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Optical characterization of natural Argyle diamonds

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Abstract

Optical centers in natural diamonds from the Argyle mine have been characterized by absorption and photoluminescence techniques. Many of those diamonds show characteristic PL spectra with a large number of slowly decaying centers, most of which can be attributed to the Ni impurity. A linear correlation was observed between the intensity of the 3107 cm^{-1} hydrogen-related peak and the concentration of IR-active nitrogen. It may be accounted for by the presence of NH_3, NH_4 and N_2 gases as sources of nitrogen during diamond growth. It is shown that the unique blue-gray color of some Argyle diamonds is determined not only by the high hydrogen content, but also a small concentration of tri-nitrogen complexes in combination with a large concentration of two- and four-nitrogen defects.

Keywords: Natural diamonds; Nickel; Luminescence; Absorption

1. Introduction

The Argyle mine in western Australia is the largest source of natural diamonds. It is also famous for its rare pink, green and blue-gray diamonds. The likely causes of coloration are based on the fact that the majority of Argyle diamonds are heavily plastically deformed. In most diamonds deformation produces a brown color, but under some, as yet unresolved circumstances it can generate a pink hue [1]. A plausible reason for the green color in Argyle stones is natural irradiation by alpha particles [2]. It produces GR1,2..8 absorption systems associated with the neutral vacancy (V^0), which absorb red, yellow and blue light producing the green coloration [3]. Finally, the blue-gray color appears to be associated with the strong hydrogen-related peaks at 3107 and 3236 cm^{-1} in IR absorption spectra [4,5]. Blue color in diamond is commonly associated with absorption from boron acceptors. However, no boron could be detected in Argyle diamonds and therefore, their blue-gray color is commonly ascribed to hydrogen-related defects.

The 3107 cm^{-1} IR absorption peak with accompanying weaker lines at 1405, 2786, 4169 and 4469 cm^{-1} was characterized in natural diamonds from other mines and ascribed to the vibrations of the $C{=}CH_2$ group [6,7]. Interestingly, it can be produced in diamonds grown by high-pressure high-temperature (HPHT) technique after their subsequent annealing at temperatures above 1900 °C. Such annealing also leads to the aggregation of nitrogen from the single substitutional atoms (N_S or C center) to the N_S–N_S pairs (A center) and further to the nitrogen-vacancy complexes $3N_S$–V (N3 and N2 centers) and $4N_S$–V (B center). In some annealed HPHT crystals the intensity of the 3107 cm^{-1} peak can be linearly proportional to the concentration of nitrogen (present mostly in the A form), but in a significant group of samples the 3107 cm^{-1} peak could not be produced by annealing [8].

The blue-gray Argyle stones show characteristic optical absorption spectra [4,5]. Electron spin resonance (ESR) applied to those samples detected the Ni-related NE2 center [5]. The NE2 center is commonly observed in HPHT diamonds grown with a Ni-containing catalyst if they are either grown or annealed after growth at temperatures above 1400 °C. The center is attributed to

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a $2N_S$–Ni complex [9]. Numerous data on Ni-related centers in diamond, obtained by optical absorption [9], photoluminescence (PL) [10] and ESR [9] techniques, are documented in the literature. Most of the existing results are summarized in a review by Zaitsev [3].

In the present paper defect centers in Argyle diamonds have been characterized by optical absorption and PL. It is found that a common feature of Argyle diamonds is the presence of optically active hydrogen and nickel atoms. Therefore, an additional comparative study of Ni-related PL centers has been performed on natural Argyle and synthetic diamonds.

2. Experimental

Twenty-four diamonds were gem-quality blue-gray Argyle crystals with no visible extended defects or inclusions. Approximately 60 samples were rough diamonds from the same mine. All of them had irregular morphology and were rich in various extended defects. Approximately 40 were gray-green, others were colorless or light gray. Several tens of natural Ia diamonds, originating mostly from Yakutia, Guinea and Venezuela, were used as examples of natural Ia diamonds, which contain IR-active hydrogen, but no optically active Ni. Nickel-related centers were identified in a suite of several tens of HPHT crystals grown with a Ni-containing catalyst. The sample used for curve 4 in Fig. 6 was annealed in the HPHT setup for a few hours. Exact details of annealing are unknown. Absorption spectra revealed that nitrogen in this sample is present in the A form at concentrations of 160 ppm with undetectable N3, N2, C and B centers. Therefore, the annealing temperature was inferred to be in the range 1900–2400 °C. This temperature is also consistent with the presence of some and absence of other characteristic Ni-related PL lines with known annealing behavior [10].

