SPECTROSCOPIC EVIDENCE OF GE POL HPHT-TREATED NATURAL TYPE IIA DIAMONDS

By David Fisher and Raymond A. Spits

Results from spectroscopic analyses of GE POL high-pressure high-temperature (HPHT) annealed nominally type IIa diamonds are presented, and these spectral characteristics are compared with those of untreated diamonds of similar appearance and type. Absorption spectroscopy reveals that any yellow coloration in such HPHT-treated diamonds is due to low concentrations of single nitrogen, which have not been observed in untreated type IIa diamonds. Laser-excited photoluminescence spectroscopy reveals the presence of nitrogen-vacancy centers in most, but not all, HPHT-treated stones. When these centers are present, the ratio of the 575:637 nm luminescence intensities offers a potential means of separating HPHT-treated from untreated type IIa diamonds. The total absence of luminescence may be another important indicator of HPHT treatment.

The announcement in March 1999 that Lazare Kaplan International (LKI) subsidiary Pegasus Overseas Limited (POL) planned to market diamonds that had undergone a new "process" developed by General Electric (GE) led to widespread unease throughout the diamond industry. Of particular concern were the statements made then and subsequently that the process—to improve color and brilliance—was irreversible and undetectable. Since this announcement, a number of articles have shown the potential for detection of such material,

on the basis of both gemological and spectroscopic features (Shigley et al., 1999; Moses et al., 1999; Chalain et al., 1999 and 2000). GE has confirmed that the process involves high-temperature annealing at high pressure (HPHT), and LKI has said that brown diamonds are used as the starting material (Moses et al., 1999; figure 1 inset).

De Beers's own extensive research program, including studies begun in the 1980s, has enabled us to compare spectroscopic observations made on several GE POL–treated samples with data obtained on experimental samples that had been HPHT-treated at the De Beers Diamond Research Laboratory (DRL) in Johannesburg. These DRL samples were treated purely for research and only to help establish identification techniques. This article will highlight spectral features that we believe are potentially indicative of such HPHT-treated diamonds. Comparison with similar natural untreated samples is very important in this respect, and this has formed a significant part of our continuing research.

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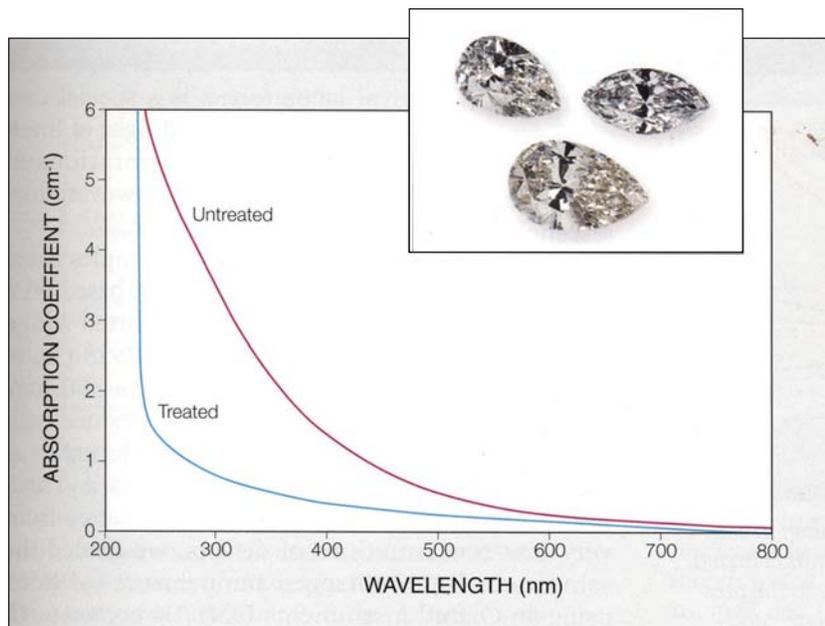*Please see acknowledgments at end of article.*

Figure 1. High-pressure, high-temperature treatment—such as that used by General Electric in their GE POL diamonds—can effectively reduce the coloration of some type IIa brown diamonds to colorless. In this UV/Vis/NIR absorption spectrum, note the increasing absorption at lower wavelengths of an untreated type IIa brown diamond. After HPHT treatment at the De Beers Diamond Research Laboratory, the same sample (now colorless) showed a significant reduction in that absorption. The inset shows three GE POL diamonds in a range of color grades: Top left = I color, 0.50 ct; top right = E color, 0.45 ct; bottom = J color, 0.77 ct. Photo by Elizabeth Schrader.

