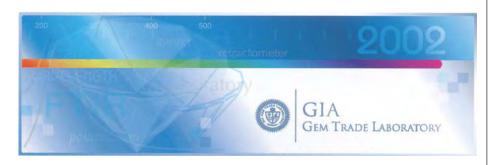
Gem Trade LAB NOTES



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AURICHALCITE

Occasionally we receive items for identification that none of our staff members can recall ever seeing in the lab. Such was the case with a combination pendant and brooch that had a banded white and greenish blue oval cabochon mounted as the center stone. The 29.50×18.10 mm cabochon was set in yellow metal and surrounded by square and round cabochon-cut blue moonstones (figure 1).

The central cabochon had a very distinctive appearance: semi-translucent to opaque with a fibrous botryoidal structure that was somewhat reminiscent of malachite. The bright greenish blue color was typical of minerals that are colored by traces of copper. We obtained spot R.I. readings of 1.63–1.75, which exhibited what is known as a "carbonate blink" on the refractometer. The stone effervesced to a small drop of dilute HCl acid applied to the back, which proved

that the material was a carbonate. Gemological testing was limited because the stone was mounted and mostly opaque. Aggregate carbonates can be difficult to identify in any case, so more-advanced testing was necessary.

Energy-dispersive X-ray fluorescence (EDXRF) spectroscopy revealed mostly zinc and copper, with a trace amount of lead (carbon and oxygen are below the detection limits of this instrument). X-ray diffraction analysis produced a pattern that closely matched both aurichalcite [(Zn,Cu)₅(CO₃)₂(OH)₆] and hydrozincite [Zn₅(CO₃)₂(OH)₆]. The relatively high copper content of this cabochon proved that the material was aurichalcite.

This is indeed the first example of aurichalcite set in jewelry that we can recall seeing in the laboratory. Aurichalcite is quite soft (1–2 on the Mohs scale) and somewhat fragile. This was illustrated by the fact that the cabochon had been repaired; that is, a small piece on the top had been reattached with glue. *SFM*

Figure 1. Advanced testing proved that the unusual center stone (29.50 × 18.10 mm) in this combination brooch and pendant was aurichalcite.



Editor's note: The initials at the end of each item identify the editor(s) or contributing editor(s) who provided that item. Full names are given for other GIA Gem Trade Laboratory contributors.

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Figure 2. After HPHT treatment, this type Ia diamond (1.07 ct) changed from brown to green-yellow.

DIAMOND

Another Commercial U.S. Facility Offers HPHT Annealing

It recently came to our attention that another U.S. facility is offering high-pressure, high-temperature (HPHT) annealing of diamonds to improve their color. Phoenix Crystal Corp. of Ann Arbor, Michigan, an industrial supplier of diamond abrasives and cutting tools, is commercially treating gem diamonds using a standard belt-type press.

Dr. Robert Frushour, president of Phoenix Crystal Corp., has informed us that his facility has both decolorized brown type IIa diamonds and produced a range of colors in type I diamonds. Figure 2 shows a 1.07 ct type Ia diamond before and after HPHT annealing at this facility. Similar to other samples with comparable properties, this one illustrates the predictable color enhancement from brown to green-yellow (see, e.g., I. M. Reinitz et al., "Identification of HPHT-treated yellow to green diamonds," Summer 2000 Gems & Gemology, pp. 128-137). Figure 3 shows the mid- and near-infrared absorption spectra of the 1.07 ct diamond before and after treatment. The presence of the H2 absorption feature

at 10,126 cm⁻¹ (approximately 987 nm) in the near-infrared spectrum is indicative of similarly annealed type I diamonds, as are the graphitized "feathers" seen with a gemological microscope. Some variations were also observed in the mid-infrared range.

