NATURAL-COLOR PURPLE DIAMONDS FROM SIBERIA

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Twelve natural-color purple diamond crystals from Siberia, and seven round brilliants that were faceted from some of these crystals, were studied using spectroscopic methods to better understand their color and its causes. Aspects of their color and the various structural defects in these purple diamonds are due to their post-growth plastic deformation in the earth. All the samples exhibited prominent parallel planar lamellae along which the purple color was concentrated.

Predominantly purple diamonds are quite rare, and few have been documented in the literature. While their color is thought to be due to plastic deformation, the exact nature of the defects responsible for purple coloration is not yet fully understood. With the availability of treated-color purple-to-red diamonds in the marketplace (e.g., Wang et al., 2005), it is important for gemologists to be able to properly identify natural-color purple diamonds, despite their rarity.

Natural-color purple diamonds are usually described with various hue modifiers such as pink, gray, and brown (Hofer, 1998). In the few published gemological reports on purple diamonds, their geographic origin is usually not mentioned (see, e.g., Liddicoat, 1977; Hargett, 1990; Hofer, 1998; Moses et al., 2002). However, one recurring source is Russia's Siberia region (see Federman, 1995). Purple diamonds are occasionally found in all the Siberian deposits explored to date, but they are most often recovered from the pipes of the Mir kimberlite field (comprising the Dachnaya, Internatsional'naya (also spelled Internationalaya), Mir, and Sputnik mines). In those mines, they typically account for 1% of all diamonds from the deposits, although as much as 6% of some parcels have been described as purple (Gnevushev et al., 1961; Orlov, 1977; Zintchouk and Koptil', 2003). The majority of purple diamonds from the Siberian deposits are pale, but crystals with more highly saturated purple colors are sometimes found.

To study the cause of color in diamonds with a dominant purple hue, we first characterized 12 purple crystals from Siberia using various spectroscopic methods. The results of earlier electron paramagnetic resonance (EPR) studies of structural deformation defects in these same purple diamonds were reported by Mineeva et al. (2007). That article showed that purple diamonds contain thin mechanical microtwins, which are parallel to octahedral planes and were previously interpreted as slip planes. Optical dichroism was also observed in these purple diamond crystals; this phenomenon is unusual for crystals with cubic symmetry and was investigated using a special spectroscopic technique (Konstantinova et al., 2006). The present article reports the gemological characteristics of faceted purple diamonds and the results of ultraviolet-visible-near infrared (UV-Vis-NIR), Fourier-transform infrared (FTIR), and photoluminescence (PL) spectroscopy on a number of rough and/or faceted samples. For convenience, the diamonds examined

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Figure 1. This group of purplish pink to gray-purple diamonds (0.28–0.85 ct) is representative of similarly colored material recovered from mines in the Mir kimberlite field in Siberia. These particular samples, which were studied for the present report, came from the Internatsional'naya pipe. Photo by C. D. Mengason.

here are usually referred to simply as "purple" diamonds, although the GIA color descriptions of the faceted samples include modifying colors.

BACKGROUND

Over the past 50 years, extensive research has characterized the color centers in natural diamonds (see, e.g., Fritsch, 1998; Collins, 2001). However, the origin of purple coloration has not yet been fully established. The spectra of purple diamonds exhibit a broad absorption band centered at 550 nm (Collins, 1982; Moses et al., 2002), but the cause of this band remains unclear. The band is also observed in the spectra of other fancy-color diamonds, such as red (Shigley and Fritsch, 1993), pink, and pink-brown (King et al., 2002). However, in pink-to-red diamonds, the 550 nm band is typically accompanied by an intense 390 nm band and a strong N3 system (zero-phonon line [ZPL] at 415 nm). The absorption spectra of red diamonds also contain the H3 system, with its ZPL at 503.2 nm (Shigley and Fritsch, 1993). In pink-brown diamonds, the 550 nm band is superimposed on an absorption spectrum that continuously increases in intensity from the red to the blue region of the visible spectrum, which causes the brown coloration. Faceted purple diamonds without a modifying color typically do not show these other features (Moses et al., 2002).