MATERIALS AND METHODS

Samples Examined. The Gemological Institute of America (GIA) has published demographic information on 858 GE POL diamonds (Moses et al., 1999). Almost all of these samples were nominally type IIa diamonds. While GIA records infrared (IR) spectra on some of the diamonds submitted to the Gem Trade Laboratory, the laboratory uses transparency to short-wave ultraviolet radiation, and the presence of a cross-hatched ("tatami") strain pattern seen with crossed polarizers, as a practical means of distinguishing type IIa diamonds during its normal diamond grading procedure (J. Shigley, pers. comm., 2000). Technically, type IIa diamonds are defined as those that have no detectable nitrogen in that part of the IR absorption spectrum between 1332 and 400 cm^{-1}, the so-called single-phonon region, where absorption due to vibration of impurity atoms is present. (A phonon is a quantum of lattice vibration within a crystal; see, e.g., Collins, 1982. For more on diamond types, see, e.g., Fritsch and Scarratt, 1992.) Most of the 858 samples were colorless or near-colorless, although some had a distinct yellow hue.

For the present study, we examined 16 commercially purchased GE POL diamonds ranging in color from D to U/V and in weight from 0.36 to 4.09 ct. All were laser-inscribed "GE POL" and accompanied by GIA Gem Trade Laboratory grading reports. Some showed distinctive internal features such as discoid fractures around solid inclusions and partial graphitization of pre-existing fractures; all showed banded or cross-hatched strain patterns when viewed between crossed polarizers with a microscope, as described by Moses et al. (1999). All these samples were nominally type IIa. By "nominally" we mean that there was no easily detectable nitrogen-related absorption between 1332 and 400 cm^{-1}. However, as is described below, careful high-resolution IR measurements did, in some cases, detect features due to single substitutional nitrogen atoms (i.e., where a nitrogen atom substitutes for a carbon atom, hereafter referred to as "single nitrogen"), which typically are not seen in untreated type IIa diamonds.

We also analyzed samples that had been subjected to what we believe are similar HPHT conditions using technology developed at the DRL. Seventeen originally brown, type IIa rough diamonds were treated. In some cases, multiple samples were cut from the same piece of rough before treatment, so that the total number of DRL-treated pieces studied was 23 (visually estimated to be 18 colorless, 5 near-colorless). Parallel windows were polished on opposite sides of each sample to permit accurate absorption spectroscopy measurements. Nine of these samples were fully characterized before HPHT treatment, and all were fully characterized after treatment.

Five additional brown (14 total) and 16 colorless type IIa rough diamonds were examined fully for comparison with the HPHT-treated samples. These samples were also windowed to permit accurate absorption measurements. One advantage of using rough diamonds taken directly from De Beers mines is that we can guarantee absolutely that our comparison samples were untreated.
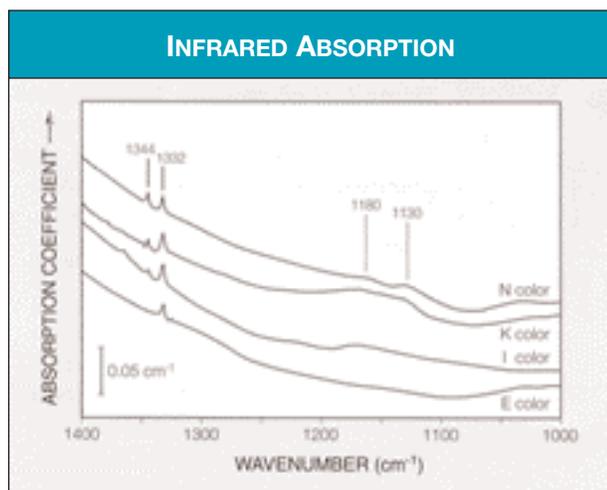
Figure 2. These IR absorption spectra were taken in the single-phonon region on four GE POL diamonds. Three (I, K, and N colored samples) show features due to the presence of single nitrogen (i.e., the peak at 1344 cm–1 and broad band at 1130 cm–1), and weak absorption at about 1180 cm–1 due to low concentrations of B-centers. The sharp peak at 1332 cm–1 is observed in most diamonds.

