SERENITY COATED COLORED DIAMONDS: DETECTION AND DURABILITY

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Gemological, spectroscopic, and chemical properties of diamonds treated using the new Serenity Technologies coating technique for inducing various “fancy” colors are reported. This technique produces colors that include intense blue, green, yellow, and orange to pink to purple-pink. The presence of a coating can be identified with magnification by observation of an interference-related colored (often bronzy) film, scratches, and colorless and/or dark areas on the surface of the pavilion facets. UV-Vis absorption spectra and chemical analysis provide confirmation of the treatment. Chemical analysis also revealed that the coating is a SiO_2 film doped with Au and/or Ag for blue, pink, and yellow coloration, or with a surface Fe_2O_3 film for orange. No evidence was detected of either chemical diffusion or implantation of foreign elements into the diamond. While the colors produced by this treatment are stable to some standard jewelry repair and cleaning procedures, they are not considered permanent.

The importance of fancy-color diamonds in the marketplace, and the high price-per-carat values due to their rarity, have created a market niche for treated colored diamonds that are moderately priced and more readily available. In this article, we describe a new type of treated diamond that is surface-coated with a thin layer of foreign material to create the apparent color (figure 1). This treatment is carried out on a commercial basis by Serenity Technologies Inc. of Temecula, California. Serenity cooperated with GIA to treat several diamonds, which were documented both before and after the treatment process. GIA also examined more than 100 diamonds treated by this method that were obtained in the marketplace. Some features of this type of coated diamond were recently reported (see Epelboym et al., 2006; Wang et al., 2006a; Kitawaki, 2007; Moses et al., 2007). The present article expands our understanding of this material by detailing the gemological and spectroscopic features of the coated diamonds, the nature of the coatings used, and the durability of the coatings during standard conditions of jewelry repair, care, and wear.

BACKGROUND

For centuries, merchants have used various forms of coating, dyeing, or “painting” to add a thin layer of colored foreign material to part or all of a gemstone’s surface with the intent of masking an underlying bodycolor or creating a more desirable color appearance. In 1950, Edward Gübelin discussed the physics related to several possible newer gemstone coatings, including CaF_2 and SiO_2 (Gübelin, 1950). One of the first reported examples of this treatment in modern history was a blue-coated diamond briefly described by Crowningshield (1959). Because of the apparent proliferation of coated diamonds at that time, several practical methods were developed to aid gemologists (Miles, 1962, 1964). In these articles, Eunice Miles described the detection of such coatings by careful examination with a binocular

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See end of article for About the Authors and Acknowledgments.

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Figure 1. A variety of colors can be produced on polished diamonds by a new coating technique from Serenity Technologies, Inc. These color-treated diamonds are now being sold commercially in the jewelry market. The stones shown here range from 0.01 to 0.70 ct. Composite photo by Jessica Arditi and Jian Xin (Jae) Liao.

microscope. She used a combination of reflected and diffused transmitted light to examine the facets for iridescent colors, scratches, a spotty or blotchy appearance to the color, or areas where the color had been removed (e.g., along facet junctions). Coated diamonds continued to be reported periodically in the Lab Notes column of *Gems & Gemology* over the next several decades (see, e.g., Crowningshield, 1965; Liddicoat, 1966; Fryer, 1983; Hargett, 1989; Sheby, 2003).

The coating materials used in the past varied in adhesion, durability, and extent of coated area on the diamond's surface. Although we do not know for certain, we believe that a variety of coating materials were used. These older color-treatment processes may seem unsophisticated or outdated in comparison to permanent techniques such as irradiation and high-pressure, high-temperature (HPHT) annealing, which are most prevalent today. However, recent technological advances could have significant implications for the application of coatings onto diamonds with respect to their color, thickness, and durability. This "modernization" of coating materials and application technologies was evident in diamonds recently identified as having a thin coating of calcium fluoride (CaF_2) doped with gold to produce a pink color (Evans et al., 2005; Wang et al., 2006b).

The number of coated diamonds being submitted to the GIA Laboratory has increased dramatically in recent months. Most of these diamonds have CaF_2 coatings and appear pink and strongly saturated, but others are much lighter in tone (e.g., figure 2). These lighter coated diamonds have proved to be a challenge for even our most experienced gemologists, as the interference colors associated with the coating are very subdued and areas (such as facet junctions) where the film might have been polished off are not readily discernable. Therefore, the presence of the CaF_2 coating can be missed if careful examination protocols are not followed.

Figure 2. The light pink coloration of these diamonds (each about 1 ct) is created by a thin coating of calcium fluoride doped with gold. This gives a convincing resemblance to the color of some natural pink diamonds and creates a challenge in gem identification. Photo by C. D. Mengason.

Figure 3. These six diamonds (0.34–0.40 ct) were colorless or near-colorless before being subjected to a new coating process to create their gray-violet, yellow, purplish pink, and pink-to-orange color appearances. Additional information is provided in table 1. Composite photo by Jian Xin (Jae) Liao.

Consequently, when we learned that a new source was introducing large quantities of coated diamonds with various colors into the marketplace, we undertook the following investigation into the characterization and identification of this treated material. We also studied the durability of these coated diamonds to standard jewelry manufacturing, repair, and cleaning procedures, as well as to household products they might come into contact with during consumer wear.

MATERIALS AND METHODS

Materials. A total of 102 coated diamonds were obtained from dealers who had their diamonds treated by Serenity Technologies (see, e.g., figure 1). The hues of these diamonds included blue (16 samples), green (16), orange (16), pink (20), purple-pink (18), and yellow (16). The green and yellow diamonds were all round brilliant cuts with weights of 0.01–0.03 ct. No larger coated diamonds with these colors were available in the market during this study. The other colors ranged from 0.03 to 0.70 ct and were faceted into various styles. According to the treater, there is no limitation on the size of diamond that can be coated by this process (J. Neogi, pers. comm., 2007). In addition, Serenity Technologies agreed to treat six colorless to near-colorless natural diamonds that GIA supplied (0.34–0.40 ct; see figure 3 and table 1), which allowed us to examine their gemological and spectroscopic properties before and after treatment.

Gemological Examination. All samples were observed with a gemological microscope and various lighting conditions. Electrical conductivity of two blue-coated diamonds was tested using the method previously described by King et al. (1998). Reactions to UV radiation for all samples were checked in a darkened room with conventional four-watt long-wave (366 nm) and short-wave (254 nm) Ultraviolet Products lamps, and—for the six GIA before/after stones—with the DTC DiamondView deep-ultraviolet (<230 nm) imaging system (Welbourn et al., 1996). The colors of these treated diamonds were described in accordance with the GIA colored diamond grading system (King et al., 1994) to facilitate discussion of color changes during these experiments. Note that GIA does not issue grading reports on coated diamonds, so these color designations should be interpreted as *equivalent* colors only.

Spectroscopic Analysis. Absorption spectra were recorded for all six of the before/after samples and several randomly selected market samples (eight blue and 21 orange, purple-pink, or pink coated diamonds; the yellow and green samples were too small to analyze). They were recorded in the mid-infrared (6000–400 cm^{-1}, 1 cm^{-1} resolution) and near-infrared (up to 11,000 cm^{-1}, 4 cm^{-1} resolution) ranges at room temperature with a Thermo-Nicolet Nexus 670 Fourier-transform infrared (FTIR) spectrometer equipped with KBr and quartz beam splitters. A diffuse reflectance apparatus was used to focus the incident beam on the sample, and a total of 512 scans per spectrum were collected to improve the signal-to-noise ratio. Absorption spectra in the ultraviolet/visible/near-infrared (UV-Vis-NIR) range were recorded on two samples of each color except green with a Thermo-Spectronic Unicam UV500 spectrophotometer over 250–850 nm (sampling interval of 0.1 nm). The samples were mounted in a cryogenic cell and cooled to liquid nitrogen temperature (–196°C).

Raman and photoluminescence (PL) spectra were recorded on four (gray-violet, orange, purplish pink, and yellow) of the six diamonds treated for this study—both before and after treatment—using a Renishaw inVia Raman confocal microspectrometer with an Ar-ion laser operating at two excitation wavelengths, 488.0 and 514.5 nm. Raman spectra were collected at room temperature. Up to five scans were accumulated to achieve a better signal-to-noise ratio. For the PL analyses, the samples were cooled by direct immersion in liquid nitrogen. The lasers were carefully focused on the coated surfaces.

