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Citation: Chin. Phys. B, 2024, 33 (**6**): 068102. DOI: 10.1088/1674-1056/ad2a6d
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Synthesis and nitrogen content regulation of diamond in a high-pressure hydrogen-rich environment

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(Received 25 November 2023; revised manuscript received 4 February 2024; accepted manuscript online 19 February 2024)

The regulating nitrogen content of diamond in a hydrogen-rich high-temperature and high-pressure (HPHT) growth environment was systematically investigated in this work by developing three growth systems, namely, "FeNi+Ti", "FeNi+$C_3N_6H_6$", and "FeNi+Ti+$C_3N_6H_6$". Optical microscopy, infrared spectroscopy, and photoluminescence (PL) spectroscopy measurements were conducted to analyze the spectroscopic characteristics of diamonds grown in these three systems. From our analysis, it was demonstrated that the presence of hydrogen in the sp^3 hybrid C–H does not directly affect the color of the diamond and facilitates the increase of the nitrogen-vacancy (NV) center concentration in a high-nitrogen-content diamond. In addition, titanium plays an important role in nitrogen removal, while its impact on hydrogen doping within the diamond lattice is insignificant. Most importantly, by regulating the ratio of nitrogen impurities that coexist in the nitrogen and hydrogen HPHT environment, the production of hydrogenous IIa-type diamond, hydrogenous Ib-type diamond, and hydrogenous high-nitrogen-type diamonds was achieved with a nitrogen content of less than 1 ppm to 1600 ppm.

Keywords: diamond, HPHT, superhard material, hydrogen-rich environment

PACS: 81.05.ug, 81.05.uj, 07.35.+k, 81.10.–h **DOI:** 10.1088/1674-1056/ad2a6d

1. Introduction

Diamond, as a material with extreme functional properties, is widely used for various industrial applications, including abrasives and cutting tools, while large-sized diamond single crystals have an even broader range of applications.[1–7] Particularly, they are used in aerospace infrared windows, high-end wiredrawing dies, precision machining tools, high-end camera filters, and other fields.[8–12] At present, the fabrication of high-end diamond products has attracted the attention of the scientific community, and diamond-based materials are regarded as strategic materials for any country. The two mainstream methods for synthesizing large-sized diamonds internationally are HPHT and chemical vapor deposition (CVD), both of which also have their advantages. Among them, the high-temperature and high-pressure (HPHT) method can effectively regulate the types of doping elements and make it easier to achieve high-concentration doping. Meanwhile, the CVD method has unique advantages in achieving high-purity and larger-sized crystal growth. In recent years, typical new progress has also been made in the research of high-pressure synthesis of diamond, such as the preparation and hardness mechanism of nanotwinned diamond,[13] as well as the self-healing behavior of cracks in nanotwinned diamond composite materials.[14] More specifically, the direct phase-transition mechanism from graphite to diamond under static high pressure,[15] and the transformation and migration process of diamond grain boundary structures have been examined in the literature.[16] Moreover, the high-pressure synthesis of all sp^3 amorphous carbon bulk materials[17] and the high-pressure synthesis of paracrystalline diamond have also been explored.[18] These important results clearly demonstrate the importance of high-pressure synthesis methods in diamond research. In summary, although the basic growth of large-sized diamond single crystals has become relatively mature, further research is required, in terms of element doping functions, especially in the synergistic regulation of element doping. Therefore, it is of great significance to examine the development of element-doped diamonds and expand the performance of diamonds.