Although several theories about the origin of the purple coloration have been proposed (e.g., Raal, 1958; Gnevushev et al., 1961; Taran et al., 2004), Orlov (1977) showed that purple (as well as pink, in most cases) in natural diamonds is restricted to a set of parallel octahedral planes, which were referred to as slip planes. These "planes" appear to be a result of natural post-growth plastic deformation of the diamonds, which probably occurred during their transportation from the mantle to the earth's surface by kimberlite magma.

MATERIALS AND METHODS

Twelve natural diamond crystals with a dominant purple color were selected for study by rough-diamond graders of Alrosa (Russia's largest diamond mining and processing company), according to the De Beers color classification system for rough diamonds. These crystals (0.4–1.4 ct) were recovered from the Internatsional'naya kimberlite pipe. Plates of 1.6 mm thickness were cut from two crystals so that the colored lamellae were perpendicular to a cutting plane. All rough crystals and the plates were examined with a binocular microscope using daylight-equivalent illumination. Following spectroscopic analysis (Konstantinova et al., 2006; Mineeva et al., 2007), 10 of the crystals were faceted as round brilliants.

Gemological examination of seven of the round brilliants (figure 1) was conducted at GIA. Experienced graders determined color grades using the standard conditions and methodology of GIA's color grading system for colored diamonds (King et al., 1994). Internal features were observed with a gemological microscope using brightfield and darkfield lighting techniques. Reactions to UV radiation were checked with a conventional four-watt combination long-wave (365 nm) and short-wave (254 nm) lamp. The seven diamonds were also examined with a DiamondView deep-UV (<230 nm) luminescence imaging system. A desk-model spectroscope was

Figure 2. The purple diamond crystals studied exhibited strong parallel color banding, so that in one orientation (left) the color was unevenly distributed, while in the other (right) it appeared much more uniform. Photos by M. A. Bogomolov.

used to view absorption features at room temperature in the visible range.

Infrared spectra of the seven faceted stones were obtained at room temperature at GIA with a Thermo-Nicolet Nexus 670 FTIR spectrometer equipped with KBr and quartz beam splitters. Spectra were collected in the mid-infrared (6000–400 cm^{-1}; 1 cm^{-1} resolution) and near-infrared (11000–4000 cm^{-1}; 4 cm^{-1} resolution) ranges. A 6× beam condenser was used to focus the beam through the girdle region of the samples, and a total of 1,024 scans per sample were collected to improve signal-to-noise ratios.

Low-temperature photoluminescence spectra of the seven faceted samples were also collected at GIA, using a Renishaw 1000 Raman microspectrometer with an Ar-ion laser at two different laser excitations (488.0 and 514.5 nm). The samples were cooled by direct immersion in liquid nitrogen.

UV-Vis-NIR absorption spectroscopy of the 12 crystals was performed in Russia. Spectra were recorded over the 300–800 nm range with a Perkin Elmer two-beam Lambda 9 spectrometer at liquid nitrogen (77 K) temperatures using a fabricated cryostat mounted in the spectrometer. The spectral resolution was ~0.2 nm.

RESULTS

Visual Observations of the Crystals and Polished Plates. The 12 crystals were purple, with pink, gray, and brown modifiers, and had weak-to-moderate saturation. All displayed an octahedral habit; most also showed thin, rough parallel straight striations with subordinate development of shield-like striations. This indicated that they experienced only minor post-growth dissolution (Orlov, 1977). They also exhibited varying degrees of fracturing or cleavage.

For crystals with native flat faces, we readily observed that the purple coloration was restricted to thin bands parallel to octahedral planes (figure 2, left); between these planes, the diamonds were nearly colorless, with a very light yellowish or pinkish brown hue. When the crystals were examined in a direction perpendicular to these planes, the purple color