Chemical Analysis. We used several techniques to determine the chemical composition of the coating materials. Three coated diamonds (orange, pink, and blue; 0.30, 0.30, and 0.59 ct, respectively) were imaged and chemically analyzed using a high-resolution analytical scanning electron microscope (LEO 1550 VP FESEM) at the California Institute of Technology in Pasadena. This instrument was equipped with an Oxford INCA Energy 300 X-ray energy-dispersive spectrometer (EDS) system. The analysis was performed using an accelerating voltage of 20 kV and electron beam current of 10 nA; no additional coating (e.g., carbon) was applied.

Chemical analysis was also performed with a Thermo-Noran Spectrace QuanX energy-dispersive X-ray fluorescence (EDXRF) spectrometer on a few samples of each color from the market group as well as on the six GIA samples after treatment. A collimator of 3.5 mm diameter was used. Beam current was automatically controlled to maintain a 50% dead time in data collection. The live time for data accumulation was 100 seconds. All spectra were collected under vacuum at ~0.01 Pa.

Analysis of the pink, purple-pink, yellow (no. 79539; table 1), orange, and blue surface coatings was carried out by secondary ion mass spectroscopy (SIMS) sputter depth profiling by the Evans Analytical Group, East Windsor, New Jersey (Wilson et al., 1989). This technique measures elemental signals while a small cavity (80 × 80 μm) is slowly etched into the surface of the sample. Polished diamond facets provide ideal flat surfaces for this analysis so that profiles with high depth resolution can be acquired. Therefore, we could discern the chemical composition of any chemically distinct layer with a thickness of several nanometers within the coatings. This analysis was carried out using a Phi 6600 quadrupole-based SIMS instrument. Depth profiles were acquired at one location on each diamond using an oxygen ion beam focused to a diameter of approximately 10 μm. This beam was rastered over an area of 50–80 μm^2 with a sputtering rate of 0.2 nm/second depth. This low sputtering rate was necessary to adequately sample the very thin coatings on these diamonds. Mass spectra were acquired for each sample to detect the chemical elements present in the films, and then the most abundant signals were selected to be followed in the depth profiles. The depth scales were calibrated against a SiO_2 film of known thickness. Note that the green stones acquired from the market were too small to be tested by this method.

RESULTS

Gemological Features. These coated diamonds were remarkable for the large variety of natural-looking colors (again, see figure 1). All of the commercial samples except the yellow diamonds were strongly saturated, corresponding to the GIA grades of Fancy Intense to Fancy Vivid. Most of the yellow samples showed some degree of brown modifier, and only a few of the blue diamonds had a modifying hue. A yellow modifier was seen in many of the green and orange diamonds. The before/after experiments confirmed the production of yellow, orange, orangy pink, purplish pink, and pink colors by this new coating method (again, see figure 3; table 1 lists the corresponding color grades). It is interesting to note that a Fancy

TABLE 1. Characteristics of six diamonds before and after coating.[a]

Sample no.	Weight (ct)	Clarity	Color		Fluorescence to short-wave UV		Fluorescence to long-wave UV	
			Before	After	Before	After	Before	After
79538	0.36	VS_1	I	Fancy gray-violet	Inert	Inert	Weak blue	Weak blue
79539	0.37	VS_2	I	Fancy Light yellow	Weak yellow	Weak yellow	Strong blue	Strong blue
79540	0.38	VS_1	J	Fancy Intense orangy pink	Inert	Inert	Moderate blue	Moderate blue, chalky
79541	0.34	VVS_1	E	Fancy Vivid orange	Inert	Inert	Inert	Inert
79542	0.40	VVS_2	E	Fancy Intense pink	Inert	Inert	Weak blue	Very weak blue
79543	0.40	VS_1	F	Fancy Deep purplish pink	Inert	Inert	Moderate blue	Weak blue

[a] *Note that although these six samples were of relatively high color and clarity, the industry typically sends lower-quality diamonds for this coating treatment.*

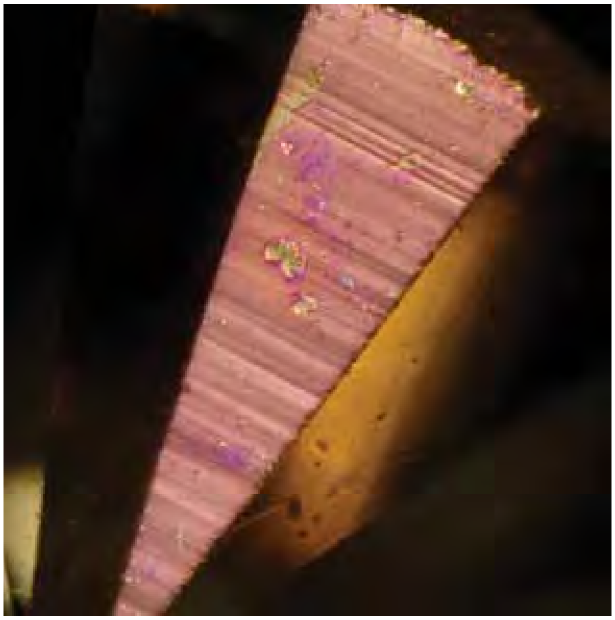


Figure 4. An interference-related colored film—here, a purple film on a green-coated diamond—was observed when the diamonds were viewed with diffused reflected light. Photomicrograph by W. Wang; magnified 105×.

Figure 5. Colorless spots, dark stains, and scratches occurred randomly on some coated facets. Photomicrograph by W. Wang; magnified 112×.

gray-violet color was produced on sample 79538, instead of the pure blue color we observed in many samples obtained from the market. Microscopic examination did not reveal any damage to the diamonds from this treatment process.

The color of all the coated samples appeared evenly distributed when they were examined face-up with magnification and diffused light. However, when their pavilion facets were viewed in diffused reflected light, the coating was visible as an interference-related colored film on the surface of all samples. This feature was particularly evident in the green-coated diamonds, which displayed an intense purple interference color (figure 4). Often, the color appeared bronzy. Randomly distributed colorless spots, dark stains, and scratch lines were also observed on some coated facets (figure 5), but many of the facets did not show such damage. For all the samples examined in this study, the coating was seen only on the pavilion facets; it was not observed on the table or crown facets.

Unlike natural-color pink diamonds, no pink graining was evident in the pink-coated diamonds when they were examined with magnification and darkfield illumination. Common internal features in natural-color diamonds (e.g., the high clarity observed in type IIb blue diamonds and the patchy distribution of color in natural yellow-orange diamonds) were generally missing in their coated counterparts. Instead, inclusions of varying sizes were typically seen, indicating the relatively low clarity of some of the stones selected for this treatment.

The coating had little effect on the UV-activated fluorescence in the six before/after samples (see table 1). After treatment, these six samples exhibited the same color of fluorescence to both long- and short-wave UV radiation, but the intensity to long-wave UV was weaker in two of them (nos. 79542 and 79543). In addition, chalky fluorescence developed in sample 79540 after the treatment.

Likewise, there seemed to be no correlation between UV-activated fluorescence and the coated color for the diamonds obtained from the market. Blue fluorescence of varying intensity was the most common (~80%) reaction to long-wave UV among the 102 diamonds, followed by weak yellow fluorescence (~20%). Weak-to-moderate yellow fluorescence to short-wave UV radiation was very common (~80%), while some samples appeared inert (~15%).

The DiamondView instrument did not show any obvious fluorescence variations on either the table or the pavilion after the coating treatment for any colors except orange. Before treatment, sample 79541 showed blue fluorescence in the DiamondView; after the orange coating was deposited, we observed a moderately strong orange fluorescence from the pavilion.