It is well known that the type and content of impurities within diamonds directly affect their optical, thermal, chemical, electrical, and mechanical properties, which in turn determine the specific application direction of diamonds.[19–22] For example, ordinary synthetic Ib diamonds have a nitrogen content of about 200 ppm and a light yellow color, and can be widely used in abrasives, grinding tools, cutting tools, and other fields.[23–25] The addition of nitrogen getters to the synthesis chamber can yield the production of type-IIa diamond with a nitrogen content of less than 1 ppm. This type is colorless and usually contains NV centers inside, which can be applied to quantum information and can also be widely used in jewelry and other fields.[26–32] With the incorporation of nitrogen-rich agents, high-nitrogen diamonds with a

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nitrogen content of about 3300 ppm can be synthesized. The large amount of nitrogen impurities inside the crystals will directly affect the properties of diamonds, which can be applied in various special industries.[33,34] When additives rich in hydrogen and nitrogen elements are added to the cavity, hydrogen-doped diamonds with a high nitrogen content can be synthesized. By further performing an annealing process, "natural-like" diamonds can be prepared, which is of great importance in terms of studying the formation mechanism of natural diamonds.[35,36] Therefore, diamonds doped with different elements have different application prospects. It is thus apparent that investigating the synthesis of multiple types of doped functional diamonds will inevitably have important scientific significance for expanding the application of diamond materials in various fields, such as technology, industry, and national defense.

Hydrogen is another widely existing impurity in natural diamonds.[37,38] According to the literature, hydrogen doping has a direct impact on the electrical properties of diamonds. However, its controllable doping in diamonds has not yet been demonstrated.[39,40] A relatively large number of experiments have shown that high-temperature and high-pressure methods are effective for the preparation of heavily doped diamonds. Nevertheless, the difficulty in sealing gas impurities in HPHT cavities also restricts the exploration of single hydrogen element doped diamonds. Therefore, the effective control of a specific element in a high-pressure cavity with a multi-element coexistence environment is considered of great significance for the production of functional doped diamonds with different properties. The synthesis of hydrogenous diamonds is not only conducive to clarifying the modulation laws of hydrogen impurities and their doping amounts on the diamond band structure, electronic structure, Fermi level, etc. It can also provide valuable insights into the understanding of the formation mechanism of natural diamonds, exploring the existence forms of hydrogen elements in the Earth's interior, and revealing the mystery of the Earth's water sphere.

Along these lines, in this work, the idea of synthesizing hydrogenous diamonds with different nitrogen content by adjusting the nitrogen content in the nitrogen–hydrogen coexistence environment under high pressure was proposed. The synthesis of controllable nitrogen-content diamonds under a hydrogen-rich environment in this work paves the way for the future industrial production of diamonds with different doping elements and contents, as well as different properties. Our work provides valuable references for the future synthesis and application of multiple types of hydrogen-containing large-sized diamond single crystals.

2. Experimental design idea and process

The experiments on diamond crystal growth via the temperature gradient method (TGM) were carried out using a China-type large-volume cubic high-pressure apparatus (CHPA) (SPD-6×1200). The synthesis and assembly processes are schematically illustrated in Fig. 1. The main experimental idea relies on the assumption that with the addition of different additives in the synthesis chamber, the growth of different types of diamonds will be achieved: FeNi system → diamond growth with nitrogen content around 200 ppm; FeNi+Ti system → growth of IIa-type diamond with nitrogen content < 1 ppm; FeNi+$C_3N_6H_6$ system → growth of hydrogenous diamonds with different nitrogen contents. Therefore, by combining the above schemes, the problem of synthesizing hydrogenous IIa diamond can be cleverly solved: namely, the traditional FeNi+$C_3N_6H_6$+Ti system → hydrogenous IIa diamond, as shown in the A1–A4 schematic diagram in Fig. 1.