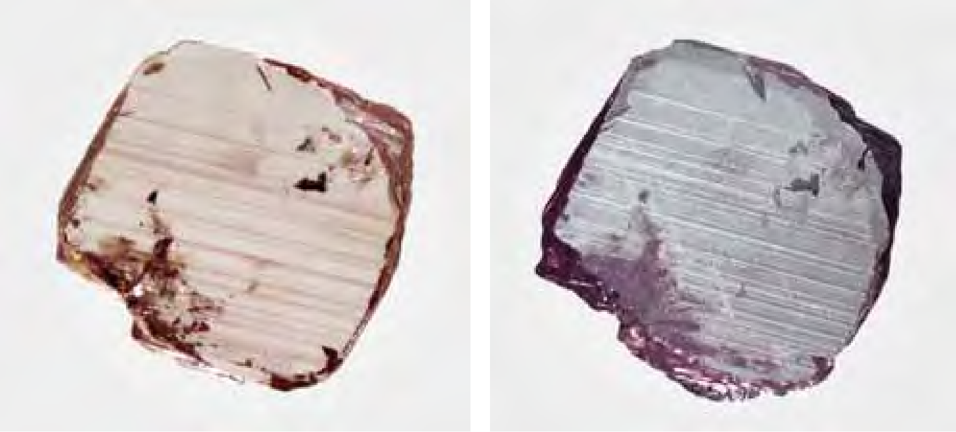


Figure 3. This flat plate (4 × 4 × 1.6 mm) cut from one of the diamond crystals shows purple coloration concentrated along thin parallel lamellae (transmitted light, left). When viewed with reflected light (right), the purple lamellae appear as mirror-like reflective sheets. Photos by M. A. Bogomolov.

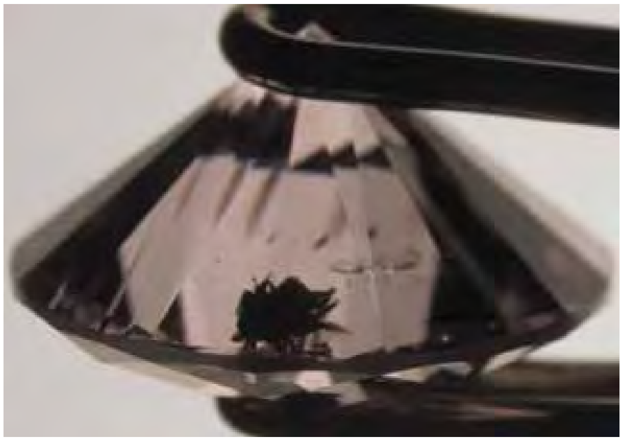


Figure 4. This 0.85 ct Fancy Light gray-purple round brilliant exhibits a dark inclusion of unknown identity. The presence of this inclusion resulted in an I_2 clarity grade. Photomicrograph by J. E. Shigley; magnified 5×.

Figure 5. Intersecting sets of purple and brown lamellae can be seen in this 0.43 ct Fancy brownish purple-pink diamond. Photomicrograph by J. E. Shigley; magnified 5×.

appeared evenly distributed (figure 2, right). Ten of the 12 crystals contained dark opaque inclusions of undetermined identity.

Study of the flat plates in transmitted light revealed that the purple planes were actually thin lamellae (~0.1 mm in thickness and about 0.1–0.5 mm apart). Typically, these were unevenly distributed throughout the volume of the crystal (figure 3). At crystal surfaces, the colored lamellae were truncated by chains of trigons, a feature that has been reported previously (e.g., Orlov, 1977).

Gemological Examination of the Faceted Samples. The seven polished diamonds were color graded as follows: one Fancy Light gray-purple, three Fancy pink-purple, one Fancy purple-pink, one Fancy brownish purple-pink, and one Fancy Light purplish pink (again, see figure 1). The difference between the color classification of the rough crystals (as predominantly purple) and the color grades of the diamonds faceted from them (three of which were predominantly pink) may be due to some disagreement between the two grading systems in the pink-to-purple continuum. Additionally, the cutting process may play an important role. Not only have significant hue changes from the original rough to the faceted stone been reported in colored diamonds, but the orientation of the table in a color-zoned diamond is also key to its face-up appearance (King et al., 2002).

The clarity grades of the seven round brilliants all fell into the "I" (included) category due to the presence of cleavages and dark inclusions (e.g., figure 4; we did not identify the inclusions). Additionally, we saw clouds of fine particles in most of the samples. All of the diamonds displayed parallel lamellae that appeared purple or brownish purple (figures 5 and 6) and were

Figure 6. In this 0.53 ct Fancy purple-pink diamond, parallel planes of purple lamellae are separated by zones that are near-colorless to light brown. Photomicrographs by J. E. Shigley; magnified 5× (left) and 10× (right).