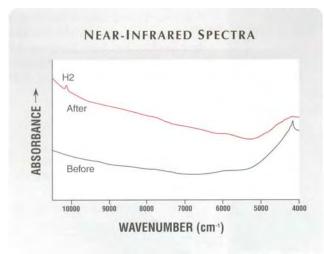
According to Dr. Frushour, the normal annealing temperature used at their facility is generally above 1800°C, with pressure in the range of 5 to 6 GPa. The diamonds annealed range from under 1 ct to over 10 ct. Phoenix Crystal Corp. is not marketing diamonds processed at its facility, but is offering the HPHT service to the trade. We do not know the exact number of commercial HPHT annealing facilities in the U.S., but we suspect that it is very small.

Wuyi Wang

Challenges in Recognizing a Color Attribute's Effect on Color Grading Fancy Colors

GIA fancy grades for colored diamonds represent a *range* of appearances based on the combination of a color's attributes (hue, tone, and saturation). If a diamond is located near the boundary of its grade range, subtle differences between it and another

Figure 3. Absorption spectra in the near- and mid-infrared range of the diamond shown in figure 2, before and after treatment, provide evidence of HPHT annealing.



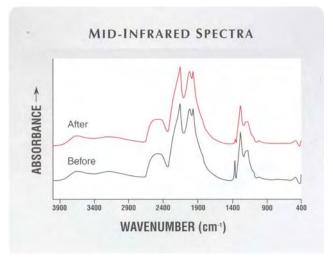


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Figure 4. These three diamonds (1.03–2.80 ct) are in the same hue range and of approximately the same saturation. The differences in appearance are due to variations in tone (i.e., relative lightness or darkness), all of which are within the range that would be described in GIA's colored diamond color grading system as Fancy Vivid yellow-orange.

diamond in one or more of these attributes can result in different grades. For example, in this section of the last issue (Spring 2002 GTLN, p. 80), we reported on two blue diamonds that had such a result based on their difference in tone (lightness to darkness). However, there are also situations in which recognizable differences in an attribute may *not* affect the grade because the different appearances occur *within* the established range (i.e., if the range of differences does not cross a boundary, the grade will be the same).

Even in situations where a grade is not affected, understanding which attribute is causing an appearance difference is important because it is not uncommon for a difference in one attribute to be confused with that in another. As a result, some observers interpret differences in an attribute, such as tone, as a difference in saturation or hue. If, for example, two diamonds of different tone were both Fancy yellow and the difference was confused with saturation, they might be thought of as Fancy and Fancy Intense; if hue, the perception might be yellow and orangy yellow. Consistent evaluations are very difficult without a systematic approach, using references of known location in color space (such as diamonds with established locations in grade ranges), and they require a familiarity with the way colors change with different attributes that can only be attained by observing considerable numbers and varieties of colored diamonds.

A group of colored diamonds recently submitted to the East Coast laboratory highlights the challenges of identifying the cause of appearance differences without a consistent approach and awareness of how colors change. The three diamonds in figure 4 appear slightly different in hue and saturation, which could lead one to believe they are of different fancy grades and hues. When observed under controlled conditions with known references, however, all were found to be in the same hue range and of the same approximate saturation. The perceived differences are due to differences in tone within an area of the GIA color space that does not cross a grade or hue boundary. In this case, all three diamonds received the same color grade and description: Fancy Vivid yellow-orange.

John M. King

Diamond "Pearl" Necklace

The 1990s saw increasing interest in antique-cut diamonds such as briolettes, rondelles, and rose, old European and old mine cuts. Due to their old-fashioned look, diamond beads have also enjoyed a dramatic rise in popularity, even though they are "without historic precedent" (E. Misiorowski, "Jewelry of the 1990s," Winter 2000 Gems & Gemology, pp. 398–417).

It was, therefore, not surprising last spring when the East Coast lab received a double-strand necklace of diamond "pearls" for determination of color origin (see figure 5). These spherical diamond beads, first seen by one of our staff members as a commercial product at the 1997 Tucson gem shows, owe their shape to a combination of physical and chemical processes. Ground to rough rounds, they acquire their shiny polish by selective dissolution as they "cook" in sodium carbonate in an inert atmosphere at approximately 800°C ("Diamond 'pearls'," Spring 1997 Gem News, pp. 60–61).