Absorption Spectroscopy. Room-temperature infrared spectra were recorded on all samples with a Nicolet Magna-IR 750 Fourier-transform IR spectrometer. We used a Perkin-Elmer Lambda 19 spectrophotometer for ultraviolet/visible/near-infrared (UV/Vis/NIR) absorption measurements on all samples. Faceted samples were mounted in such a way as to allow the beam to pass through the sample, usually through opposite pavilion facets. IR spectra were acquired at a resolution of 0.5 cm–1 to allow increased sensitivity to very low concentrations of lattice defects. Quantitative information from the IR spectra was obtained by normalizing the spectra to the intrinsic diamond absorption between 2664 and 1500 cm–1, the so-called two-phonon absorption. In this region, the IR spectrum, expressed as absorption coefficient, is the same for all diamonds. For spectra in the UV/Vis/NIR range, a semi-quantitative approach was adopted, whereby the sample was mounted in the same configuration as in the IR spectrometer and the same factor as determined from the intrinsic IR absorption was assumed for conversion to absorption coefficient.

Photoluminescence. The photoluminescence (PL) technique measures the intensity of light emitted from a sample, as a function of wavelength, in response to excitation by UV radiation or visible light. In general, different wavelengths of excitation will produce different PL spectra. Laser Raman microspectrometry, a standard analytical technique in many gemological laboratories, is a special case of PL spectroscopy where the emitted light of interest is produced by characteristic vibrations of molecules and crystals. In this study, however, fluorescence was the emitted light of interest.

For this study, we tested all of the samples listed above with custom-built PL equipment based on a Spex 1404 double-grating spectrometer fitted with a thermoelectrically cooled Hamamatsu R928 photomultiplier detector. A Spectra-Physics 300 mW multi-line argon ion laser was used to produce excitation at 488 and 514.5 nm (hereafter referred to as "514 nm"). To achieve the high sensitivity and high resolution necessary to reveal luminescence from very low concentrations of defects, we cooled the samples to liquid-nitrogen temperature (–196°C) using an Oxford Instruments DN1704 cryostat. All the PL spectra were normalized to the intensity of the first-order Raman peak.

RESULTS

Absorption Spectroscopy. UV/Vis/NIR spectra recorded on an initially brown diamond before and after treatment at the DRL are shown in figure 1. The absorption, which steadily rises with decreasing wavelength, is responsible for the initial brown color. This absorption decreased substantially following

TABLE 1. Single substitutional nitrogen concentrations calculated for the 16 GE POL diamonds, by color grade.

Color grade	Single nitrogen concentration (ppm)	
	Infrared[a]	Ultraviolet/visible[b]
D	—	<0.01
E	—	<0.02
E	—	0.04
E	—	<0.01
F	—	0.03
F	—	<0.01
F	—	<0.02
I	0.21	0.20
J	0.11	0.06
K	0.17	0.26
K	0.35	0.32
L	0.28	0.31
L	0.45	0.52
M	0.21	0.23
N	0.42	0.48
U/V	0.35	0.48

[a] *Calculated after Kiflawi et al. (1994) from the 1344 cm–1 peak.*
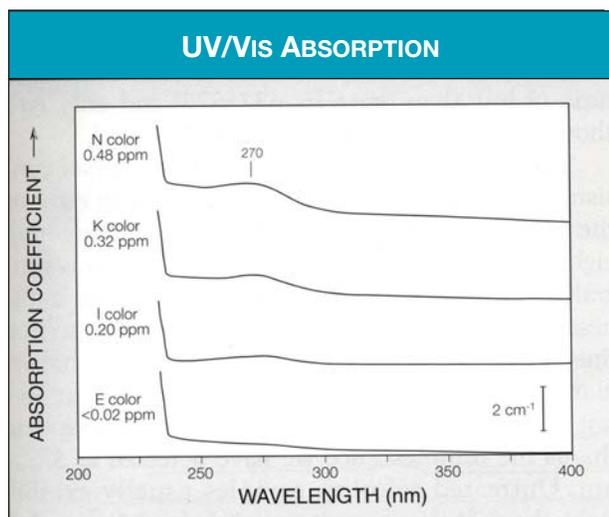[b] *Calculated after Chrenko et al. (1971) from the 270 nm peak.*

Figure 3. The broad peak at 270 nm in the UV/Vis absorption spectra of these four GE POL diamonds (same samples as in figure 2) is due to single substitutional nitrogen. Note that the strength of this peak increases as the color increases.