In each group of experiments, a high-quality seed crystal {111} crystal surface was used as the initial-growth crystal surface; a high-purity flake graphite powder (99.99%) was used as the carbon source; $C_3N_6H_6$ was used as the hydrogen and nitrogen source; and the nitrogen removal agent Ti was

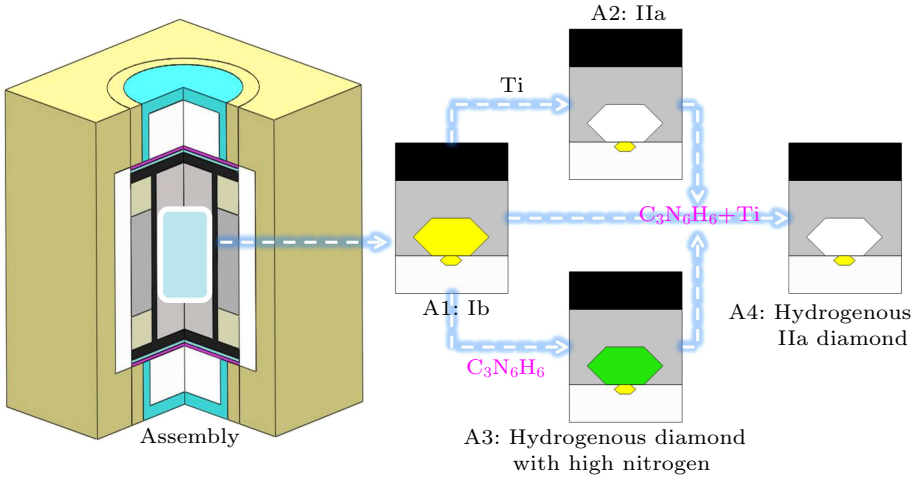


Fig. 1. A schematic diagram of the synthetic assembly process: A1–A4 experimental design ideas.

added in different proportions to the synthesis chamber containing the FeNi alloy catalyst. The pyrophyllite blocks used in the experiment were baked in a 240 °C oven for 20 h. After assembly, the experimental block was placed in a 120 °C oven and dried for 2 h before conducting a synthesis experiment to eliminate the influence of the adsorbed moisture in the air on the experimental results.

3. Experimental results and discussion

3.1. Experimental results of synthetic diamonds

The {111} crystal surface of the seed crystal was used as the initial growth crystal surface, and synthesis experiments were conducted by adjusting the different ratios of additives in the synthesis chamber under pressure and temperature conditions of 5.4–6.2 GPa and 1300–1450 °C, respectively. The experimental results are summarized in Table 1.

Table 1. The experimental results of adding different amounts of $C_3N_6H_6$ and Ti to the system.

Run	Additive (wt.%)		Pressure (GPa)	Temperature (°C)	Color
	$C_3N_6H_6$	Ti			
D-1	–	1	5.4	1300	colorless
D-2	–	–	5.5	1320	yellow
D-3	0.06	–	6.2	1400	green
D-4	0.09	–	6.2	1450	deep green
D-5	0.09	2	6.2	1440	cyan-yellow
D-6	0.09	3	6.2	1430	colorless

Based on the results presented in Table 1, yellow Ib diamond (D-2) was synthesized at 5.5 GPa and 1320 °C without additives. Colorless IIa diamond (D-1) was produced at 5.4 GPa and 1300 °C with the addition of 1.0 wt.% Ti as the nitrogen getter. Green and deep-green hydrogenous diamonds with high nitrogen contents (D-3 and D-4) were derived at 6.2 GPa and 1400–1450 °C with the addition of 0.06 wt.% and 0.09 wt.% $C_3N_6H_6$, respectively. Cyan-yellow hydrogenous Ib-type diamond (D-5) was synthesized at 6.2 GPa and 1440 °C with the addition of 0.09 wt.% $C_3N_6H_6$ and 2 wt.% Ti. Finally, colorless hydrogenous IIa-type diamond (D-6) was obtained at 6.2 GPa and 1440 °C with the addition of 0.09 wt.% $C_3N_6H_6$ and 3 wt.% Ti.

3.2. Optical characterization analysis of synthetic diamonds

Figure 2 displays the optical photographs of the crystals obtained from the experiment in Table 1.