The two strands consisted of numerous variously colored round diamond beads graduated in size from approximately 1.75 to 10.00 mm. The client had requested that we test the origin for each color represented in the necklace. As a result, we randomly selected and tested a total of five beads: yellow, dark gray, reddish brown, black, and near colorless. These sample beads ranged in diaphaneity from transparent to opaque. All had refractive indices that were over-the-limit (OTL) of the standard refractometer and had a hardness greater than 9 on the Mohs scale. Only the transparent, near-colorless bead showed a spectrum with the desk-model spectroscope—a 415 nm line.

The dark gray and black beads were inert to both long- and short-wave UV. The yellow bead fluoresced weak orange to long-wave and very weak orange to short-wave UV. The reddish brown bead fluoresced patchy medium-to-strong blue to long-wave



Figure 5. This double-strand necklace of graduated diamond "pearls" was sent to the East Coast lab for determination of color origin. The beads ranged from approximately 1.75 to 10.00 mm.

Internal Laser Drilling Update

In the Summer 2000 issue of Gems & Gemology, S. McClure et al. documented a new laser treatment ("A new lasering technique for diamond," pp. 138-146). The purpose of this treatment, as with typical laser drill holes, is to provide a path for acid solutions to bleach dark, totally internal inclusions. By focusing a laser beam on or near such an inclusion, the technician creates feathers (cleavages) or enlarges existing ones so that they extend to the surface of the faceted diamond. Within the feathers one can see irregular lines or channels that have been left behind by the laser. These channels are typically black or white (figure 6), with a sugary or frosted appearance. Occasionally, we see larger areas that contain numerous small feathers in a steplike progression to the surface. These also may have a white, sugary look (figure 7). In all three of these examples, the treatment is fairly obvious when the diamond is examined with magnification.

Since the 2000 article, we have seen many variations in the appearance of this internal laser drilling.

and patchy medium orangy yellow to short-wave UV. Blue fluorescence was seen in the near-colorless bead in medium and weak strengths with long- and short-wave UV, respectively. Magnification, with the help of fiberoptic lighting, revealed numerous feathers and fractures in each bead. The dark gray bead also contained many whitish clouds of pinpoint inclusions. The reddish brown bead owed its color to orangy red iron staining in many of its surface-reaching fractures. The black bead contained numerous dark crystals and pinpoints.

In summary, standard gemological testing revealed a combination of properties and features that both proved the identity of the beads as diamond and confirmed that the colors in all five beads were of natural origin.

Wendi Mayerson

Figure 6. Laser drilling channels typically are black or white. Left—a nest of black channels in the center of the image is surrounded by a barely visible transparent feather, also created by the laser. Right—the white channels may appear sugary or frosted. Magnified 63× and 45×, respectively.





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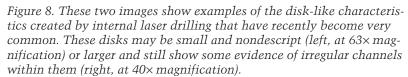
Figure 7. Internal laser drilling can create a series of small step-like feathers that connect an inclusion to the surface of the diamond, often leaving behind a white area with a sugary texture in the center of the feathers.

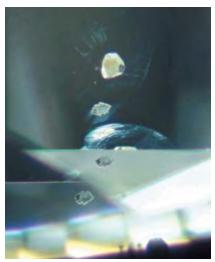
Magnified 37×.

Recently, in our laboratories on both coasts, we have encountered a number of feathers that have small, white, disk-like areas with irregular outlines and a sugary texture, rather than the channels described above (figure 8). These disk-like features are located in the same areas of the fractures where we would expect to see the more obvious irregular channels. The "disks" are visible when one looks perpendicular to the plane of the feather, but they are practically invisible when the viewing direction is more parallel to the feather.