IR absorption spectra were recorded at 300 K using a commercial Fourier-transform spectrometer. Visible absorption and PL were measured in the temperature range 77–300 K. The following excitation sources were used in PL experiments: the 514.5-nm line of a CW Ar^+ laser, a tunable CW Ti-sapphire laser tuned to 740 nm and an N_2 laser coupled to a dye module. The latter system provides 3-ns pulses at 337.1 nm (pump) and in the range 430–700 nm. PL signals were detected using a lock-in amplifier at a fixed frequency in the range 4–1130 Hz.

3. Results and their interpretation

3.1. IR absorption

Fig. 1 presents selected IR spectra from Argyle diamonds. Nitrogen in those samples is mostly present in the B form and its concentration increases from samples 1 to 5. The spectra of Fig. 1 show the diamond two-phonon absorption spectrum in the range 1600–2650 cm^{-1} and sharp peaks at 3236, 3107, 2960, 2920, 2850, 2786, 1540, 1500, 1460, 1434, 1405, 1365, 1333, 1260, 1170, 1095, 1020, 1016 and 800 cm^{-1}. The following interpretation of the observed peaks can be given based on previously reported results: several peak combinations are well known from the spectra of organic molecules. They include those at 2960, 2920, 2850 and 1460 cm^{-1} originating from the stretching and bending modes of CH_3 and CH_2 groups [11]. The hydrogen-related lines at 3107, 2786 and 1405 cm^{-1} were discussed in Section 1. The 3236 cm^{-1} line can be attributed to the $C{\equiv}CH$ group [11]. The peaks at 1333, 1170 and 1020 cm^{-1} form a characteristic spectrum of the B nitrogen, which is always accompanied by a peak at ~1365 cm^{-1} due to extended planar defects termed 'platelets' [3].

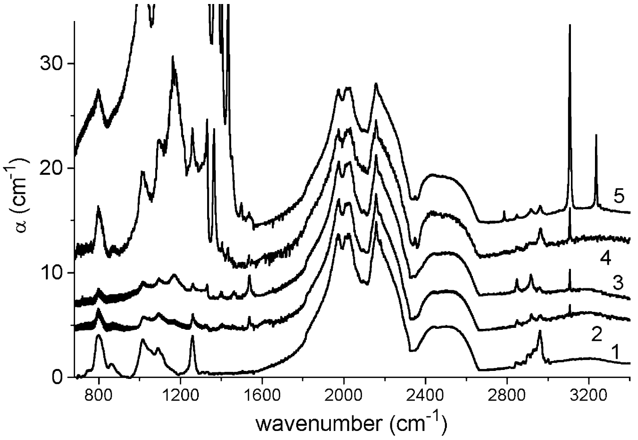

Fig. 1. Room-temperature IR absorption spectra from IaB Argyle diamonds. Nitrogen content increases from sample 1 to 5.

The four peaks at 1260, 1095, 1016 and 800 cm^{-1} compose another well-defined spectrum. It is often seen in irradiated and annealed diamonds with low nitrogen content and its detection in the Argyle stones may be attributed to natural irradiation [12]. The apparent correlation of the intensity of this spectrum and of the hydrogen-related lines in the range 2850–2960 cm^{-1} is probably coincidental because neutron irradiation of IIa diamonds produces the former spectrum without the latter one [12].

Another tendency seen in Fig. 1 is an increase in the intensity of the 3107 cm^{-1} hydrogen-related peak with the nitrogen content. Nitrogen content is determined from the intensity of the corresponding IR spectra [3]. In order to reveal a possible correlation the integral under the 3107 cm^{-1} peak was plotted vs. the concentration of optically active nitrogen for a large group of Argyle samples (see Fig. 2). Despite significant scatter a clear trend towards increasing 3107 cm^{-1} peak intensity with the nitrogen concentration can be found in Fig.