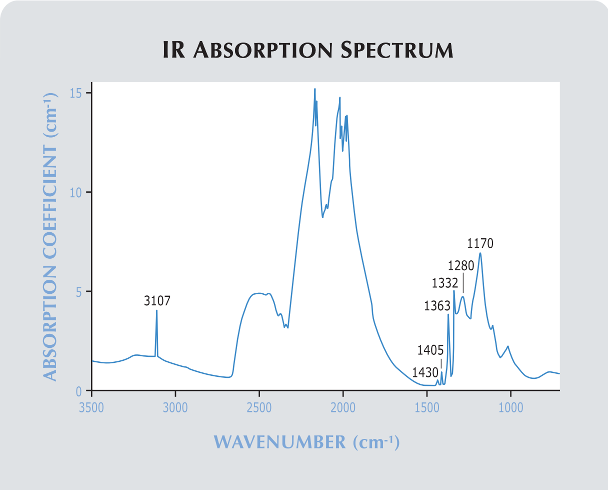
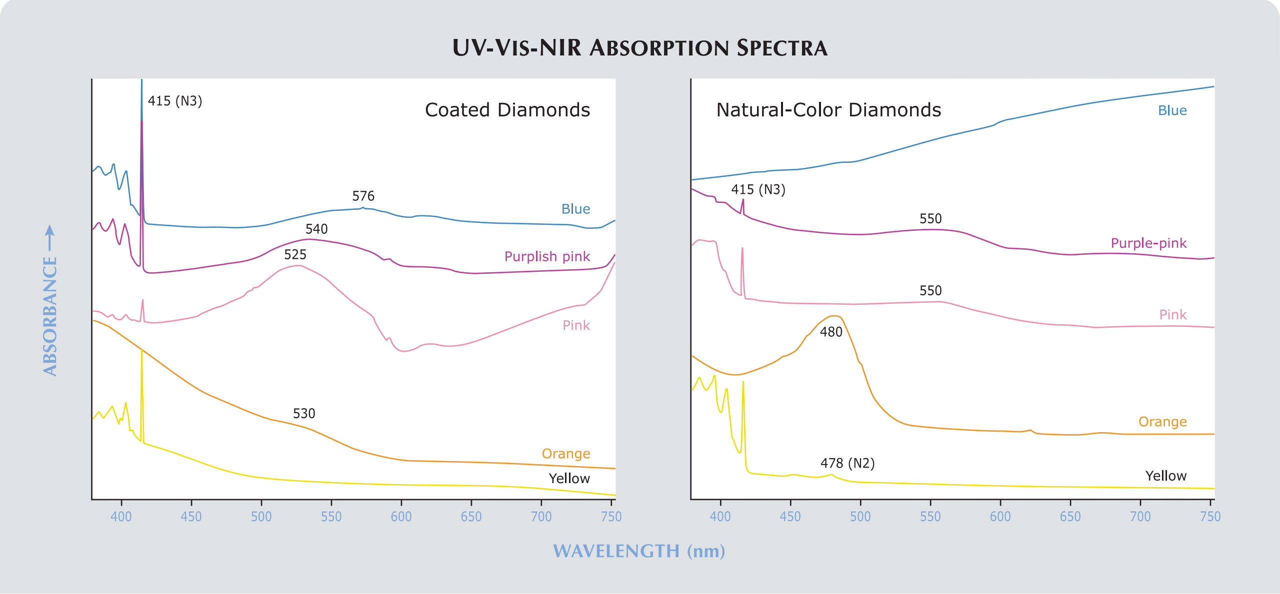


Figure 6. This absorption spectrum of a blue-coated diamond in the mid-infrared region shows that it is type IaAB with a moderately high to very high concentration of nitrogen (>250 ppm) and high hydrogen. No boron-related absorption was detected in any of the treated blue diamonds.

Spectroscopic Features. Infrared absorption spectra for the 21 randomly selected orange, purple-pink, and pink treated diamonds indicated that they were all type IaAB with very high concentrations of nitrogen. The spectral features of many samples (>50%) were similar to those of cape yellow diamonds. Eight blue samples analyzed in this study showed moderately high to very high concentrations (well over 250 ppm; type IaAB) of nitrogen. Two of these stones had higher concentrations of B-aggregate and were rich in hydrogen, as evidenced by the 3107 cm^{-1} peak in figure 6. No boron-related absorptions, such as those observed in natural or synthetic type IIb diamonds, were detected in any of the blue diamonds analyzed. Identical mid- and near-infrared absorption spectra were recorded in the six diamonds before and after treatment, and all showed cape series features. We did not observe any absorption features in these regions of the spectrum that could be attributed to the coatings.

Consistent absorption features in the UV-Vis-NIR region were recorded from samples of the same color categories; typically, they differed from those associated with their natural-color counterparts (figure 7). Absorption by the N3 system (zero phonon line at 415 nm) was detected in all of these samples with varying intensity, indicating the natural origin of these treated diamonds. The pink-coated diamonds showed an absorption band centered at ~525 nm that was so broad that a significant amount of blue light was absorbed (full width at half maximum [FWHM] ~89 nm). A similar broad absorption band occurred in the purplish pink diamonds. However, the center of this band shifted to ~540 nm, and it was slightly narrower (FWHM ~82 nm). As a result, absorption of blue light was less intense

Figure 7. The visible spectra of the Serenity coated diamonds (left) exhibited absorption features that were distinctly different from the spectra of their natural-color counterparts (right). Spectra are offset vertically for clarity.



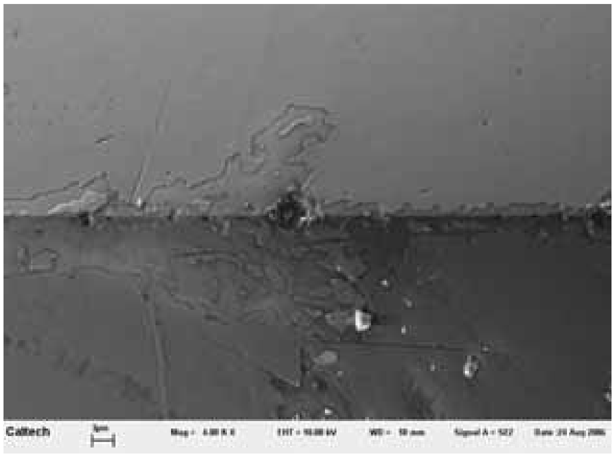


Figure 8. Backscattered electron imaging taken with the SEM reveals a thin film on the pavilion of this diamond. Some damage to the coating is visible along this facet junction. Image by Chi Ma.

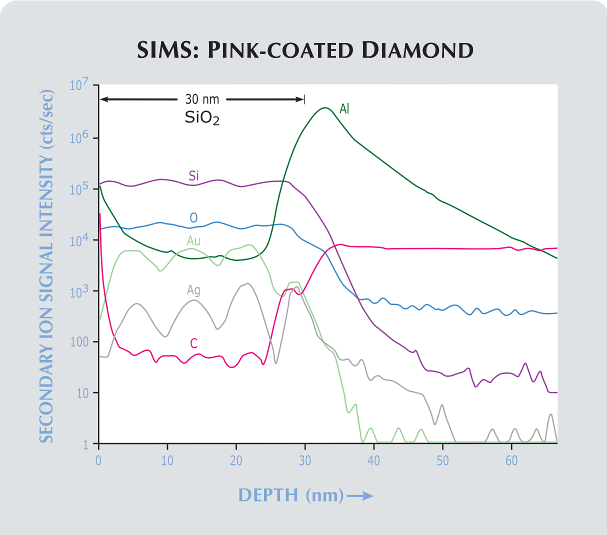


Figure 9. This SIMS depth profile reveals a silicon dioxide (SiO_2) coating with oscillating metal-bearing layers (Au>Ag) on one of the pink samples. The elemental intensities are shown on a logarithmic scale.

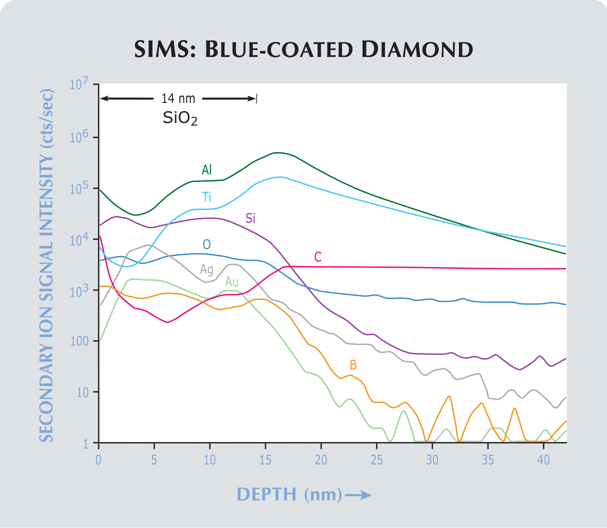
than in the pink-coated diamonds. In the blue samples, a broad band extended from 500 to ~700 nm, with the maximum at ~576 nm—absorbing a wide range of visible light and creating a transmission window in the blue region. In the orange-coated diamonds, we observed a decrease in absorption from the UV region to ~560 nm, as well as a weak feature at ~530 nm. The yellow-coated diamonds displayed a smooth decrease in general absorption from the UV region to ~550 nm. Although the other coated diamonds showed absorption features in the region of 520–580 nm, no such features were observed in the yellow-coated diamonds.

No Raman or photoluminescence signals attributable to the coatings were detected. The spectra before and after treatment were nearly identical.