As can be observed, in the absence of additives, yellow Ib diamonds were synthesized, as depicted in Fig. 2(b). However, when nitrogen getters (Ti) were added to the synthesis system, the synthesized diamonds became colorless, forming type-IIa diamond, as shown in Fig. 2(a). Upon the addition of $C_3N_6H_6$ to the synthesis system, dark green and green hydrogenous diamonds were produced with high nitrogen content, as displayed in Figs. 2(c) and 2(d). Moreover, by adding different ratios of $C_3N_6H_6$ and Ti to the synthesis system, hydrogenous diamonds with variable nitrogen content were synthesized. For instance, a low nitrogen content hydrogenous Ib diamond was derived with a cyan-yellow color when the proportion of $C_3N_6H_6$ was less with respect to Ti, as depicted in Fig. 2(e). Similarly, a colorless hydrogenous IIa diamond was synthesized when the nitrogen content was below 1 ppm, as seen in Fig. 2(f).

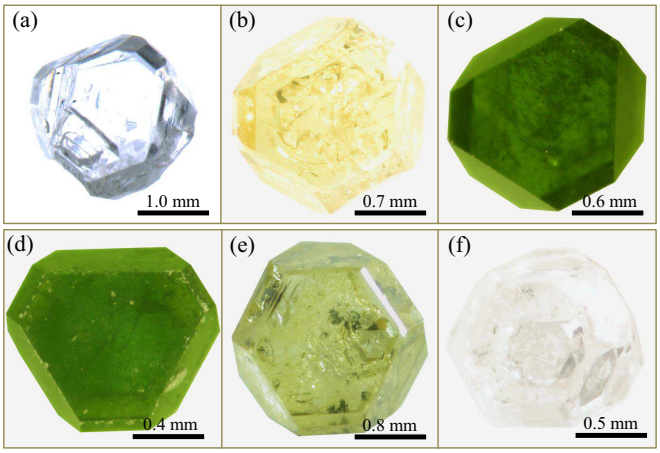


Fig. 2. Optical photographs of the diamonds obtained under different growth systems: (a) FeNi+1 wt.% Ti; (b) FeNi (no additive); (c) FeNi+0.06 wt.% $C_3N_6H_6$; (d) FeNi+0.09 wt.% $C_3N_6H_6$; (e) FeNi+2 wt.% Ti+0.09 wt.% $C_3N_6H_6$; (f) FeNi+3 wt.% Ti+0.09 wt.% $C_3N_6H_6$.

Based on the optical characterization results, a colorless and transparent diamond was obtained in the absence of nitrogen. As the nitrogen content increased, the crystal changed color, appearing yellow and then green. It is also worth noting that the presence of hydrogen impurities inside the diamond does not affect its coloration, as demonstrated by the experimental results after the nitrogen removal.

3.3. Infrared spectral analysis of synthetic diamonds

Hydrogen and nitrogen impurities in diamond crystals can be effectively detected by performing infrared spectroscopy measurements. Figure 3 shows the infrared spectrum of D1–D4 diamonds. The FeNi synthesized diamond (Fig. 3(b)) not only displays intrinsic diamond peaks of 1970 cm^{-1}, 2030 cm^{-1}, and 2160 cm^{-1}, but also exhibits a single alternate form of a nitrogen infrared absorption peak at 1130 cm^{-1}, which corresponds to the C-center nitrogen — a typical Ib diamond. The addition of Ti to the FeNi system induced the removal of the nitrogen-related absorption peak at 1130 cm^{-1}, leaving only the intrinsic diamond peaks at 1970 cm^{-1}, 2030 cm^{-1}, and 2160 cm^{-1}, as shown in Fig. 3(a) — a typical IIa diamond. The infrared spectra of diamonds grown with different amounts of $C_3N_6H_6$ additions in Figs. 3(c) and 3(d) show the C-center nitrogen absorption peak at 1130 cm^{-1} and the A-center nitrogen absorption peak at 1282 cm^{-1}. Significantly, the C-center nitrogen absorption

peak is more intense, indicating a high concentration of nitrogen within the crystal. At the same time, symmetrical and anti-symmetrical stretching vibration absorption peaks corresponding to the sp^3 hybrid C–H hydrogen bond at 2850 cm^{-1} and 2920 cm^{-1} were also observed in these two infrared spectra (illustrated in the upper-right corner of Fig. 3). These peaks indicate the formation of hydrogenous diamonds with a high concentration of nitrogen.

