearl's exposure to chemicals such as hydrogen peroxide. All types of pearls are routinely bleached: natural, bead cultured, and non-bead cultured. Akoya cultured pearls continue to be routinely bleached and "pinked" (Roskin, 2002b). Bleaching is also known to be a major component of the proprietary process used to produce the "chocolate" cultured pearls (figure 24) that entered the market during the decade (Zachovay, 2005; Hänni, 2006b; Wang et al., 2006c; Federman, 2007c).

Other Materials. While there are undoubtedly addi-

Figure 24. It is believed that most of the "chocolate" cultured pearls on the marketplace in the last decade were originally black cultured pearls that were treated by a process that involved bleaching to achieve this color. This strand (12.0–13.7 mm) is courtesy of Emiko Pearls International; photo by Robert Weldon.

tional porous materials that could be bleached, the only other one we could find reference to is coral. Black coral is bleached to "golden" coral, which is easily identified by its distinctive structure (Weldon, 2003).

IMPREGNATION

Impregnation of aggregate stones and other porous materials was seen more often in the first decade of the 2000s. This is largely due to increased demand for inexpensive stones, a phenomenon primarily driven by television shopping networks. The practice now extends to some unusual materials as well. A number of the gems were only usable in jewelry when they were treated by impregnation (often referred to as "stabilization").

Jadeite. The polymer impregnation of jadeite following the bleaching process described above was common during the last decade and will likely remain so in the future. At least one new analytical method was reported to detect this treatment (Liu et al., 2009), but its identification is still usually done with IR spectroscopy.

Nephrite. Nephrite was reported to have been polymer impregnated after bleaching with the intent of imitating "Hetian white" nephrite (Jianjun, 2005). It, too, can be positively identified by IR spectroscopy.

Turquoise. The greater demand for turquoise (a favorite of TV shopping networks) led to the use of more lower-quality impregnated material. Sometimes the treatment is so extensive that the material is actually a composite (figure 25), and gemological properties such as SG and RI no longer match turquoise (Choudhary, 2010; McClure and Owens, 2010). Materials used for impregnating turquoise include wax and hardened polymers. A UV-hardened polymer was identified as a filler for the first time using Raman spectroscopy (Moe et al., 2007).

Identification of this treatment is still mostly accomplished via IR spectroscopy (Henn and Milisenda, 2005; Chen et al., 2006), although many examples show veins and cavities filled with polymers that are visible with magnification.

Late in the decade, a product marketed as "Eljen" turquoise was claimed to be treated by a new proprietary process that improved the hardness and polish of soft porous turquoise. Testing showed it to be impregnated with a polymer, but it did seem harder than most impregnated turquoise, which would account for the improved polish (Owens and Magaña, 2009).

Opal. Natural opal—a hydrous, porous material—has a tendency to dry out and crack spontaneously. This tendency is so strong in opal from some deposits that most of the material is not usable in jewelry (e.g., Virgin Valley, Nevada). To address this problem, two new treatments were reported in the 2000s: (1) oil or wax impregnation of Mexican fire opal (Gambhir, 2001); and (2) a drying-out process followed by impregnation with a silica compound, used on Ethiopian opals (Filin and Puzynin, 2009).

Other Materials. As mentioned at the beginning of this section, impregnation was used on a number of more unusual materials during the decade. These include quartzite (Kitawaki, 2002; Juchem et al., 2006), seraphinite (Henn, 2008), and sillimanite (Singbamroong, 2005). It even extended to some manufactured materials, most notably a much-

Figure 25. The demand for turquoise is so strong that low-quality material is being treated by polymer impregnation and pressed into a composite material to make it salable. The treatment is sometimes so extensive that the gemological properties are altered. These carvings, 3.0–6.0 cm wide, are courtesy of Silver Express; photo by Robert Weldon.

debated material from Mexico called "Rainbow Calsilica" (Kiefert et al., 2002). This material required impregnation with polymers to be useful in jewelry, as it was very porous and would not take a polish in its original state (Kiefert et al., 2002; Frazier and Frazier, 2004).