Electron Imaging and Chemical Composition. The back-scattered electron images generated by the scanning electron microscope confirmed the presence of a thin surface film on the pavilion facets of all three diamonds tested (e.g., figure 8). The thickness of the coating could not be determined with this technique, but these images showed it to be <100 nm. Some visible damage to the coating, or lack of deposition, was seen along facet junctions of each of the three diamonds. The irregular embayments near the facet junctions are not known to occur on the surface of a polished diamond, and they are a strong indication that a coating has been applied. EDS chemical analysis detected Fe, Si, O, and C in the orange-coated diamond, and Si, Au, O, and C in the pink- and blue-coated diamonds. Since these films are very thin, the concentrations of these chemical elements could not be determined reliably. Only carbon was detected on the crown facets.

Using EDXRF analysis, we detected Fe and Si in an orange-coated diamond, and Au and Si in the

Figure 10. The SIMS depth profile of a blue coating shows similar chemical components as in the pink film, but with the added presence of Ti. (The boron signal is likely due to contamination.) However, the signal intensity of Ag is much higher than that of Au, and parallel trends for Al and Ti are observed. The blue coating also exhibits fewer variations in the apparent number of film layers. The elemental intensities are shown on a logarithmic scale.



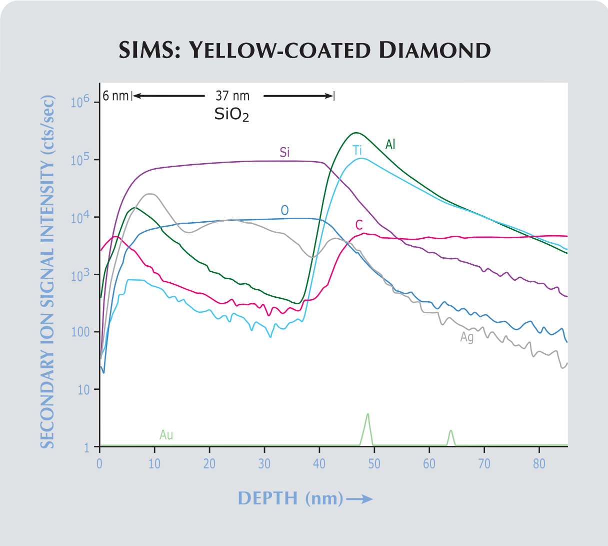


Figure 11. In the yellow coating, the concentration of Ag is very high, while Au is virtually undetectable. Unlike the other colors analyzed, the outermost layer (~6 nm) of the yellow coating appears to contain carbon. Two isotopes of silver (^{107}Ag and ^{109}Ag) were identified for this sample. The elemental intensities are shown on a logarithmic scale.

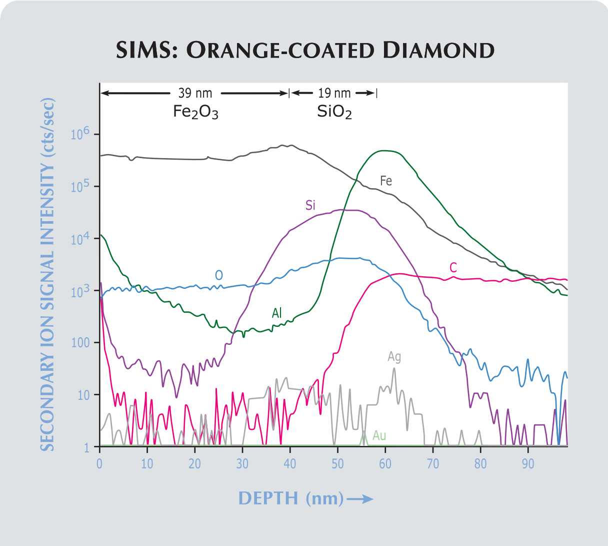


Figure 12. The orange coating was the only color analyzed that had a surface layer of iron oxide (likely as Fe_2O_3). Beneath the iron oxide layer is a SiO_2 film and an Al adhesion layer. The orange coating contains virtually no Au or Ag. The elemental intensities are shown on a logarithmic scale.

blue, pink, and purple-pink diamonds, similar to the observations of other researchers (Kitawaki, 2007). Signals of Fe and Au were usually very clear. Although Si has been shown by other techniques to be a major element of the coating, with this type of analysis it appeared as a weak "shoulder" beside a strong diffraction peak.

The depth profiles as measured by SIMS showed that these films, except the orange, consist of silicon and oxygen (likely as SiO_2) with various impurity elements added as coloring agents (figures 9–12). The interface between the coating and the diamond is marked by an increase in the intensity of the carbon signal, which then levels off within the diamond.

In the pink-coated diamond, Au had a higher intensity than Ag in the SiO_2 film (figure 9). Oscillations in the Au and Ag intensities with depth indicate that the film is composed of three SiO_2 layers (about 30 nm total thickness) that contain varying amounts of Au and Ag. A very thin layer of Al metal appears to be present between the diamond and the SiO_2 layers.

The blue-coated diamond had a SiO_2 layer only 14 nm thick that was doped with both Au and Ag (figure 10); Al is evident as well. This film also contained significant Ti. A very weak boron signal was also seen in the blue coating; this likely is due to contamination. Its intensity decreased gradually with depth before reaching the diamond, and then it decreased sharply. In addition, there was no increase in boron intensity as the sputtering ion beam reached the diamond surface, whereas the intensity of carbon increased gradually.

The SiO_2 film of the yellow-coated diamond was found to be Ag-doped, with virtually no Au detected (figure 11). It is interesting to note that a high concentration of carbon was observed in the outermost layer of the coating. After attaining a maximum in the near-surface region, the intensity of the carbon signal decreased with increasing sputtering depth before reaching the diamond—a profile that was not observed for other elements including Si. An ~9 nm layer of Al and Ti was identified between the SiO_2 coating and the diamond. The total thickness of the coating on the yellow diamond was about 52 nm.

In contrast to the other colored samples, the orange-coated diamond had a surface coating of nominally undoped iron oxide approximately 39 nm thick (figure 12). Beneath the iron oxide film was a SiO_2 film about 19 nm thick, with an underlying Al layer of ~4 nm.

DURABILITY TESTING

When a new gem treatment method is introduced, inevitably the question arises "Is it durable?" To test this, we set up a limited series of experiments that focused mainly on the conditions such treated diamonds might encounter in real-life situations. The tests included controlled heating; standard jewelry setting, repair, and cleaning procedures; exposure to common household cleaners; and scratching.

Methods. *Controlled Heating.* Controlled heating experiments were performed using a Lindberg/Blue M Moldatherm box furnace. One sample of each color (pink, purple-pink, yellow, green, blue, and orange) was heated at 300–800°C for 30 minutes in air, with a 100°C increment between each step. In each experiment, the target temperatures were reached and then the samples were placed in the oven.

Jewelry Setting, Repair, and Cleaning. Three coated samples (pink, blue, and orange) were exposed to a series of procedures designed to assess this treatment's durability to standard jewelry manufacturing, repair, and cleaning practices. The samples had to be larger than 0.30 ct to fit in a standard solitaire setting that would allow adequate viewing of the pavilion. After each test, the stones were examined carefully with a standard gemological microscope and photographed as appropriate. Only one of the stones (the pink) was used for the rhodium plating test, and seven other coated diamonds were exposed to sulfuric acid.

1. *Setting:* The diamonds were mounted in standard 14K white gold rings, the prongs filed, and then one pavilion facet of each mounted diamond was polished using a wheel charged with a Tripoli or Rouge compound for about 5 seconds (as would be the case if the jeweler inadvertently touched the stone while polishing a prong).

2. *Retipping of Prongs:* The procedures used were similar to those previously performed on lead glass–filled rubies (McClure et al., 2006):
   - A. The rings were soaked in a borax solution and gently heated. A small bead of low-temperature solder ("easy flow," melting temperature 621°C [1150°F]) was dipped in Battern's flux (a mixture of borax and ammonium chloride) and placed on top of a prong. Using a bench jeweler's torch, the solder was heated until it flowed around the broken prong.
   - B. Following the same procedure, a high-temperature solder ("hard flow," melting temperature 788°C [1450°F]) was tested on a different prong, where the adjacent coating seemed undamaged.
   - C. A small drop of Battern's flux was placed on the coating and the stone was heated to the melting temperature of the low-temperature solder.

3. *Rhodium Plating:* This simple electrode plating process for white-metal jewelry involves immersing the set object in a sodium hydroxide bath, as well as in a sulfuric acid bath containing dissolved rhodium. In both baths, the jewelry serves as an electrode with current passing through it. The ring set with the pink-coated diamond was plated and cleaned with ultrasonic and steam cleaners.

4. *Steam Cleaning:* A standard steam cleaner (40–50 psi) was used on the three mounted diamonds, held ~2.5 cm from the nozzle for 10–30 seconds.

5. *Immersion in a Pickling Solution (Sodium Bisulfate):* The three rings were immersed for one minute in a warm pickling solution after the low- and high-temperature retipping processes, and then steam cleaned for a several seconds.