HPHT treatment, resulting in a colorless diamond.

IR absorption spectra from four GE POL stones (color graded E, I, K, and N) are shown in figure 2. All four spectra show a sharp line at 1332 cm^{-1}, which is often observed at very low intensity in untreated type IIa diamonds. The spectra of the I, K, and N color samples also show, albeit very weakly, a broad absorption feature peaking at 1130 cm^{-1} and a sharp line at 1344 cm^{-1}. This line would not have been detectable in all cases at a resolution lower than 0.5 cm^{-1}. The shape of the 1344 and 1130 cm^{-1} absorption is consistent with that due to single nitrogen. Kiflawi et al. (1994) determined conversion factors to relate the strength of the 1130 cm^{-1} absorption to the concentration of nitrogen, and we used these conversions to calculate the concentration figures presented in table 1. From the infrared spectra, we determined single-nitrogen concentrations ranging from about 0.5 atomic parts per million (ppm) down to about 0.1 ppm in the near-colorless GE POL samples. We also detected an additional weak, broad peak at 1180 cm^{-1} in some samples that was due to the presence of low concentrations of B-centers (aggregates of nitrogen consisting of four nitrogen atoms and a vacancy).

Single nitrogen gives a characteristic broad absorption peak at 270 nm in the UV region (Dyer et al., 1965). UV/Vis spectra for the same E, I, K, and N color samples are given in figure 3. Although spectra often are distorted due to reflections within the sample, the single nitrogen signature can be clearly detected for the I, K, and N color samples.
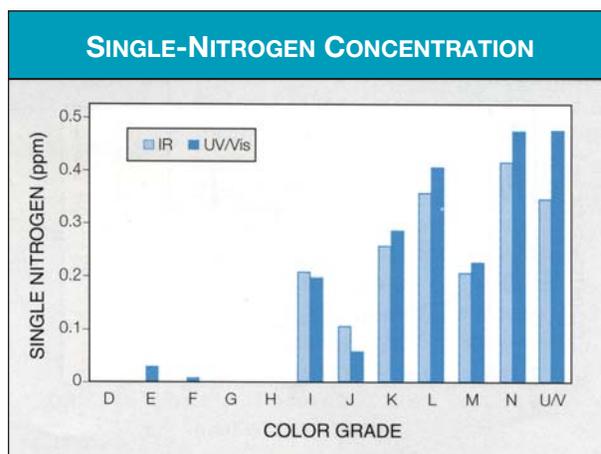
Figure 4. This bar chart illustrates the variation in single-nitrogen concentration with intensity of color (indicated here by color grade) for the GE POL diamonds tested. The nitrogen values were calculated using IR and UV/Vis absorption features (see table 1). No samples with G and H color were examined; no single nitrogen was detected in the one D color sample. Average values are shown for the E (n = 3), F (n = 3), K (n = 2), and L (n = 2) colored samples.

Only a hint of the 270 nm peak was detected in the E color diamond, for which the nitrogen concentration was estimated to be no more than 0.02 ppm.

For comparison with the IR values, we determined nitrogen concentrations from this absorption using the formula published by Chrenko et al. (1971), with modifications to allow for any additional absorption in the UV region that our own experimental work had shown to be necessary.

These values show reasonable agreement with those obtained from IR absorption (again, see table 1). As a general trend, single-nitrogen concentration increases as color becomes more evident (figure 4).