Figure 7. These DiamondView images reveal the banded appearance of blue fluorescence in the faceted diamonds. In the 0.39 ct sample on the left, the complex sculptured shape of the banding is indicative of the diamond's growth history. The 0.85 ct diamond on the right shows weak green luminescence (due to the H3 defect) along traces that correspond to the purple lamellae. Photos by C. M. Breeding.

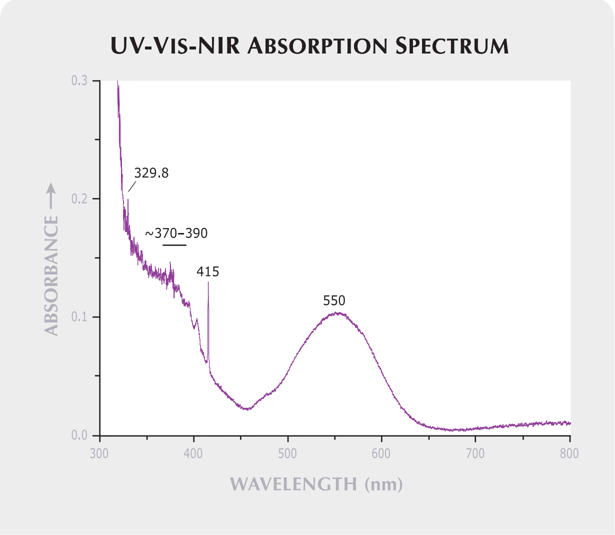
more evident in the strongly colored samples (in some instances, these colored lamellae were easily seen with the unaided eye or low magnification; again, see figures 5 and 6). When the diamonds were examined with fiber-optic illumination, green luminescence was commonly visible along the colored planes.

None of the diamonds displayed distinct absorption features when examined with a desk-model spectroscope. When observed in a darkened room, the diamonds fluoresced very weak to weak yellow or were inert to long-wave UV radiation, and fluoresced very weak to weak orangy yellow or were inert to short-wave UV. The DiamondView revealed bright blue luminescence patterns that depicted the complex growth structure of these diamonds (figure 7, left). In addition, H3-related green luminescence banding was seen following the purple lamellae (figure 7, right).

UV-Vis-NIR Absorption Spectroscopy. Figure 8 shows the absorption spectrum at 77 K for a crystal with polished octahedral faces that were parallel to the colored lamellae. The spectrum exhibited increasing absorption below 450 nm and a broad absorption band centered at about 550 nm, which contributes to the purple coloration. A broad band at 370–390 nm and the N3-related ZPL at 415 nm (with weak phonon sidebands at 383, 394, and 403 nm) were superimposed on the increasing absorption below 450 nm. With the exception of the most intensely colored crystal, the N3 center was observed in all the purple diamonds studied, but in very low intensities (sometimes near the detection limit of the spectrophotometer). The exact position of the maximum for the 370–390 nm band was difficult to determine because of overlapping absorption below 450 nm. We believe that it corresponds to a band at 390 nm observed in pink diamonds (Collins, 1982; King et al., 2002). On the background of the absorption below 450 nm, a sharp ZPL was observed at 329.8 nm; this is due to the N5 center, an electronic transition of the A aggregate of nitrogen (Zaitsev, 2001).

The intensity of the 550 nm band changed noticeably over a series of spectra as the angle between the sample and the beam changed. This phenomenon may be due to the localization of purple coloration within the thin lamellae. For accurate studies of this unusual phenomenon, absorption spectra were recorded using polarized light (Konstantinova et al., 2006). That research found that these purple diamond crystals possessed dichroism (i.e., different absorption of polarized light depending on crystallographic direction), which

Figure 8. This UV-Vis-NIR absorption spectrum of a purple crystal exhibits increasing absorption below 450 nm, the N5 system (ZPL at 329.8 nm), a band at about 380 nm, the N3 system (ZPL at 415 nm), and a broad band centered at about 550 nm.