6. *Exposure to Sulfuric Acid:* Seven other coated diamonds were boiled in sulfuric acid for 30 minutes, a method commonly used to clean master stones. We included one sample of each color (two were pink). Except for one orangy pink sample (0.50 ct), all of these samples were small (0.01–0.07 ct).

Exposure to Common Household Cleaners. The durability of 15 coated diamonds (pink, yellow, green, blue, and orange) was tested by exposure to kitchen detergent (water-diluted Palmolive), rubbing alcohol (isopropyl), acetone (reagent grade), and bleach (undiluted Clorox, which is 5.25% sodium hypochlorite). The diamonds were immersed in each substance and removed for observation after one minute, 5 minutes, and every 10 minutes up to one hour, then every hour for 6 hours. Another observation was made after 24 hours of immersion, and the last was made after 48 hours. All samples were then reimmersed and transferred to an ultrasonic cleaner for 60 minutes. Note that the limited time span of the testing may not correlate with the long-term durability of the coatings.

Scratch Tests. We tested seven coated diamonds of pink, blue, and orange colors using the following materials: a household sandpaper (Mohs hardness ~ 9), a synthetic corundum boule (Mohs hardness = 9), an abrasive powdered cleanser (Mohs hardness ~ 7), and a stainless steel needle (Mohs hardness ~ 5.5). The coated surface was gently rubbed against these materials. The powdered cleanser was made into a slurry and spread on a cloth, and the diamonds were gently rubbed between two layers of the cloth for 10 minutes.

TABLE 2. Summary of durability tests on different colors of coated diamonds.

Testing method	Blue	Green	Orange	Pink	Purple-Pink	Yellow
Jewelry setting and repair						
Controlled heating in air up to 500°C (figure 13)	No damage	No damage	No damage	No damage	No damage	No damage
Controlled heating in air from 600°C to 800°C (figure 13)	Color became pink at 600°C; cloudy appearance at 800°C	Cloudy appearance at 700°C, coating loss at 800°C	Cloudy appearance, coating loss at 800°C	Cloudy appearance, coating loss at 800°C	Cloudy appearance, coating loss at 800°C	Cloudy appearance, coating loss at 800°C
Setting—filing	No damage	N/T[a]	No damage	No damage	N/T	N/T
Setting—polishing (Tripoli or Rouge on metals)	No damage	N/T	No damage	No damage	N/T	N/T
Setting—polishing (Tripoli or Rouge on a pavilion facet; figure 15)	Damaged	N/T	Damaged	Damaged	N/T	N/T
Retipping—low-temperature (easy flow) solder	Color changed (figure 16)/reacted to flux	N/T	Burned appearance (figure 17)	Reacted to flux (figure 18)	N/T	N/T
Retipping—high-temperature (hard flow) solder	Color changed (figure 16)/reacted to flux	N/T	Changed color/ burned appearance (figure 19)	Reacted to flux (figure 18)	N/T	N/T
Exposure to Battern's flux	Reacted	N/T	N/T	Reacted (figure18)	N/T	N/T
Rhodium plating	N/T	N/T	N/T	Dissolved (figures 20 and 21)	N/T	N/T
Jewelry cleaning						
Steam cleaning (10–30 seconds)	No damage	N/T	No damage	No damage	N/T	N/T
Pickling solution (one minute)	No damage	N/T	No damage	No damage	N/T	N/T
Sulfuric acid bath (boiling; 30 minutes)	Damaged	Damaged	Damaged	Damaged (figure 22)	Damaged	Damaged
Ultrasonic cleaning	No damage	No damage	No damage	No damage	No damage	No damage
Exposure to household chemicals						
Kitchen detergent (diluted Palmolive)	No damage	No damage	No damage	No damage	N/T	No damage
Isopropyl alcohol	No damage	No damage	No damage	No damage	N/T	No damage
Acetone	No damage	No damage	No damage	No damage	N/T	No damage
Bleach (undiluted Clorox—two days)	Color changed	Color changed (figure 23)	No damage	No damage	N/T	Color changed (figure 23)
Scratch tests						
Sandpaper (3M Wetordry Tri-M-ite silicon carbide paper)	Scratched	N/T	Scratched	Scratched	N/T	N/T
Synthetic corundum boule	Scratched	N/T	Scratched	Scratched	N/T	N/T
Powdered cleanser slurry (Waxie brand)	Scratched	N/T	Scratched	Scratched (figure 24)	N/T	N/T
Stainless steel needle point	Not scratched	N/T	Not scratched	Not scratched	N/T	N/T

[a] *N/T = not tested.*

Results. The results of the durability testing are listed in table 2 and summarized below.

Controlled Heating. All colors remained virtually the same up to 500°C (figure 13). Only the blue-coated stone turned pink after heating at 600°C. However, severe damage to the coatings (as well as the diamonds) was observed after heating at 700–800°C: Almost all samples became cloudy, and on all of them microscopic examination

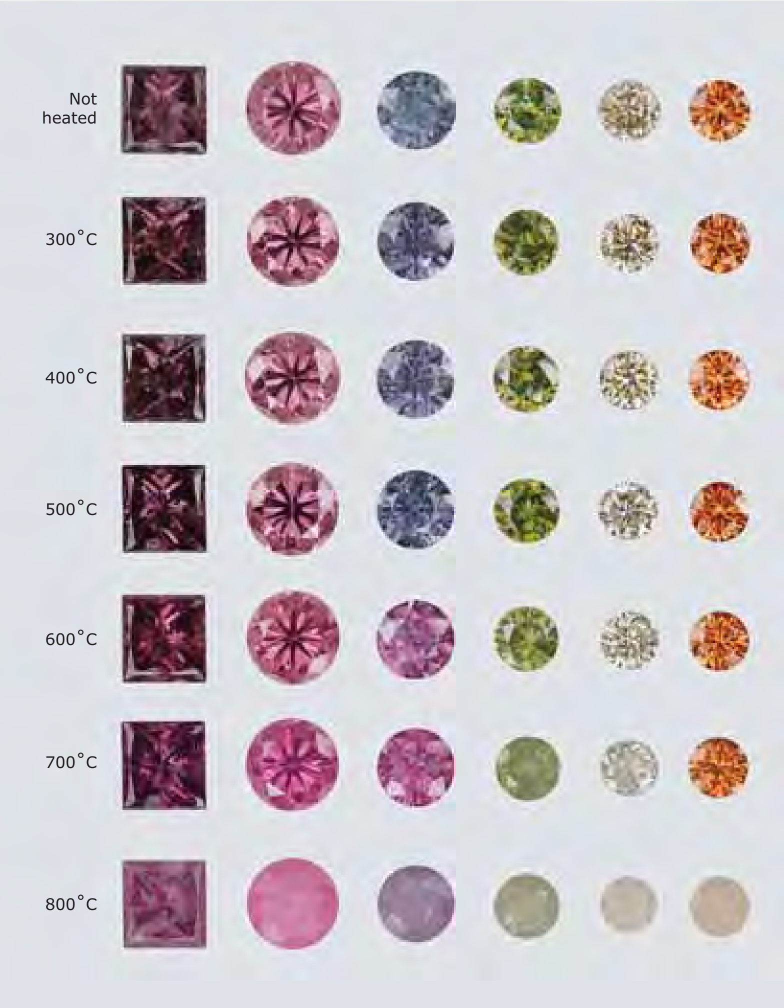


Figure 13. Heating of the coated diamonds in air demonstrated that the coatings are stable up to 500°C, as shown here, but substantial change could occur between 600°C and 800°C. The blue coating turned pink after heating at 600°C for 30 minutes. Composite photo by Jessica Arditi.

revealed that most of the coating had peeled off, with only small remnants remaining (figure 14). It should be mentioned that even untreated diamonds would display surface damage if heated at this temperature in air.

Epelboym et al. (2006) studied the impact of annealing similar coated diamonds in a vacuum, which reduced the effects of oxidation. They reported that while some stones began to change color at temperatures as low as 300°C, most changed color at 900–1000°C.

Jewelry Setting, Repair, and Cleaning. No damage was noted from the filing or polishing of the prongs, but the coatings on all three diamonds were removed where the pavilion facet was polished with a wheel charged with either Tripoli or Rouge (figure 15).

The most dramatic changes during retipping were seen in the blue-coated diamond. This Fancy gray-blue sample turned Fancy purplish gray after the low-temperature solder procedure (figure 16). This is not surprising, given the results of the heating tests (again, see figure 13). The blue-coated diamond turned pink at 600°C, and the easy-flow solder has a nominal melting temperature of 621°C. The orange coating showed a burned appearance on a relatively large area adjacent to the retipped prong (figure 17). The pink-coated diamond did not show any obvious color alteration after retipping with either type of solder. However, the pink and blue

Figure 14. After heating at 800°C for 30 minutes, most of the green coating peeled off this diamond and only a small portion remained. Photomicrograph by W. Wang; magnified 70×.