The fact that we have seen these unusual features in a large number of diamonds clearly indicates that they are being created by design. These disk-like characteristics are more difficult to recognize than the irregular channels noted earlier in these laser-induced feathers, which makes this treatment more challenging to detect. It is, therefore, very important to use high magnification and various light sources to check all surface-reaching fractures that extend from totally internal inclusions.

Vincent Cracco and Halina Kaban





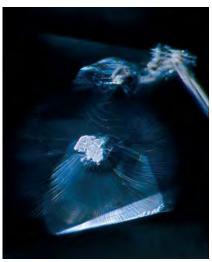




Figure 9. This 3.58 ct heart-shaped diamond was graded Very Light pink in a standard laboratory viewing environment. When exposed to short-wave UV radiation, however, it became F color. Its appearance with standard lighting provided no clue to its unusual luminescence behavior.

Unusual "Pink"

Some diamonds display a change of color when exposed to light or heat. The most frequently encountered diamonds exhibiting this phenomenon are chameleon diamonds, which change from yellow to yellowish green or green when exposed to light after storage in the dark. Gentle heating of this type of diamond produces a temporary bright yellow color. In addition, some Argyle pink diamonds have been observed to change from pink to brownish pink or brown when exposed to ultraviolet radiation. In both cases, the color change is temporary, with the diamond quickly returning to a stable color state once it is removed from the environment that produces the temporary color.

Recently a 3.58 ct Internally Flawless type IIa diamond was submitted to the West Coast lab for investigation of its color-change phenomenon. When examined in the lab's standard viewing environment, this particular diamond was given a color grade of Very Light pink (figure 9). When it was exposed to shortwave UV radiation, however, the gem



Figure 10. GE has started commercial production of synthetic jadeite in a range of colors and qualities. The samples shown here weigh 1.54–4.06 ct.

changed to an F color. The pink could be restored by exposure to either longwave UV or daylight.

In contrast to the above-mentioned more common "chameleon" change in color, this diamond showed no change during or immediately after storage in the dark. Thus, the diamond remained pink if it was stored as a pink diamond or colorless if it was stored as a colorless diamond. The pink color "returned" slowly after exposure to long-wave UV radiation or daylight.

Note that, in addition to producing a strong orange fluorescence when exposed to long- or short-wave UV radiation (which is typical for some type IIa pink diamonds), the 3.58 ct diamond also exhibited orangy red fluorescence to the wavelengths of blue light. Depending on the initial diamond color, the excitation wavelength, exposure duration, and number of exposures, up to four different phosphorescence states were observed in this diamond:

- 1. Diamond color "pink": medium orange phosphorescence lasting 3 to 15 seconds
- Diamond color changing from "pink" to "near colorless": no phosphorescence
- 3. Diamond color "near colorless": strong greenish blue phosphores-

- cence lasting 2 to 60 minutes or more
- 4. Diamond color changing from "near colorless" to "pink": weak orange phosphorescence lasting 1 to 2 seconds, changing to moderate greenish blue phosphorescence lasting 5 to 30 seconds

Although the observation of phosphorescence typically requires a darkroom environment, the strong greenish blue phosphorescence was visible for quite some time even in dim light under certain exposure conditions.

This is one of the very rare colorchange "pink" diamonds in which

Figure 11. The finest green GE synthetic jadeite, such as this 6.11 ct cabochon, has an appearance very similar to "Imperial" jade.



the illumination history of the diamond can produce different fluorescence and phosphorescence phenomena and result in different color states. For these types of diamond, a careful neutralization of the temporary phenomenon is necessary before a final color grade can be determined. A more detailed report on the unusual properties of this particular diamond is currently underway.