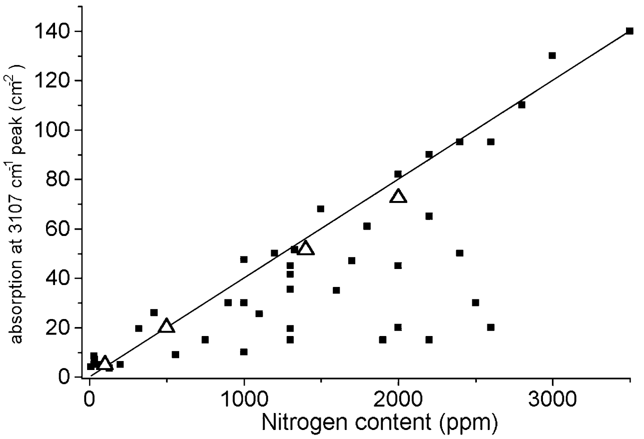

Fig. 2. The integrated absorption of the $3107\ \mathrm{cm}^{-1}$ hydrogen-related peak vs. the concentration of optically active nitrogen in the studied Argyle diamonds. Open triangles and solid squares correspond to samples with dominant A and B nitrogen centers, respectively. The straight line points to a possible correlation.

2. Since many diamonds, especially when not originating from Argyle mine, have a high nitrogen concentration, but undetectable $3107\ \mathrm{cm}^{-1}$ peak, and given that sample regions with low hydrogen content could also be present in hydrogen-rich samples, the appearance of many data points below the straight line in Fig. 2 can be understood. The solid squares in Fig. 2 are plotted for diamonds with dominant B nitrogen defects while open triangles present results for IaA diamonds. Interestingly, they both show the same tendency, indicating that the correlation is not sensitive to the nitrogen form.

3.2. Visible absorption

Fig. 3 presents absorption spectra from Argyle diamonds in the range 370–1230 nm. Curves 1–3 correspond to blue-gray samples. The green color of the sample used for curves 4 and 5 is determined by strong absorption from the N2 and N3 systems in the blue and by several centers in the red region of the spectrum. Narrow lines at 1184, 1004.5, 998, 991, 979, 974, 967, 944, 935, 550, 477 and 415 nm are accompanied by broader bands at 840, 730 and 540 nm. Comparison of spectra obtained from different samples with published results [4,5] reveals that none of those feature are mutually related, except for two: The 540-nm band probably belongs to the 550-nm zero-phonon line (ZPL). This system is particularly strong in Argyle pink diamonds [5]. The 550-nm line is rather broad both at room temperature (curve 2) and no sharpening is seen when cooling it down to 77 K (curve 3). This behavior may be attributed to local strain around the 550-nm centers and it agrees with the assignment of the pink color to plastic deformations [1].

Our measurements on different samples confirm the reported observation [4,5] that the optical centers, responsible for the absorption in the red and yellow parts of the spectrum, are strong only in samples with intense hydrogen-related IR peaks at 3107 and 3236 cm^{-1}.

The inset in Fig. 3 reveals the presence of the vacancy-related GR1 system with a ZPL at 741 nm. This system was seen in most of the studied green-gray Argyle diamonds. Annealing at 800 °C for 2 h removes the GR1 system as expected (curve 5 in Fig. 3), thus reducing the intensity, but not eliminating the green color.

3.3. Photoluminescence in Argyle and HPHT diamonds

Many Argyle diamonds show PL from the irradiation-related GR1 center. Confocal micro-PL measurements reveal that GR1 PL is concentrated at the surface of the crystal, suggesting that they were naturally irradiated by alpha particles [2].

Selected parameters of the PL centers, observed in the natural Argyle and HPHT diamonds, are summarized in Tables 1 and 2. Fig. 4 presents PL spectra from an Argyle and an HPHT diamond sample measured at 77 K under 740-nm excitation. Spectra are normalized to the diamond Raman peak. The Ni-related PL features, in particular, the 793.6-nm ZPL with associated vibronic structure and the broad band at 900 nm, are seen in both samples. ZPLs at 786.6 and 928 nm are present in the natural, but not HPHT sample.