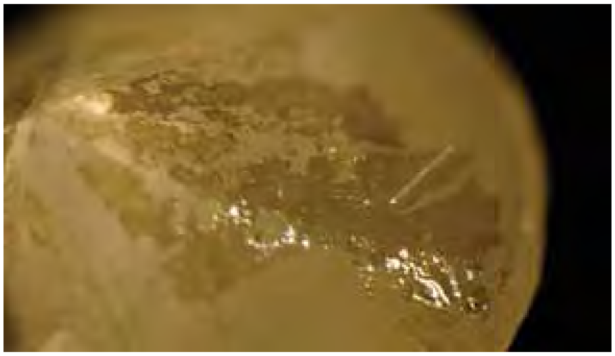


Figure 15. Note the difference in reflection on this pavilion facet of the orange sample after polishing (with Tripoli), which removed the coating where it touched the stone. Photomicrograph by A. H. Shen; magnified 40×.

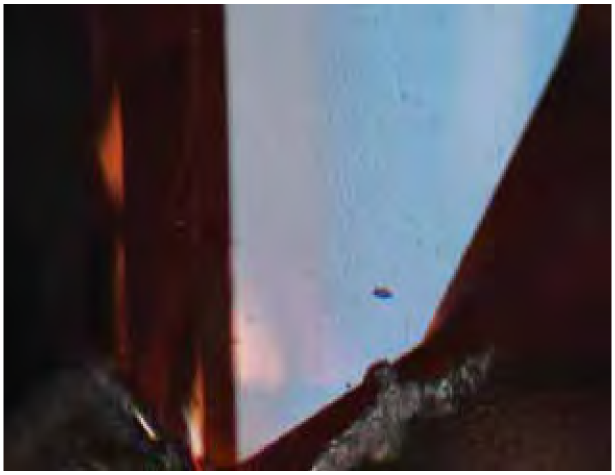


Figure 16. Retipping of a prong with a low-temperature solder caused a pronounced change in the appearance of this 0.36 ct coated diamond, from Fancy gray-blue (left) to Fancy purplish gray (center). The subsequent use of high-temperature solder caused the color to become slightly more purple (right). Photos by Jian Xin (Jae) Liao (left) and Robison McMurtry (center and right).

coatings reacted with the Battern's flux, as shown by mottled features that did not exist before testing (figure 18).

After the higher-temperature retipping process, the blue-coated diamond appeared more purple, but retained its equivalent grade of Fancy purplish gray (again, see figure 16). The original Fancy Vivid orange sample changed to a Fancy Intense yellowish orange and had a burned appearance (figure 19).

After retipping and rhodium-plating, the pink-coated diamond remained Fancy Intense pink, but there was a slight change of color and obvious damage to the surface (figure 20), as the film showed signs of dissolution (figure 21). This is not surprising because the coating can be completely dissolved in strong sulfuric acid, as described below.

No damage was seen in any of the stones with steam cleaning, ultrasonic cleaning, or immersion in a pickling solution. However, boiling in sulfuric acid entirely removed the coatings from six of the seven samples, restoring their original light yellow or light brown coloration (e.g., figure 22). However, a very light pink hue was preserved in the purplish pink coated diamond, and examination with the microscope revealed some coating remnants. In comparison, examination of such coated diamonds at intervals shorter than 30 minutes by Kitawaki (2007) found no effect after three minutes of boiling with aqua regia (25% nitric acid and 75% hydrochloric acid) and no macroscopic evidence of damage after 10 minutes of boiling.

Exposure to Common Household Cleaners. There was no observable damage to the pink and orange coatings with exposure to the various chemicals. The blue, green, and yellow samples were stable in

Figure 17. The film on the orange-coated diamond was damaged ("burned") over a relatively large area during the retipping process with a low-temperature solder. Photomicrograph by A. H. Shen; magnified 20×.

Figure 18. Exposure to Battern's flux, which was used during the retipping process, caused a mottled appearance on this area of the pavilion in the pink-coated stone. Photomicrograph by A. H. Shen; magnified 40×.

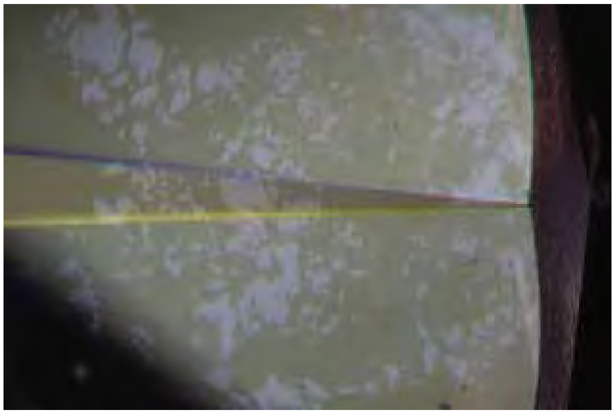


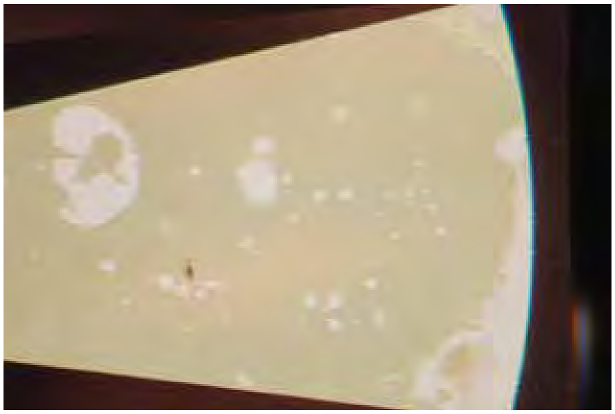
Figure 19. This orange-coated diamond (0.30 ct) was Fancy Vivid orange before the retipping test, but changed to Fancy Intense yellowish orange during exposure to the high-temperature solder. The sample also developed a burned appearance. Photos by Jian Xin (Jae) Liao (left) and Robison McMurtry (right).

Figure 20. These images show that the jewelry repair tests had some effect on this 0.40 ct pink-coated diamond's apparent color. While the stone was graded Fancy Intense pink both before and after the tests, following rhodium plating approximately 10% of the surface showed evidence of damage in the form of uneven color distribution (see, e.g., circled areas). Photos by Jian Xin (Jae) Liao (left) and Robison McMurtry (right).

the detergent, alcohol, and acetone, but the colors of all three had faded after two days of immersion in the bleach (e.g., figure 23). Although prolonged exposure to bleach for this amount of time is unlikely in normal household use, regular exposure over months or years could produce the same result.

Scratch Tests. The stainless steel needle did not scratch the coatings, but the sandpaper, the tip of the synthetic corundum boule, and the powdered cleanser slurry did cause damage. For example, after the stones were rubbed with a powdered cleanser slurry, their coatings showed a mottled pattern (figure 24). According to the Materials Safety Data Sheets (MSDS) database, the powdered cleanser we used contained a bleach agent, calcium carbonate, and crystalline silica.

Figure 21. After the rhodium plating process, this pavilion facet on the pink-coated diamond in figure 20 showed damage that was not evident prior to the test. The pattern on the area adjacent to the girdle suggests dissolution of the coating. Photomicrograph by A. H. Shen; magnified 40×.



DISCUSSION

Nature of the Treatment. Diamond treatments to alter color are now routine. Well-known methods include irradiation (with and without subsequent heat treatment) and HPHT annealing. The treated colors produced by these methods generally are caused by defects similar to those present in natural diamonds. Consequently, most such treated colors are considered permanent with respect to the condi-

Figure 22. When this pink-coated diamond (0.50 ct) was boiled in sulfuric acid for 30 minutes, the coating was completely removed and the sample regained its original light yellow coloration. Photos by Jian Xin (Jae) Liao.

tions that a diamond would be subjected to during jewelry manufacture, repair, cleaning, and wear. In contrast, the treated diamonds in this study all received their perceived color from the presence of a thin coating, as confirmed by microscopic examination as well as SEM and SIMS analyses. Compared to some other coatings seen in the laboratory, the Serenity Technologies coating appeared to be very even, and the numerous colors produced were quite natural. Strong saturation was seen in all the hues except yellow.