Photoluminescence. *488 nm Ar Ion Laser Excitation.* Figure 5 presents spectra acquired from the four GE POL samples (E, I, K, and N color) cited above, in this case using 488 nm excitation from the argon ion laser. The dominant feature in most cases was the H3 luminescence. This can be distinguished from other centers that give sharp-line luminescence at 503 nm by the structure of the associated vibronic band (Collins, 1982). Our annealing experiments on initially brown samples showed that there is a decrease in the H3 luminescence on HPHT annealing. In some, but not all, cases, additional features were observed. The lines at 575 and 637 nm are due to emission from the nitrogen-vacancy center in its neutral, (N-V)^0, and negative, (N-V)$^-$, charge state, respectively (Mita, 1996).
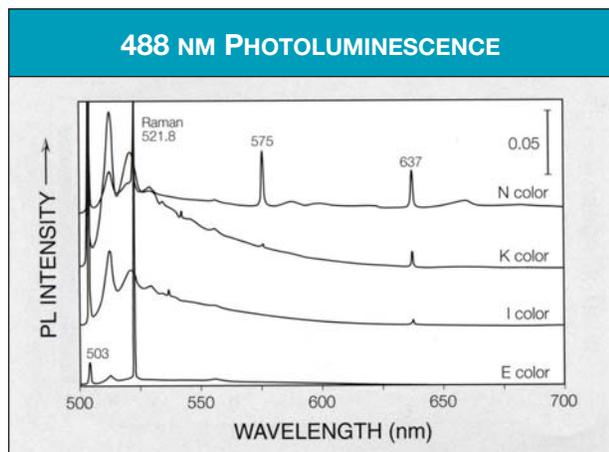
488 NM PHOTOLUMINESCENCE

Figure 5. These photoluminescence emission spectra of four GE POL diamonds (same as in figure 2) were obtained at liquid-nitrogen temperature using 488 nm Ar ion laser excitation. The dominant features are luminescence from the H3 center (503 nm) and from the nitrogen-vacancy center in its neutral (575 nm) and negative (637 nm) charge states. The line at 521.8 is the first-order Raman peak.

514 nm Ar Ion Laser Excitation. The first room-temperature PL spectra of GE POL diamonds under 514 nm excitation using a Raman spectrometer were published by Chalain et al. (1999 and 2000). Liquid-nitrogen temperature PL spectra from the E, I, K, and N color GE POL samples are shown in figure 6, and table 2 presents a summary of the main features observed in the range of samples studied under these conditions. All the spectra we obtained showed the first-order Raman line at 552.4 nm and the second-order Raman spectrum between 578 and 597 nm. In some cases, a line was observed at 535.8 nm. The defect responsible for this luminescence has not been identified, but emission at this wavelength has been observed in untreated type Ia brown diamonds and in type Ia diamonds that have been irradiated and annealed (see, e.g., Anderson, 1963; Collins, 1982).

The key GE POL features are lines at 575 and 637 nm related to the N-V center. Chalain et al. (2000) noted an increase in intensity of the 637 nm luminescence with more strongly colored samples. Although there was such a trend in most of the GE POL diamonds we examined, we did not observe a strict correlation of 637 nm emission with either color grade or single-nitrogen content.

However, we did observe that the ratio of the 575:637 nm emission intensities in HPHT-treated diamonds was, in general, lower than that typically found in untreated colorless type IIa diamonds. Of the 14 GE POL diamonds examined with detectable 575 and 637 nm luminescence, 12 had a 575:637 nm

ratio of less than one (i.e., 637>575) and only two showed a ratio greater than one (i.e., 575>637).

The luminescence at around 575 nm in fact consisted of two lines, at 574.8 and 575.8 nm, in eight of the 16 GE POL samples examined. In the remaining eight samples, the 575.8 nm luminescence was generally absent in samples where the 574.8 nm luminescence was weak or also absent. A luminescence line at 575 nm has been observed in type Ia brown diamonds that show yellow luminescence to 365 nm excitation (Jorge et al., 1983); it is possible that this is the luminescence we have detected at 575.8 nm. Untreated colorless samples usually exhibit only the 574.8 nm emission (of the 16 samples examined, 14 showed detectable 574.8 nm luminescence and none showed the 575.8 nm line). In contrast, most untreated type IIa brown diamonds exhibit either (1) both 574.8 and 575.8 nm luminescence, or (2) in more strongly colored samples, neither of these lines but instead a line at 578.8 nm. Strongly colored brown samples often reveal a line at 559.0 nm as well (see figure 7). Just two of our 14 type IIa brown samples showed only 574.8 nm luminescence. We are continuing to investigate whether this also might be a useful diagnostic characteristic.