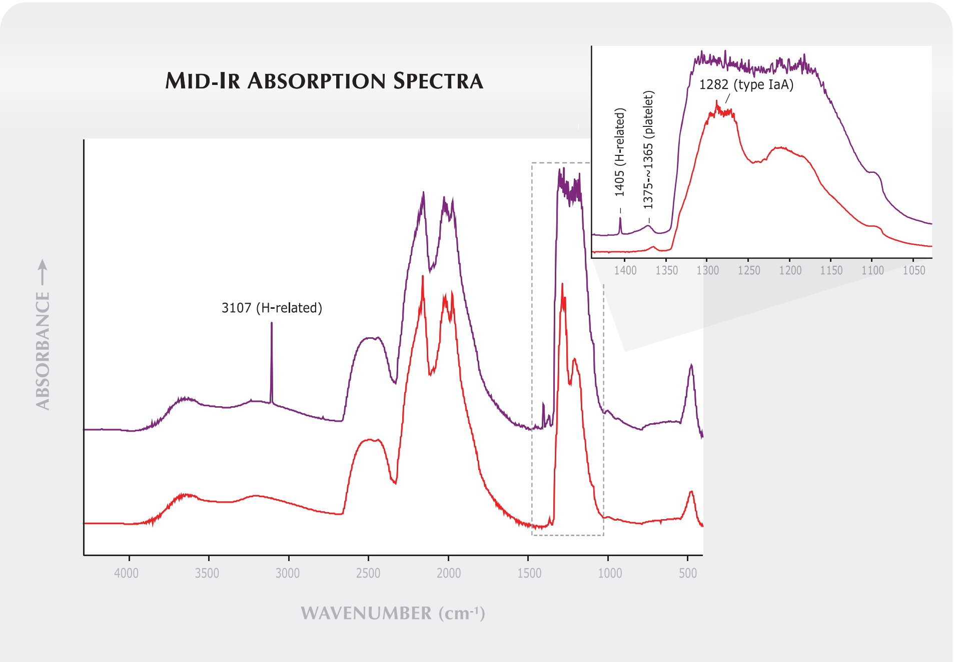


Figure 9. These mid-infrared spectra from two faceted samples (top, 0.53 ct Fancy purple-pink; bottom, 0.85 ct Fancy Light gray-purple) reveal spectral features that are characteristic of type IaA diamonds, which may include hydrogen-related defects (3107 cm^{-1}), platelet features (~1375–1365 cm^{-1}), B-aggregated nitrogen (1175 cm^{-1}), and the dominant A-aggregate of nitrogen (~1282 cm^{-1}). Spectra are offset for clarity.

may be caused by low-symmetry defects in the diamond structure formed during plastic deformation.

Infrared Spectroscopy. All the faceted diamonds proved to be type IaA. Figure 9 presents typical mid-infrared spectra for two of them. Absorption features in the one-phonon region (1400–1000 cm^{-1}) indicate the presence of structural defects involving nitrogen impurity atoms, such as A aggregates along with minor amounts of B aggregates and platelets (polyatomic segregations up to a few microns in size in cubic planes of the diamond lattice, which involve nitrogen and/or carbon atoms; Zaitsev, 2001). Single substitutional nitrogen (associated with type Ib diamond) was not detected in the IR spectra of any of these samples, although it was observed in very low concentrations (20–60 ppb) in the EPR spectra of all the diamond crystals (Mineeva et al., 2007).

Five of the diamonds showed the 4496, 3107, and 1405 cm^{-1} peaks that are associated with a hydrogen structural impurity (see Zaitsev, 2001). However, these peaks were not all present in the IR spectra of the other two samples (e.g., the bottom spectrum in figure 9). In addition, six of the seven faceted diamonds showed H1b and H2 defect centers, and a few showed amber centers in the near-infrared range (figure 10).