The consistently high concentrations of Si and O in all the coatings demonstrate that the film has SiO_2 as a major component, while other metals or metal oxides appear to have been intentionally introduced as dopants or additional layers. Au and/or Ag likely are the major dopants in the purple-pink, blue, and yellow diamonds, while a high content of Fe was detected in an orange diamond. These thin coatings have strong selective absorptions in the visible light region, which induce the various colors that are observed. These observations are consistent with a recently published patent application (Neogi and Neogi, 2006).

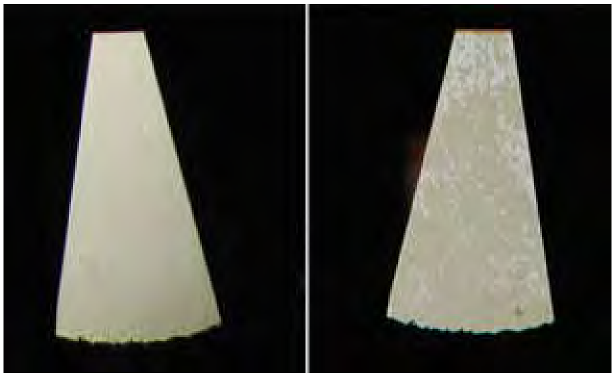
According to the SIMS analyses, the coatings are very thin (<60 nm). Interference of light reflected from the coating surface and from the interface with the diamond creates the colors observed with diffused reflected light (again, see figure 4). However, the presence of the thin coatings cannot be detected using standard techniques for recording Raman spectra or infrared absorption spectra. The sampling depth of the Raman microspectrometer used in this study is ~3 μm, which is about two orders of magnitude thicker than the coatings. As a result, the Raman spectra were dominated by the underlying diamond, and no scattering signal could be detected from the coatings. Although many details, such as the presence of a silica layer and gold impurities, correspond to those reported by Epelboym et al. (2006), this study could not confirm their observation of a Raman peak associated with silicon carbide (SiC).

The SIMS depth profiles showed gradual, rather than abrupt, changes in the elemental concentrations at the transition between the coating and the diamond. However, this should be interpreted as an artifact of the SIMS sputtering method and resolution. In reality, there is likely a distinct interface between the coating and the diamond, with no significant interdiffusion of the elements.

Metal layers in these coatings are commonly used to help the subsequent nonmetal layers adhere to the underlying substrate material (Mattox, 1998). Most commonly, Cr, Ti, or Pt have been used as adhesion promoters; however, thin films of these metals may be more opaque than Al (Ohring, 1992). Very thin layers of Al and/or Ti (several nanometers) detected immediately above the diamond surface on all the samples we tested by SIMS were probably applied to help the coatings

Figure 23. The coatings were removed from these diamonds (0.01–0.02 ct) by immersion in undiluted Clorox bleach for 48 hours. Top—a Fancy Deep green-yellow diamond (left) received a color grade of N on the D-Z color scale (right). Bottom—a Fancy Light yellow diamond (left) was graded V color (right). Photos by Robison McMurtry.

Figure 24. A mottled pattern was evident on this pavilion facet of a pink-coated diamond after being rubbed with a slurry of a powdered household cleanser for 10 minutes. Photomicrographs by A. H. Shen; magnified 40×.



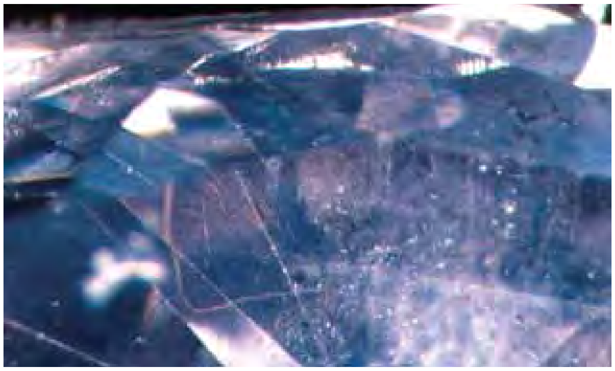


Figure 25. Sometimes surface features on the pavilion of a diamond are more visible when viewed through the table, as with these scratches in the coating on a 0.59 ct stone. Photomicrograph by S. F. McClure; magnified 26×.

adhere to that surface. The authors also detected a similar thin layer of Al in a pink diamond coated with CaF_2. We believe that the improved binding between the diamond and the coating that results from the use of metal adhesive layers is responsible for the greater evenness in color along the facet junctions than earlier coatings seen in the GIA Laboratory.

Identification. Microscopic examination should be the first step in the identification of any colored diamond. Unlike some of the pink CaF_2-coated diamonds, which could be difficult to detect with magnification, these silica-coated diamonds typically may be identified by an experienced gemologist using a gemological microscope. First, since these coatings were only found on the pavilion, it is important to examine the entire diamond when looking for treatment. Many gemologists will inherently begin examination of any stone with the microscope by looking at and under the table facet using darkfield illumination. What they often do not do is look deeper through the table to the pavilion. This type of examination is useful for several identification scenarios, but coatings in particular are often more visible in this viewing geometry (figure 25).

Flipping the stone over and examining the surface of the pavilion with diffused reflected light has proved to be the best method of detecting these coated diamonds. Transmitted light is also very helpful. With these two techniques, the coating is revealed by interference-related colors (again, see figure 4) or a bronze appearance at the surface (figure 26), small uncoated areas or irregularities in the coating (figures 27 and 28), and damage to the coating such as scratches or abrasions along facet junctions or elsewhere (figure 29). Additionally, the GIA sample that received an orange coating (no. 79541) showed whitish marks that made the stone look as if it needed to be cleaned, but they could not be wiped off (figure 30). These methods are similar to those recommended by Miles (1962 and 1964) for coated diamonds seen in the 1950s and ’60s, which are also used to detect coatings on other gems, such as “Aqua Aura” quartz (Kammerling and Koivula, 1992). For additional confirmation, immersion in methylene iodide shows concentrated color in the surface region.

Part of the normal testing procedure for a pink diamond is to check for colored graining and the strain patterns typically associated with it. As stated earlier, the Serenity Technologies coated pink diamonds did not show any evidence of colored graining. However, a number of the CaF_2 coated diamonds we have seen (including some of those in figure 2) *did* display planar brown graining with associated strain patterns. We emphasize this as a word of caution, for if a gemologist was not careful, he or she might easily mistake this for the graining and strain present in most natural pink diamonds. Light brown diamonds typically show these features, and we believe these stones are likely candidates for coating.

By the same token, there is no assurance that a pink diamond that does not display colored graining or strain patterns is treated based on this fact alone.

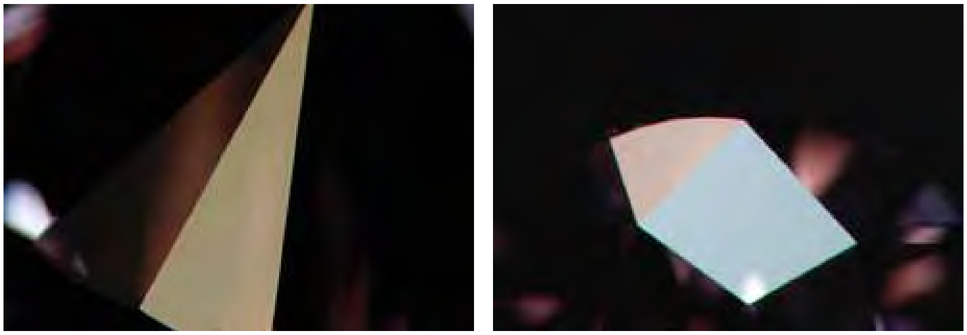


Figure 26. These coatings often have a bronze-colored appearance when viewed in reflected light (left), which makes their presence obvious when compared to the reflected-light appearance of an uncoated facet, here seen on the crown of the same diamond (right). Photomicrographs by S. F. McClure; magnified 23×.

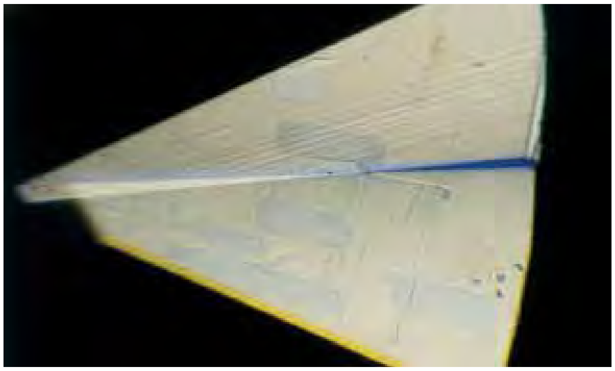


Figure 27. Occasionally, areas are left uncoated on these diamonds and can have several different appearances. Here, with reflected light, they are visible as rectangular patches on a blue-coated stone. Photomicrograph by John I. Koivula; magnified 10×.