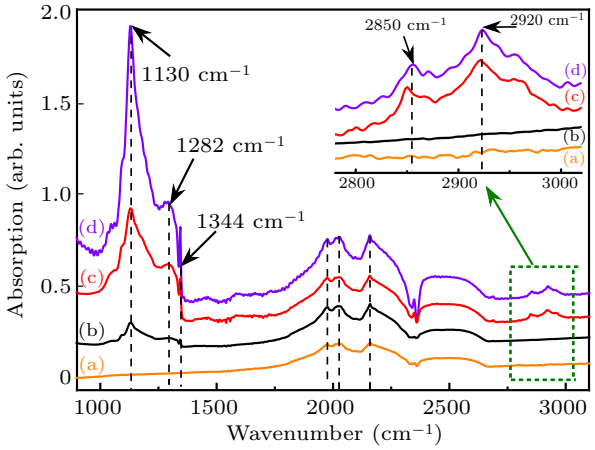


Fig. 3. Infrared spectra of diamond: (a) FeNi+1 wt.% Ti; (b) FeNi (no additive); (c) FeNi+0.06 wt.% $C_3N_6H_6$; (d) FeNi+0.09 wt.% $C_3N_6H_6$.

To provide a more detailed analysis of the internal impurities of hydrogenous Ib (D-5) and hydrogenous IIa (D-6) diamonds synthesized after the addition of titanium, multi-point measurements were taken at different positions of the two crystals, as shown in Figs. 4 and 5.

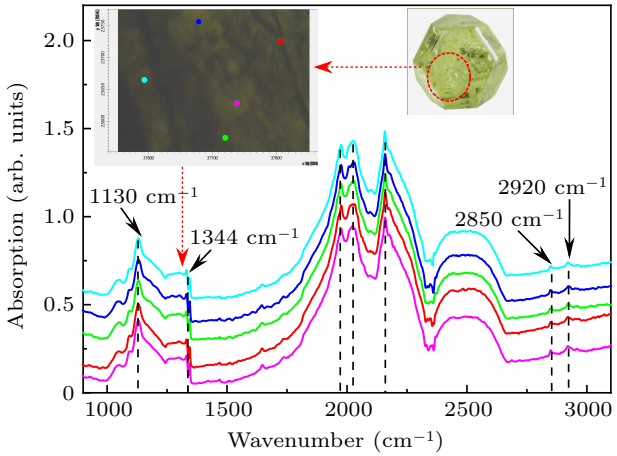


Fig. 4. The infrared spectrum of hydrogenous Ib-type diamond: FeNi+2 wt.% Ti+0.09 wt.% $C_3N_6H_6$.

Figure 4 displays the infrared spectra of diamond synthesized with 2 wt.% Ti and 0.09 wt.% $C_3N_6H_6$. The multi-point measurements from the different crystal positions demonstrate the presence of C-center nitrogen absorption peaks in the infrared spectrum. This effect signifies that the Ti content within the cavity is insufficient to eliminate the nitrogen element decomposed from $C_3N_6H_6$. Measured at various positions, there are absorption peaks related to hydrogen in the spectra, indicating that the hydrogen element decomposed as a result of $C_3N_6H_6$ doping into the diamond. Therefore, it can be argued that hydrogenous Ib-type diamond was synthesized in the FeNi+Ti+$C_3N_6H_6$ system.