Shane Elen and Ronnie Geurts

High-Quality SYNTHETIC JADEITE from General Electric

General Electric (GE) Gem Technology has developed a proprietary process for manufacturing synthetic jadeite. GE shared a small number of early production samples with the GIA Gem Trade Laboratory. The samples ranged in color and quality (figure 10), but the finest green material rivaled "Imperial" jade in appearance (see figure 11). Although GE has not yet released details about the new technology, a GE spokesperson did tell us that it is achieved in a high-pressure environment.

The gemological properties of the small number of samples we have tested to date overlap those of natural jadeite. For example, the 6.11 ct cabochon $(13.60 \times 12.00 \times 4.00 \text{ mm})$ shown in figure 11 had a refractive index of 1.66, measured on the flat base, and a specific gravity of 3.34, determined hydrostatically. Using a desk-model spectroscope, we observed a 437 nm line along with three strong chrome lines at approximately 630, 655, and 690 nm. We are currently analyzing the results of additional spectroscopic testing, as we work with GE to determine identification criteria for this new product.

GE research on jadeite synthesis extends back 20 years (see, e.g., R. C. Devries and J. F. Fleischer, "Synthesis of jadeite for jewelry," *Symposium Proceedings of the Materials Research Society*, Vol. 22, 1984, pp. 203–207; and K. Nassau and J. E. Shigley, "A



Figure 12. These two sapphires (2.74 and 2.23 ct) were submitted to the laboratory together. The lighter one (left) showed evidence of standard diffusion treatment with titanium. The darker stone was colored by a very shallow surface layer that contained cobalt.

study of the General Electric synthetic jadeite," Spring 1987 *Gems & Gemology*, pp. 27–35). GE plans to publish a detailed article on the characteristics of this new material in the next several months.

Cobalt-"Diffused" SAPPHIRE

Since the introduction of diffusiontreated blue sapphire in the early

Figure 13. When the cobalt-colored sapphire was immersed in methylene iodide and examined with magnification in diffused light, irregularities in the color were evident. Note the light-colored spots and paler facet junctions.



1980s, many articles have been written about such stones and their identification (see, e.g., R.E. Kane et al., "The identification of blue diffusion-treated sapphires," Summer 1990 *Gems & Gemology*, pp. 115–133). The blue color was produced by diffusing titanium into the surface of the sapphire.

About the time the aforementioned article was published, we were also shown some experimental sapphires in which cobalt was used instead of titanium as the diffusing agent. This produced a vibrant blue color, but the color layer was so thin that no real penetration of the corundum could be seen at 60× magnification. In the years that followed, we never saw this product sold on the market.

A short time ago, two blue sapphires (2.23 and 2.74 ct) were submitted to the West Coast laboratory for identification (figure 12). The 2.74 ct sapphire was easily proved to be diffusion treated, as it possessed the characteristics typical of this process: high relief in methylene iodide, concentrations of color at facet junctions, and patchy coloration.

The 2.23 ct sapphire, however, was much different. The color of this stone was darker and more saturated

than the first. The refractive index was over the limits of the refractometer, and the desk-model spectroscope showed three broad bands that are typically associated with cobalt. Microscopic examination did not reveal any inclusions. However, there were numerous spots of lighter color on the surface, and some facet junctions appeared paler than the surrounding facets (figure 13).

Because the refractive index of this stone was so high, we used the laser Raman microspectrometer to determine its identity. The Raman spectrum matched that of sapphire. With EDXRF, we then determined that there was a very high concentration of cobalt at the surface of the stone.

It was clear that the high cobalt concentration at the surface was the cause of the extremely high R.I. readings. We have encountered this before, particularly with red diffusion-treated corundum that has very high concentrations of chromium at the surface and, more recently, with blue-to-green topaz that derives its color from a cobalt-rich surface layer (see, e.g., S. F. McClure and C. P. Smith, "Gemstone enhancement and detection in the 1990s," Winter 2000 Gems & Gemology, pp. 336-359). The blue-to-green treated topaz is often referred to in the trade as "diffusion-treated," even though it has not been proved that diffusion is actually taking place.