The colorless HPHT-treated samples generally showed weaker luminescence than the near-colorless to yellow stones. For two of the seven GE POL diamonds of D to F color, and for 15 of the 18 DRL-treated samples of similar treated color, no significant luminescence was generated under these excitation and detection conditions. Conversely, none of our 16 colorless untreated type IIa diamonds or 14 type IIa brown diamonds showed the complete absence of luminescence features under such conditions. Therefore, the presence or absence of luminescence, in itself, is a useful diagnostic characteristic for a diamond that has been confirmed to be type IIa by IR spectroscopy.

DISCUSSION

Single Nitrogen. In the vast majority of natural diamonds, any hint of yellow color is due to the presence of N3 centers, and stones with strong N3 absorption are usually referred to as Cape diamonds (Collins, 1982). The N3 center consists of three nitrogen atoms surrounding a common vacancy (van Wyk, 1982). In contrast, our spectroscopic results show that those GE POL samples that appear pale yellow do so because of the presence of low concentrations of single nitrogen. Although single nitrogen has been found in natural diamond—in

514 nm Photoluminescence

Raman 552.4 · 574.8 · 637.0 · 0.05 · 535.8 · 575.8 · N color · K color · I color · E color · PL INTENSITY · WAVELENGTH (nm)
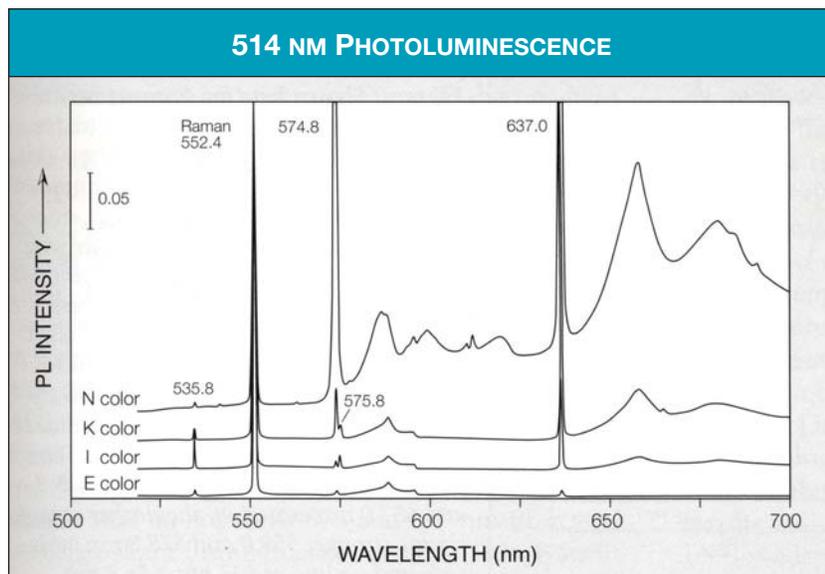
Figure 6. Photoluminescence emission spectra of the same four GE POL diamonds as in figure 5 were obtained at liquid-nitrogen temperature using 514 nm argon ion laser excitation. The 574.8 and 575.8 nm lines are clearly visible in the spectra of the I and K colored samples, and the stronger 637 nm—relative to the 575 nm—emission is evident. Also observable is a line at 535.8 nm.

so-called Canary yellows—it usually is present in samples that also contain significant concentrations of aggregated nitrogen; that is, the diamonds are of mixed type Ia/Ib. In the course of many years of research, we have never encountered a natural diamond that is nominally type IIa, but that contains sufficient single nitrogen to produce an observable yellow color. This includes not only the samples used in this research but also a suite of 50 type II or very weakly type Ia diamonds studied previously (the only non–type Ia diamonds found among 6,000 D to R color polished stones examined). As discussed below, the presence of single nitrogen in GE POL diamonds is important in the interpretation of other spectroscopic observations.

The single nitrogen in GE POL diamonds is probably the product of dissociation of aggregates of nitrogen that were initially present in small concentrations in the untreated sample. Brozel et al. (1978) have shown that it is possible to dissociate A-centers at temperatures over 1960°C and B-centers at temperatures above 2240°C. We believe that these temperatures are consistent with those used in the

GE POL treatment. (Over geological time, single nitrogen atoms in natural diamonds aggregate to form more complex defects. A-centers are pairs of nitrogen atoms; B-centers are formed of four nitrogen atoms surrounding a vacancy; see, e.g., Evans, 1991; Fritsch and Scarratt, 1992.) The presence of single nitrogen in nominally type IIa diamond provides an indication of HPHT treatment.