LUSTER ENHANCEMENT

This term is sometimes used to describe a treatment common to jade and some other gem materials in which a substance such as wax is rubbed on the surface of the stone to improve its appearance. The wax is only present on the surface and in depressions such as grooves in carvings, so it is not considered an impregnation. Although such substances are sometimes applied to pearls (Petersen, 2000), *luster enhancement* of pearls typically has a somewhat different meaning.

In the cultured pearl industry, the name *Maeshori* is associated with this kind of treatment (Akamatsu, 2007; Shor, 2007). Developed in the 2000s to improve the prepolishing process, it involves the use of solvents to "clean" nacreous pearls and hence produce a more lustrous surface. Various other forms of this treatment also exist (Lingyun et al., 2007). Polishing continues to be used on all types of nacreous and non-nacreous pearls to improve their salability. It takes place at all steps of the supply chain (Pousse, 2001), starting with the farmers, who often tumble their cultured pearls with walnut chips (N. Paspaley, pers. comm., 2008) and/or other materials and then polish them.

CONCLUSIONS

The first decade of the 2000s brought many new, unanticipated enhancements. Some of these—such as HPHT treatment and beryllium diffusion of corundum—usually cannot be identified by gemologists with standard equipment. In most cases, stones that might be treated by these methods must be sent to a well-equipped gemological laboratory to get a conclusive identification. Still, today's gemologist can benefit by developing their ability to recognize when a stone shows evidence it has *not* been treated (particularly for rubies and sapphires) and also recognizing when they cannot tell and the stone must be sent for further testing.

It is interesting that in their retrospective of the 1990s article, McClure and Smith (2000) predicted that new filling processes would bring clarity enhancement to ruby, sapphire, and alexandrite. Three years later, at least part of this prediction came true with the development of a lead-glass filler for ruby. There is every reason to believe that this treatment, or a similar one, will soon extend to other relatively high RI materials.

Already in 2010 we have seen several new developments, including lead-glass filling of star rubies (Pardieu et al., 2010a) and a combination treatment of rubies from Mozambique that includes partial healing of fractures and partial filling with a glass that does not contain lead (Pardieu et al., 2010b).

With these developments, disclosure has become a significant topic at every trade show and gemological conference. As the trade discovered with emerald fillers (and the impact of nondisclosure on emerald sales) in the '90s, they neglect this subject at their peril. Consensus is critical. Discovering a treatment exists and developing identification criteria are an important start, but the trade and gemological community must work together to address the issues of what to call a treated material, how to disclose it, and how to make sure it gets disclosed. Important steps in this direction have been made, but more are needed.

McClure and Smith (2000) also predicted—correctly—that technology would advance at an even faster rate during the next decade. This will undoubtedly be the case from now on, making the unforeseen the norm in the gemological world as it is in the world at large.

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Key to abbreviations: *Australian Gemmologist* = *AusG*; *Canadian Gemmologist* = *CanG*; *Colored Stone* = *CS*; *Diamond and Related Materials* = *DRM*; *Gemmology* = *Gem.*; *Gems & Gemology* = *G&G* (Gem News = GN, GNI = Gem News International, Lab Notes = LN); *Jewelers' Circular-Keystone* = *JCK*; *Jewellery News Asia* = *JNA*; *Journal of Gemmology* = *JofG*; *Journal of the Gemmological Association of Hong Kong* = *JofGemHK*; *Journal of Gems & Gemmology* = *JofG&G*; *Lapidary Journal* = *LJ*; *Modern Jeweler* = *MJ*; *National Jeweler* = *NJ*; *Professional Jeweler* = *PJ*; *Rapaport Diamond Report* = *RDR*; *Revue de Gemmologie a.f.g.* = *Rev. de Gem.*; *(Gemmologie:) Zeitschrift der Deutschen Gemmologischen Gesellschaft* = *(Gem:) Z. Dt. Gemmol. Ges.*

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