Photoluminescence Spectroscopy. The PL spectra revealed the presence of several defect centers (figure 11). The H3 center (503.2 nm) occurred in all the faceted samples (although it was not seen in the UV-Vis-NIR spectra, likely due to its low concentration). Other centers, including those located at 612 nm (unknown origin), 693 and 700 nm (commonly attributed to nickel and/or hydrogen impurities), 787 nm (unknown origin, although possibly related to hydrogen), and 793 nm (related to nickel) were present in most of the diamonds (see Zaitsev, 2001).

DISCUSSION

The results of this gemological and spectroscopic study of natural-color purple diamonds, when considered with those of previous studies on the same samples (Konstantinova et al., 2006; Mineeva et al., 2007), provides a comprehensive characterization of their defect centers and their relation to the purple color.

Nitrogen-Related Defects. Most of the defects were formed during post-growth plastic deformation, which caused a transformation of some of the most abundant defects in the purple crystals—the A centers, which consist of two nitrogen atoms in nearest-neighbor structural sites. This transformation resulted in the formation of many new centers, such as H3, H2, H1b, amber centers, W7, and M2 defects (the latter two were revealed with EPR spectroscopy; Mineeva et al., 2007). Of the less-commonly described defects in the gemological literature, the W7 center represents a ring consisting of four carbon atoms and two nitrogen atoms on the opposite sites, with one of the nitrogen atoms in the ionized state

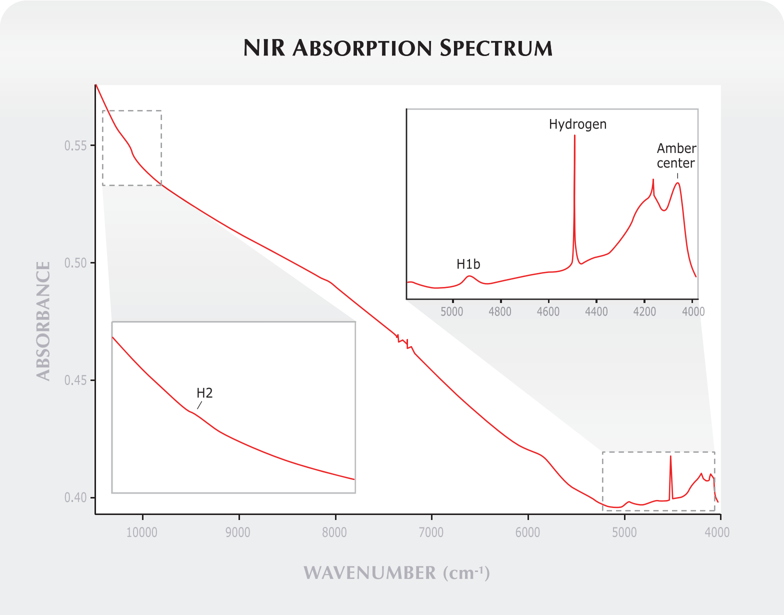
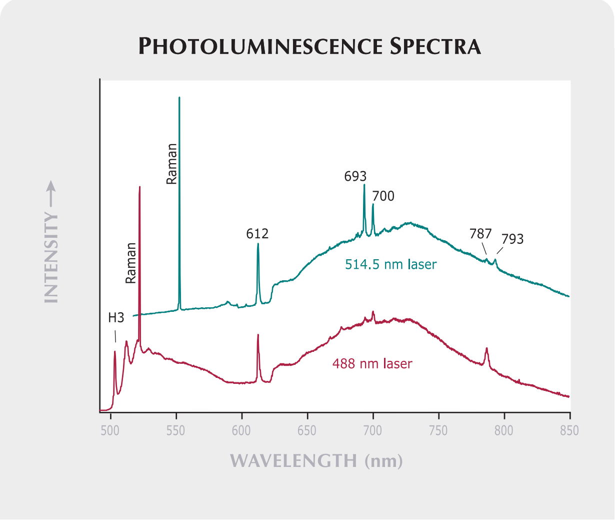


Figure 10. This near-infrared spectrum of a 0.53 ct Fancy purple-pink diamond reveals features due to H2 (~10130 cm^{-1}) and H1b (~4935 cm^{-1}) defects, as well as hydrogen impurities (4496 cm^{-1}) and the amber center (~4300–4000 cm^{-1}).