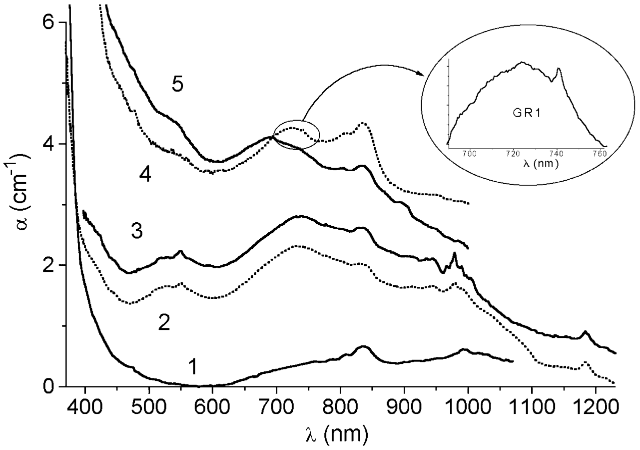

Fig. 3. Optical absorption spectra from gray (curve 1), blue-gray (curves 2 and 3) and green-gray (curves 4 and 5) Argyle diamonds measured at 300 K (curves 1and 2) and 77 K (curves 3–5). Curves 4 and 5 present spectra from the same sample before (4) and after (5) annealing at 800 °C for 2 h. The inset shows the magnified view of the GR1 (V^0) system from curve 4.

PL spectra under 337.1-nm excitation from Argyle diamonds are shown by curves 1–3 in Fig. 5. During the course of this comparative study an undocumented PL center was observed in HPHT diamond at 547.2 nm (see curve 4 of Fig. 5 and Table 1). The center could

Table 1
Properties of some Ni-related PL centers in HPHT diamond observed in the present work (Pw) or in the literature [3,10]

λ (nm)	E (eV)	E_{ph} (meV)	τ (μs)	Thermochromic	Reference
477.2	2.6	40	240	–	NE3 center;
523.2	2.369				
477.7	2.596	42, 72	3.5	–	S2 center;
488.8	2.537				[3,10], Pw
484.0?	2.560		10	–	Pw
496.8	2.496	40, 70	180	–	S3 center; [3,10], Pw
547.2	2.266	64, 127	150	–	Pw
573.5	2.162			+	Pw
597.6	2.073	55	<10	+	[10], Pw
601.6	2.054	44, 100	70	–	Pw
688.0	1.802		10 000	–	Pw
725.5	1.709			+	Pw
727.7	1.704	34, 59	3000	+	[10], Pw
747	1.660	59	3000	+	[10], Pw
793.6	1.563	35, 70	0.027	–	[3,10]
881.0	1.408		<1	–	[3,10]

E_{ph} is phonon mode energies; τ is PL lifetime at 77 K. The accuracies for the E_{ph} and τ values reported in this work are 5 and 50%, respectively.

Table 2
Properties of PL centers, which are not observed in HPHT diamond, but are common for natural Argyle diamonds

λ (nm)	E (eV)	E_{ph} (meV)	τ (μs)	Thermochromic
574.5	2.158		200	
603.5	2.055	33, 90	200	+
641.0	1.934		200	–
645.0	1.922		200	–
659.0	1.882		<0.1	–
669.0	1.854		<0.1	–
688.5	1.801		100	
694.5	1.785		200	
700.3	1.771	40, 60	100	+
786.6	1.576		<1	–
928.0	1.336	34	<1	–

E_{ph} is phonon mode energy. τ is PL lifetime at 77 K. The accuracies for the E_{ph} and τ values are 5 and 50%, respectively.

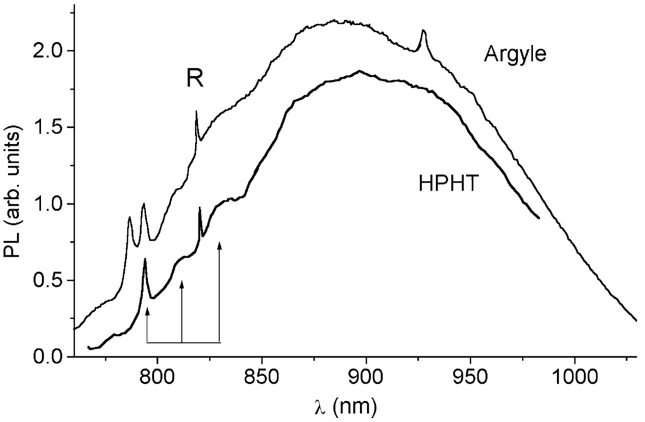

Fig. 4. PL spectra from Argyle and HPHT diamonds measured at 77 K under 740-nm excitation. R marks the diamond Raman peak. The trident points to the 793.6-nm vibronic system.