Nitrogen-Vacancy Centers. With 514 nm Ar ion laser excitation, most of the GE POL samples tested showed luminescence at 575 and 637 nm due to the neutral and negative charge states of the nitrogen-vacancy center. N-V centers will be produced by the combination of a proportion of single nitrogen with vacancies. The most probable source of these vacancies is dislocations. The starting specimens for the GE POL process, brown type IIa diamonds, have undergone plastic deformation as part of their geological history. The brown coloration is thought to be associated with the dislocations produced by the deformation (e.g., Wilks and Wilks, 1991). The reduction in color on annealing is presumably due to a modification of the structure of the defect that is responsible for the absorption states. It has been observed that GE POL diamonds exhibit a high degree of strain and that the pattern of strain birefringence is similar to that seen in brown type IIa diamonds (Moses et al., 1999).

Recently there have been reports of HPHT color alteration of type Ia brown diamonds (Moses and Reinitz, 1999; Collins et al., 2000). Here the color change is to yellowish green due to the production of H3 absorption and fluorescence. It is thought that during HPHT annealing, vacancies are generated at

TABLE 2. Summary of 575/637 nm luminescence in untreated and HPHT-treated type IIa samples.[a]

Category	Total no.	Only 575 nm	Only 637 nm	Both 575/637	Neither 575/637	575<637
GE POL	16	0	0	14	2	12
DRL-treated	23	0	3	0	20	3
Colorless	16	7	0	7	2	1
Brown	14	2	4	4	4	4

[a] *Luminescence features generated by 514 nm Ar ion laser excitation.*

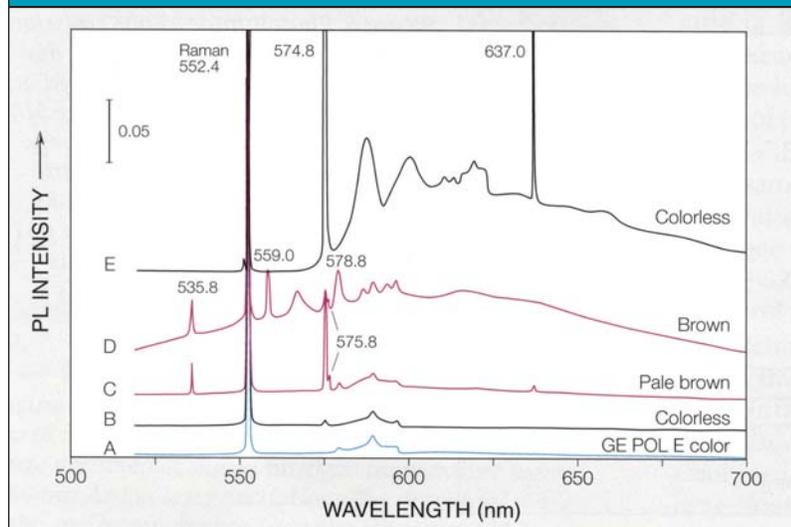**514 NM PHOTOLUMINESCENCE COMPARISON SPECTRA**

Figure 7. Shown here for comparison are photoluminescence emission spectra (at 514 nm excitation) for a range of type IIa diamonds. The E-color GE POL sample (A) shows no sharp luminescence lines, only Raman features. Spectra B and E are from two untreated type IIa colorless diamonds: B shows only the 574.8 nm line, whereas E shows strong emission at 574.8 and 637.0 nm from the neutral and negative nitrogen-vacancy center, respectively. Spectra C and D are from a pale brown and a darker brown type IIa sample, respectively. The pale sample shows stronger 574.8, 575.8, and 637.0 nm emission; the darker sample shows stronger 559.0 and 578.8 nm emission and no line at 637 nm; 535.8 nm emission is seen in both.

the dislocations, and these vacancies combine with pre-existing A-centers in the vicinity of the dislocations to produce the N-V-N centers. This is evidenced by the distribution of the resulting H3 luminescence in banded or cross-hatched patterns. We believe that a similar process has taken place in these type IIa diamonds to produce the observed N-V centers. In addition, both the 575 and 637 nm luminescence peaks, seen in some type IIa brown diamonds, may be preserved during annealing.