N$^+$. The amber center is considered the optical analogue of the paramagnetic W7 center (Massi et al., 2005). The M2 center involves two nitrogen atoms and eight carbon atoms, which form an octahedron-like polyhedron with nitrogen atoms at opposite corners. Among these various deformation-related centers, the M2 is the most important from a gemological standpoint. This center is characteristic only for diamonds with absorption spectra that exhibit the 550 nm band. Additionally, both the M2 center and the purple coloration were concentrated within the microtwin lamellae (Mineeva et al., 2007).

Figure 11. The photoluminescence spectra of this 0.39 ct Fancy Light purplish pink diamond, recorded with 488 and 514.5 nm laser excitations, revealed features due to several defect centers at 503.2 (H3), 612, 693, 700, 787, and 793 nm.



H1b, H2, and H3 centers may be produced in diamond by artificial irradiation and subsequent heating (Collins, 2001). The H3 center also has been seen in many natural plastically deformed brown diamonds (Collins, 1982), and it likely accounts for the green luminescence observed in our samples (again, see figure 7, right). However, only a few occurrences of the H2 center in natural plastically deformed diamonds have been reported (e.g., De Weerdt and Van Royen, 2001). Natural plastic deformation in the earth may generate vacancies in the diamond structure, including the H1b, and this may be similar to the processes of laboratory irradiation and subsequent annealing (see, e.g., Collins, 2001).

Effects of Lamellae and Dichroism on Coloration. This and previous studies by many of the same authors have revealed the complicated origin of color in purplish pink to purple diamonds. The coloration is strongly restricted to thin microtwin lamellae, so the color intensity may appear quite different depending on whether a stone is viewed perpendicular or parallel to the lamellae. Additionally, the purplish pink to purple color centers exhibit optical dichroism (Konstantinova et al., 2006). To the best of our knowledge, the absorption anisotropy of the 550 nm band in diamond has previously been reported only by Zaitseva and Konstantinova (1989).

Figure 12. Natural-color purple diamonds, among the rarest of colored diamonds, owe their purple hue to post-growth plastic deformation. These purple and pink-purple diamonds from Siberia weigh 0.73, 0.62, and 0.63 ct, respectively. Photo by M. A. Bogomolov; courtesy Rony Carob Ltd.

Purplish pink to purple diamonds have been observed to shift color when viewed in various lighting conditions (Hofer, 1998) and after cutting (Moses et al., 2002). Hofer (1998) attributed the change in color to differences in the spectral composition and color temperature of the light source. The occurrence of dichroism in these diamonds implies that the viewing direction and the degree of light polarization may also be important. This dichroism may have been a cause of the predominant pink hue in some of the faceted diamonds in this study. In addition, it may be a characteristic feature of not only purple diamonds, but also other diamonds that receive their color from plastic deformation, such as red, pink, and some of the more common pink-brown stones. As with purple diamonds, the optical spectra of those diamonds also show the 550 nm band, and their coloration often is restricted to deformation lamellae with boundaries that exhibit a characteristic mirror-like reflective appearance (Shigley and Fritsch, 1993; King et al., 2002).

Comparison with Treated-Color Purple Diamonds. Natural-color purple diamonds may be easily distinguished from their treated-color counterparts. The coloration in natural purple diamonds is confined to the deformation lamellae, while in treated diamonds reported on thus far it is evenly distributed or is located in growth zones (Wang et al., 2005).

In the optical spectra of natural purple diamonds, the 550 nm band is not accompanied by sharp peaks at 575 and 637 nm, as are seen in the spectra of treated diamonds. Moreover, the absorption spectra of these natural diamonds with a dominant purple color show dichroism.

CONCLUSIONS

Natural purple coloration in diamond (figure 12) is thought to arise from post-growth plastic deformation in the earth. Using several spectroscopic methods, this study of purple crystals from Siberia and their faceted counterparts revealed various nitrogen-related defects that formed in the diamond lattice during this deformation. The color appearance of purple diamonds is influenced by localization of the coloration within microtwin lamellae and by the optical dichroism of the color centers. On the basis of their spectroscopic properties and gemological characteristics, natural-color purple diamonds may be easily distinguished from their treated-color counterparts.

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