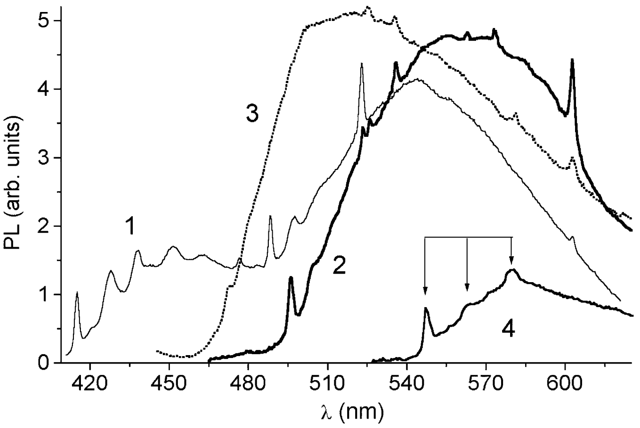

Fig. 5. PL spectra measured under 337.1-nm excitation from a blue-gray Argyle diamond (curve 1) at 77 K, from a green-gray Argyle diamond at 77 K (2) and 180 K (3) and from an HPHT diamond (4) at 77 K.

only be seen in crystals grown with Ni-containing catalyst at elevated temperatures (>1400 °C). The N3 system with ZPL at 415 nm is observed in PL and absorption from most natural diamonds (see curve 1). The lines at 525 and 536 nm (curve 2) are common for natural IaB diamonds [3], while those at 563, 574 and 603 nm (curves 2 and 3) are specific for Argyle diamonds. Peaks at 472, 477, 489, 496 and 523 nm are assigned to the Ni-related S2, S3 and NE3 centers [3,9,10]. The Ni-related broad PL band at ~540 nm is responsible for strong green PL from Argyle and HPHT samples under UV excitation. The specificity of transition metal-related PL centers is that the corresponding defect levels originate from the atomic d-shells, optical transition between them being forbidden. This deter-

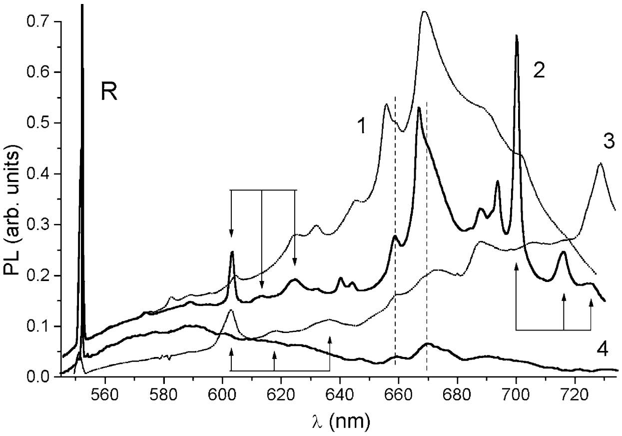

Fig. 6. PL spectra measured at 300 K (curves 1, 3 and 4) and 77 K (2) under 514.5-nm excitation from a green-gray Argyle diamond (curves 1 and 2), from a yellow HPHT diamond grown at 1450 °C (3) and from a colorless HPHT sample annealed at $T>1900$ °C for a few hours. R marks the diamond Raman peak. The tridents point to the identified vibronic systems.