As with the topaz, the depth of the blue surface coloration on the 2.23 ct sapphire could not be seen. The layer was obviously very shallow, with even small chips and scratches penetrating through to the colorless core. Thus, we could not be certain whether the color of the sapphire was due to diffusion treatment, or the product of some other type of surface reaction with the cobalt.

The submission of this cobalt-colored sapphire to the laboratory raises some questions about whether this material has now entered the market-place. Since we saw only the one stone, we do not know the answer at this time.

SFM



Figure 14. Advanced testing revealed that these three beads (each approximately 14.00 × 10.00 × 5.00 mm) were fashioned from the rare gem material schlossmacherite.

SCHLOSSMACHERITE, Yellow-Green Beads

In early 2002, staff members in the West Coast laboratory were challenged to identify several opaque yellow-green beads that had been submitted for analysis. The material had been represented as variscite, an essentially colorless aluminum phosphate that appears green-blue when iron and chromium are present. Green-blue variscite is often used as an ornamental stone, or dyed and sold as a substitute for turquoise.

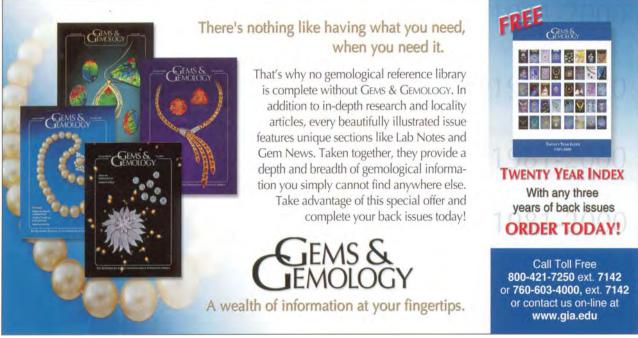
Because these beads were an unusual yellow-green (see, e.g., figure 14), our client had questioned their identity as variscite. The lenticular beads, which measured approximately $14.00 \times 10.00 \times 5.00$ mm, were evenly colored and appeared to have been fashioned from the same piece of material. A few beads also showed some prominent darker green "veins." When we examined the beads with a microscope at standard 10× magnification, we noticed that the material had a fine-grained, almost colloidal-appearing structure. We also observed that the darker-appearing "veins" were actually a finer-grained phase of this material. Despite the poor polish, we were able to determine a vague spot refractive index reading of 1.57. The specific gravity was measured at 2.35. The beads did not show an absorption spectrum or any reaction to long- or short-wave ultraviolet radiation. Since these properties are not within the reported range for variscite, we turned to advanced testing methods to identify the beads.

With the client's permission, Dino DeGhionno performed an X-ray powder diffraction analysis and determined that the beads had been fashioned from schlossmacherite, a sulfate-arsenate member of the beudantite group. To confirm the identity of this rare material, Sam Muhlmeister performed EDXRF qualitative chemical analysis. The analyses revealed that the material primarily contained aluminum, sulfur, and arsenic, as well as calcium, copper, and other minor trace elements, which substantiated the identification as schlossmacherite.

This rare mineral was named after the dean of German gemology, Prof. Dr. Karl Schlossmacher. Originally found in Guanaco (Chile), it was first introduced by K. Schmetzer and H. Bank in 1979 (Zeitschrift der Deutschen Gemmologischen Gesellschaft, Vol. 28, No. 3, pp. 131–133) and described as a new mineral in 1980 by K. Schmetzer et al. (Neues Jahrbuch für Mineralogie Monatshefte, Vol. 1980, No. 5, pp. 215–222). KNH

PHOTO CREDITS

Maha Tannous—figures 1, 9, and 12; Elizabeth Schrader—figures 2, 4, 5, 10, and 11; Vincent Cracco—figures 6, 7, and 8 (left); Shane McClure—figures 8 (right) and 13; Don Mengason—figure 14.



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