The ratio of the 575:637 nm emission intensities in GE POL diamonds varies considerably, but in most of the samples we tested the ratio was less than one (i.e., 637>575). This indicates that a significant proportion of the N-V centers are in their negative charge state. Because substitutional nitrogen in the diamond lattice acts as a deep donor, and a large proportion of these GE POL diamonds show detectable quantities of single nitrogen, one would expect negatively charged N-V centers to be present in them. However, we did not find a strict correlation between color grade (or single nitrogen concentration) and the 575:637 nm emission ratio; this may be due to the presence of other donor and/or acceptor centers that will affect the relative numbers of N-V centers in respective charge states.

Chalain et al. (2000) found that 637 nm photoluminescence was present in all five GE POL, and in three untreated brown type IIa, diamonds studied, but not in the seven D-color untreated type IIa diamonds that they tested. They therefore tentatively suggested that the presence of 637 nm PL in non-brown type IIa diamonds might be diagnostic of HPHT treatment. However, they make the point that this is a preliminary result based on the small number of samples,

and that further investigation is needed. They have now examined 14 GE POL samples in the D to L color range, all of which showed 637 nm luminescence (J.-P. Chalain, pers. comm., 2000).

In this work, we found two GE POL diamonds (D and F color) that did not show 637 nm PL. An analysis of GE POL diamonds that fall exclusively in this color range would probably yield a significant number of such samples, although detection of 637 nm PL will undoubtedly depend on the sensitivity of the instrument being used. The diamonds HPHT-treated at the DRL were carefully selected initially to ensure very low nitrogen content, and a high proportion of the resulting samples (20 out of 23) exhibited no 637 nm luminescence. We also found seven *untreated* colorless type IIa diamonds that did show 637 nm PL (see, e.g., figure 7E). So, although the presence of 637 nm PL may be used as an indication that further investigation is warranted, it cannot be regarded as a diagnostic characteristic on its own.

However, the ratio of the 575 to 637 nm luminescence, where present, might provide a more secure means of discriminating between untreated and treated samples. Note that the ratio we have reported here is that generated with 514 nm Ar ion laser excitation; a different ratio would be obtained under different excitation conditions. The detection response of the system will also influence the measured ratio, but a system giving a reasonably linear response over the short wavelength range between 575 and 637 nm should produce consistent results. Care must be taken to ensure that this was the case if the technique were to be transferred between laboratories.

Although there is certainly some overlap

between treated and untreated type IIa diamonds in the 575:637 nm luminescence ratio observed, in general the lower the 575:637 nm ratio the more likely it is that a particular diamond has been HPHT-annealed. This gives a more secure identification than observation of 637 nm luminescence alone. In the rare cases of untreated colorless diamonds that exhibit relatively strong 637 nm luminescence, additional spectral features should provide evidence that the diamond has not been treated. Investigation into the usefulness of such features forms a significant part of our continuing research in this area. In general, those HPHT-treated diamonds that do not show 575 and 637 nm PL reveal no other sharp luminescence features. This in itself is an important indication that a type IIa diamond might be HPHT-treated.

CONCLUSIONS

The spectroscopic data presented highlight some of the differences we have observed between natural type IIa diamonds and GE POL–treated diamonds. In particular, the presence of detectable quantities of single nitrogen and the relatively high levels of 637 nm luminescence relative to 575 nm luminescence, as observed with 514 nm Ar ion laser excitation, appear to be characteristic of some heat-treated samples. The total absence of luminescence with strong 514 nm excitation may

be another important indicator that a near-colorless type IIa diamond has been HPHT-treated.

At the time of the early POL announcements in spring 1999, it was thought in the trade that this HPHT treatment was undetectable. We believe that by using a combination of absorption features and the low-temperature, high-resolution PL results presented here, in conjunction with the gemological data presented by Moses et al. (1999), the vast majority of HPHT-treated diamonds can now be detected. De Beers is in close contact with the leading gemological laboratories. We hope that, through this collaboration, a practical application for these results will be devised shortly. This should be possible by adapting laser Raman microspectrometers, now available in many gemological laboratories, for use at low temperature and with appropriate lasers.

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