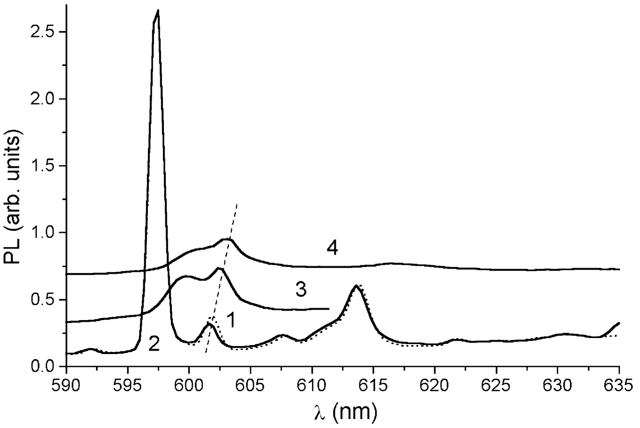

Fig. 8. PL spectra from a HPHT diamond measured at 77 K (curves 1 and 2), 200 K (3) and 300 K (4) under 514.5-nm excitation modulated at frequencies of 1.1 kHz (2) and 10 Hz (1, 3, 4).

mines the long PL lifetime of PL from the Ni-related centers. None of the absorption centers in Argyle diamonds (Fig. 3), except for the N3 system (see Fig. 5), could be detected in PL and vice versa, none of the PL centers were present in absorption. It is unlikely that all of those absorption centers are not PL-active. More probable is that the PL in Argyle diamonds is dominated by small amounts of centers with long PL decay, which may originate from Ni, but are not seen in absorption.

There is a remarkable change in the PL spectrum shape between 77 K (curve 2) and 180 K (curve 3). It manifests itself in the shift of the green band peak position and a strong decrease in the amplitude of the 496-nm and 603-nm peaks. The decrease in ZPL intensity is normally accompanied by a strengthening of the vibronic sideband. However, the latter is not observed for the 603-nm center, indicating a thermal quenching behavior, namely decrease of the integral PL with increasing temperature.

Following the above observations, a wider search was undertaken for thermal quenching and slow-decaying PL centers in natural and HPHT samples. The results are presented in Tables 1 and 2 and in Figs. 6–10, which present PL spectra under 514.5 nm excitation. Curves 1 and 2 in Fig. 6 show PL spectra from a blue-gray Argyle diamond measured at 300 and 77 K, respectively. The spectra were normalized to the diamond Raman peak in order to allow the amplitude comparison. Similar to Fig. 5, a thermal quenching behavior is observed in Fig. 6: the systems with ZPLs at 603.5 and 700.3 nm strongly increase in intensity when the sample is cooled from 300 to 77 K.

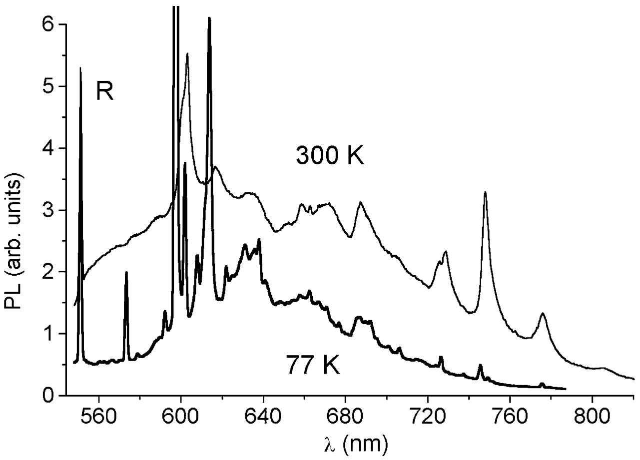

Fig. 7. PL spectra from a HPHT diamond measured at 77 and 300 K under 514.5-nm excitation modulated at 10 Hz. R marks the diamond Raman peak.

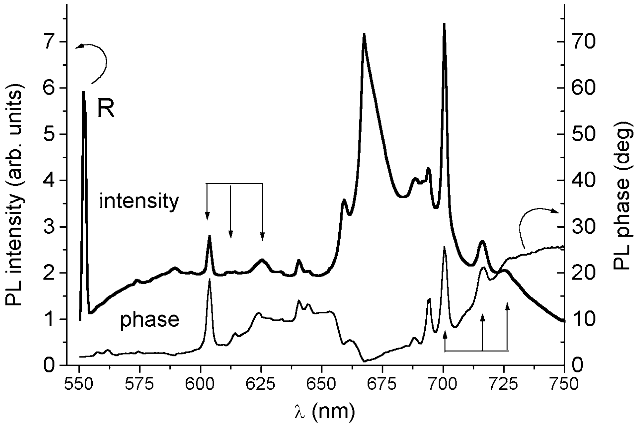

Fig. 9. The intensity and phase of PL signals from a blue-gray Argyle diamond. PL was excited at 77 K by 514.5-nm light, modulated at 1.1 kHz. R marks the diamond Raman peak. The tridents point to the identified vibronic system.