A rare type of natural pink diamond, often referred to as a "Golconda pink," can follow this scenario, in which case the UV fluorescence becomes important. These diamonds fluoresce orange to both long- and short-wave UV radiation (stronger to long-wave), which would be an unlikely scenario for a coated diamond.

Other techniques that we used to characterize the coatings, such as SEM and SIMS, also can identify the presence of this treatment, but they are not available to the vast majority of gemologists and are not necessary. However, an anomalous IR or UV-Vis spectrum should alert the gemologist to the possibility of treatment, and EDXRF analysis will help determine if the coating is primarily CaF_2, SiO_2, or some other material.

Coating Durability. The color permanence of these diamonds is entirely determined by the durability of the coatings—how stable the films themselves are and how strongly they adhere to the diamond's surface. SiO_2 films, occurring either as amorphous silica or as quartz, have a Mohs hardness close to 7, which is significantly higher than the hardness of 4 for the CaF_2 diamond coating described by Evans et al. (2005), but considerably lower than that of diamond (10). Therefore, it was not surprising that these coatings were not damaged by scratching with a metal needle (~5.5 hardness), while the CaF_2 coatings we examined in the GIA Laboratory did show signs of damage when similarly tested. SiO_2 is also chemically more stable than CaF_2, but it still can be damaged by strong chemical solutions and harder materials such as abrasive cleaners.

The limited durability of coated diamonds, as revealed in this study and others (e.g., Epelboym et al., 2006; Kitawaki, 2007), is expected in accordance with the kind of coating material applied. When working with these coated diamonds, jewelers must be careful not to directly expose them to high heat and polishing compounds, to rhodium plating, or to cleaning in a sulfuric acid bath. Retailers should advise consumers not to wear the diamonds on their hands while doing household chores that involve bleach or abrasive cleansers.

Color Origin of the Coatings. Recently, SiO_2 films doped with Au and Ag have been extensively investigated for their interesting optical properties (Liz-Marzan, 2004; Yu et al., 2005; Armelao et al., 2006). When SiO_2 is doped with Au and then heated, the Au forms uniformly spaced nanoparticles within the film that create an apparent color due to Mie scattering (Simmons and Potter, 2000). Since the concentrations obtained from SIMS were not calibrated against known samples, it is difficult to calculate the exact composition of the coatings. In addition, one major drawback of SIMS is that sensitivity factors used to translate measured counts to actual concentrations may differ between elements by many orders of magnitude. However, Au and Ag have approximately equal sensitivity factors, so their relative intensities reasonably can be compared. Additionally, the presence of Au and Fe in

Figure 28. Reflected light (left) and transmitted light (right) can both be used to see the small rounded uncoated areas on these diamonds. Photomicrographs by John I. Koivula (left, 25×) and S. F. McClure (right, 23×).

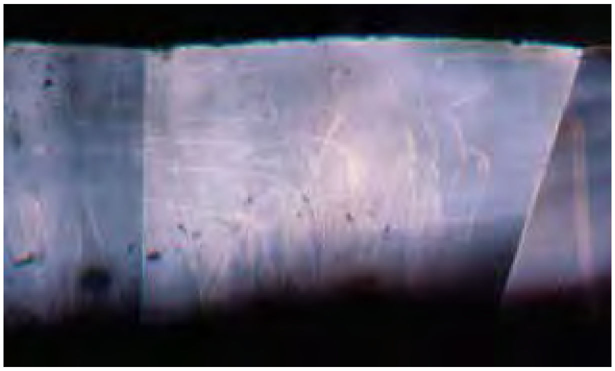


Figure 29. Scratches or other damage to a coating often make it easier to detect. Photomicrograph by S. F. McClure; magnified 35×.

the EDS and EDXRF analyses confirmed that these elements are major components of the coatings. Finally, it is important to reiterate that these analyses are based on depth profiles obtained at one location of one diamond for each color of coating.

Pink Coating. It has been reported that Au-doped films heated to 500°C acquire a light pink color due to an absorption peak at 533 nm (Yu et al., 2005). This is consistent with the absorption spectra of our pink-coated diamond (figure 7) and the concentration profile shown in figure 9. Interestingly, Au was first added to glass in Roman times to cause a red color (Wagner et al., 2000). Deposition of these thin films to create fancy-color diamonds therefore appears to be a modern application of an ancient practice.

Blue Coating. As the concentration of Ag nanoparticles increases within silica, the absorption band shifts toward the red region of the spectrum, to as high as 600 nm (Link and El-Sayed, 1999). This would make the color of the film appear blue. The intensity of the Ag signal (in absolute counts) in the blue film was the highest of any of the doped silica films we measured, and the blue color may be due to a high concentration of nanoparticles of Ag within this film (Link and El-Sayed, 1999). The change of color from blue to pink with heating at 600°C (figure 13) may indicate that such a nanoparticle configuration is unstable at these temperatures, possibly reverting to a more dispersed form similar to that of the pink film.

Figure 30. The numerous irregularities on the surface of this orange-coated sample made the diamond appear to need cleaning, but the marks did not wipe off. Photomicrograph by S. F. McClure; magnified 22×.

The apparent color of the blue-coated diamonds is not derived from boron. Extensive infrared absorption analysis confirmed that all the blue diamonds in this study were type IaAB with significant amounts of nitrogen (e.g., figure 6). None of the samples displayed electrical conductivity or exhibited any boron-related absorption features in their visible or infrared spectra. These observations rule out any involvement of ion-implantation of boron in this treatment. Additionally, these diamonds probably will not be confused with natural type Ia nonconductive gray-to-blue diamonds (see, e.g., Fritsch and Scarratt, 1992). In our experience, most of those diamonds show violet, gray, or greenish overtones and do not show the same blue hues that are observed in type IIb diamonds or these blue-coated diamonds.

Yellow Coating. Silver was the major dopant in the SiO_2 film, and Au was not detected by SIMS. It is unclear if the elevated carbon peak near the surface is intentional or due to contamination, and additional experiments are needed to make that determination. Silver doping of silica produces nanoparticles with an absorption peak near 411 nm, which may cause the samples to appear yellow (Scalisi et al., 2004). This is not entirely consistent with the gradual increase in absorption from ~550 nm to lower wavelengths observed in this study (again, see figure 7).

Orange Coating. In contrast to the other colors, in which silica was the major component of the films, SIMS analysis revealed a high concentration of Fe in the orange coating. Deposited films of Fe_2O_3 appear brown or red-brown, whereas films of FeO and Fe_3O_4 are black (Peng et al., 2003). Therefore, it is reasonable to surmise that the coating on the orange diamond is composed of Fe_2O_3, with the thickness tailored to produce a desirable orange color. Unfortunately, we could not measure the oxidation state of the iron, which would have confirmed this identification.

GEMOLOGICAL REPORTING ON COATED DIAMONDS

Due to the unstable nature of diamond coatings and the fact that a foreign material has been applied to the pavilion facets, all coated diamonds submitted to the GIA Laboratory are issued an identification report, but not a quality grading report. No color grade is provided. The identification report states that the diamond is "Surface Coated" and provides information about the diamond's carat weight, cutting style, and measurements. An additional comment explains that "A foreign material has been artificially applied to the surface, which precludes quality analysis." Other laboratories have similar practices and have indicated that coated diamonds either will not be issued a grading report (Kitawaki, 2007) or will be reported as "color-treated" (Epelboym et al., 2006).

CONCLUDING REMARKS

A new coating method has been developed to change the apparent color of diamonds. The coloration appears evenly distributed, and several colors have entered the marketplace, including blue, green, orange, pink, purple-pink, and yellow. The manufacturer reports that other colors, such as red (J. Neogi, pers. comm., 2007), can also be produced by this process. The coatings, which were applied only to the pavilions of the samples we examined, are very thin. They are composed mainly of multilayer doped SiO_2/metal and sometimes metal oxide films. No diffusion of elements into the diamond was detected.

A film-like coating on the pavilion, which can be seen with careful observation using a gemological microscope and diffused reflected light, is a good indication of this type of treatment. Damage to the film along facet junctions, as well as the presence of scratches and colorless and/or dark areas, are additional evidence of this type of treatment. Chemical analysis is required to confirm the specific identity of the coating material that has been applied to the diamond.

As the durability testing indicated, this is not a permanent treatment. Since a foreign material has been applied, the GIA Laboratory will only issue an identification report—not a grading report—on a diamond treated by this technique. The GIA Laboratory will continue to follow the development of this treatment technology to ensure that all diamonds submitted for examination are properly screened for these more sophisticated coatings.

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