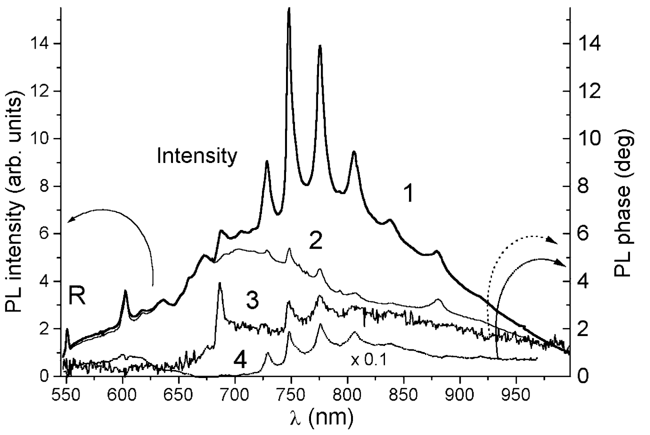

Fig. 10. The intensities (curves 1 and 2) and phases (curves 3 and 4) of PL signals from an HPHT diamond. PL was excited at 300 K by 514.5-nm light, modulated at 10 Hz (curves 1 and 3) and 550 Hz (curves 2 and 4). R marks the diamond Raman peak.

The spectra of Fig. 6 are rich in structure. Such behavior is typical for irradiated or Ni-doped diamonds. The sample used for Fig. 6 showed no irradiation-related absorption lines and thus the first hypothesis is unlikely. In order to evaluate the second one, room-temperature PL spectra from an as-grown and an annealed Ni-doped HPHT diamond were plotted in the same figure (curves 3 and 4). Nitrogen in the first HPHT sample was present mostly in the C form, while it was aggregated to the A form in the annealed crystal, thus making a closer approximation to natural conditions. Similar to the natural diamond, a ZPL at 601.6 nm is seen in the as-grown HPHT sample. However, its different vibronic modes and disappearance with annealing suggest that it belongs to a different center. Meanwhile, the 659- and 669-nm peaks are present in all samples and may all be attributed to Ni-related defects.

Thermal quenching behavior was also observed for PL from HPHT crystals. Figs. 7 and 8 present PL spectra from a HPHT sample measured at 300, 200 and 77 K and normalized to the diamond Raman peak. They reveal that the intensity of the 573.5- and 597.6-nm peaks decreases, while that of the ZPLs at 725.5, 727.7 and 747 and associated vibronic peaks increase when the sample is heated from 77 to 300 K.

The lifetime of a PL center is commonly measured by direct registration of the PL transient under pulsed excitation. However, when this method is applied to the spectra of Fig. 6, the separation of contributions from overlapping signals is not straightforward. Therefore, PL lifetimes were estimated using a method based on the registration of PL intensity and phase under modulated excitation. The absolute lifetime values in the second method can be obtained from the formulas, based on the solution of a kinetic equation for a periodically excited damped oscillator. Instead, the second method was calibrated by the direct registration of PL transient from the same PL center. The results are shown in Tables 1 and 2 and in Figs. 8–10. Curves 1 and 2 in Fig. 8 present PL intensity spectra recorded at frequency $f=10$ Hz and 1.1 kHz, respectively. While no changes are observed for the 597.6-nm center, the amplitude of the 601.6-nm peak decreases with frequency, indicating a slower PL relaxation for the latter.

Fig. 9 presents spectra of PL intensity and phase for a blue-gray Argyle diamond, measured at $T=77$ K and $f=1.1$ kHz. The observable (positive) phase shift for most of the peaks reveals that their lifetime is comparable to the $1/f=0.909$ ms value. Meanwhile, the faster decaying 659- and 669-nm PL peaks are seen as dips in the phase spectrum due to the presence of a slow background PL band. Analogous amplitude (curves 1 and 2) and phase (curves 3 and 4) spectra, measured at $f=10$ Hz (curves 1 and 3) and 550 Hz (curves 2 and 4) from a HPHT sample, are shown in Fig. 10. They reveal several slow PL centers, whose parameters are presented in Table 1.

4. Discussion

The following explanation can be proposed for the observed correlation between the intensity of the 3107 cm^{-1} hydrogen-related line and the nitrogen content in the sample. The most obvious source of nitrogen for natural diamonds is N_2, NH_3 and NH_4 gases, which should decompose at HPHT conditions of diamond synthesis. The correlation between nitrogen and hydrogen content could possibly point to diamonds grown in an NH_3 and NH_4-rich medium, while nitrogen-rich, but hydrogen-poor diamonds could have been grown in the N_2-rich environment. This explanation can also account for the impossibility to produce the 3107 cm^{-1} peak in many annealed HPHT crystals [8].

Our observations, along with previously reported results [4,5], reveal that the absorption centers that are responsible for the blue color of some Argyle diamonds, can also be present in green and yellow diamond; the necessary condition for their presence is a high hydrogen content, rather than the Argyle origin. As discussed above, a large hydrogen content is only observed in nitrogen-rich stones. Along with the A and B nitrogen, they also show absorption from the N2 and N3 systems. Therefore, the uniqueness of the Argyle blue diamonds lies not only in a high hydrogen content, but also in a rare combination of high concentrations of the 2-nitrogen (A center) and 4-nitrogen (B) defects and of low concentrations of the 3-nitrogen (N3 and N2) complexes.

The following model may be suggested to account for the thermal quenching behavior of PL lines in Argyle and HPHT samples: Let us consider a three-level system with the ground state *G* and two excited states *E*1 and

$E2$, $E2$ being within a few phonon energies above $E1$. Transition $E1 \rightarrow G$ is allowed, it produces fast-decaying and relatively inefficient PL, while the transition $E2 \rightarrow G$ is forbidden, it is responsible for slow and bright PL. In this system a temperature drop results in a decrease in population of the $E2$ level and correspondingly in PL quenching, such as observed for the 720–820-nm lines in Fig. 7. The model does not explain the observed quenching of PL intensity with increasing temperature for other lines, because exchanging the $E2$ and $E1$ levels will produce just a minor PL decrease, but not its disappearance. However, the quenching of PL with increasing temperature can be explained by photothermal ionization of a corresponding defect. If its excited state lies close to the conduction band then a temperature rise will increase the rate of the ionization of the defect via the excited state to the conduction band, thus quenching the PL.

PL spectra of Figs. 4–6 reveal the presence of Ni-related lines due to the S2, S3 NE3, 659-, 669- and 793.8-nm centers in Argyle diamonds. Peaks at 574.5, 603.5, 641.0, 645.0, 688.5, 694.5 and 700.3 nm may also be tentatively attributed to Ni-related defects based on their slow relaxation. The 603.8-, 700-, and 788-nm centers have already been attributed to Ni-related centers based on the correlation of their intensity with that of the Ni-related S2, S3 and 793.6-nm centers [13].

5. Summary and conclusions

Optical absorption and photoluminescence techniques were measured for natural diamonds from the Argyle mine. The results confirm that many Argyle diamonds were naturally irradiated at temperatures below 600 °C [2]. Those diamonds show characteristic PL spectra with a large number of slow-decaying centers, most of which can be attributed to the Ni impurity. Many of those centers show thermal quenching behavior, which is also common for synthetic diamond and which agrees with the presence of forbidden Ni-related optical transitions. IR spectra reveal that some Argyle diamonds have exceptionally strong hydrogen-related peaks and a high content of aggregated nitrogen. A positive correlation was observed between the integrated intensity of the 3107 cm^{-1} hydrogen-related peak and the nitrogen concentration. It may be explained by a model considering NH_3, NH_4 and N_2 gases as nitrogen supplies during the diamond growth. It is shown that the unique blue-gray color of some Argyle diamonds is determined not only by the high hydrogen content, but also by a low concentration of the tri-nitrogen complexes in combination with a high concentration of the two- and four-nitrogen defects.

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