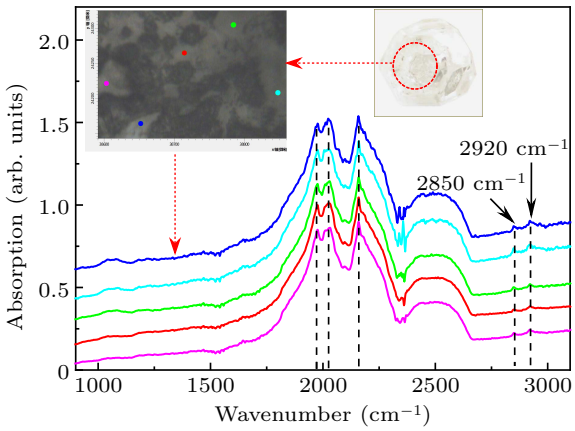


Fig. 5. The infrared spectrum of hydrogenous IIa-type diamond: FeNi+3 wt.% Ti+0.09 wt.% $C_3N_6H_6$.

Based on the above-mentioned results, under the conditions of constant addition of 0.09 wt.% of $C_3N_6H_6$, the Ti content was increased to 3 wt.% to continue synthesizing diamonds. Similarly, multi-point measurements were performed at different positions on the crystal, as shown in Fig. 5. As can be seen in the spectrum, the nitrogen-related absorption peaks' C-center and A-center disappeared, whereas only the diamond-specific peaks and the corresponding hydrogen-related absorption peaks (2850 cm^{-1} and 2920 cm^{-1}) are visible. This result indicates that there is no nitrogen or a very small amount of nitrogen (< 1 ppm) doping entering the diamond. However, there is a considerable amount of hydrogen entering the diamond. This result proves the addition of 3 wt.% Ti can remove nitrogen. Moreover, the addition of Ti did not affect the presence of hydrogen, and hydrogen can still continue to participate in both the diamond's nucleation and growth, thus achieving the growth of a hydrogenous IIa diamond. Furthermore, by combining the above-mentioned infrared spectra with the nitrogen content calculation formula, the nitrogen content in the crystals was calculated. The specific calculation formula is as follows:

$$N_C/10^{-6} = (1.1\alpha_{1130} - 0.2\alpha_{1282}) \times 25 = 25\alpha_C,$$
$$N_A/10^{-6} = (1.1\alpha_{1282} - 0.2\alpha_{1130}) \times 16.5 = 16.5\alpha_A,$$
$$\alpha_{1130} = \mu(1130\ \text{cm}^{-1})/\mu(2000\ \text{cm}^{-1}) \times 12.3,$$
$$\alpha_{1282} = \mu(1282\ \text{cm}^{-1})/\mu(2000\ \text{cm}^{-1}) \times 12.3,$$
$$\mu(1130\ \text{cm}^{-1}) = A(1130\ \text{cm}^{-1}) - A(1370\ \text{cm}^{-1}),$$
$$\mu(1282\ \text{cm}^{-1}) = A(1282\ \text{cm}^{-1)} - A(1370\ \text{cm}^{-1}),$$
$$\mu(2000\ \text{cm}^{-1}) = A(2000\ \text{cm}^{-1}) - A(1370\ \text{cm}^{-1}),$$

where N_C and N_A represent the concentrations of nitrogen in the form of C-centers and A-centers, respectively; α_C and α_A

refer to the absorption coefficients of the concentration of nitrogen in the form of C-centers and A-centers, respectively; and α_{1130} and α_{1282} denote the absorption coefficients at the 1130 cm^{-1} and 1282 cm^{-1} peaks, respectively. The uncertainty of this formula is less than 5%.[41,42] The results are summarized in Table 2.

Table 2 presents the nitrogen content calculation, demonstrating that by adjusting the Ti and $C_3N_6H_6$ ratios in the synthesis chamber, a range of hydrogenous diamonds with nitrogen concentrations from $\leq$ 1 ppm to 1600 ppm can be synthesized. Therefore, it was demonstrated that the growth of hydrogenous Ib, IIa, and high-nitrogen diamonds can be achieved.

Table 2. The nitrogen concentration of diamonds.

Run	Additive (wt.%)		Nitrogen concentration (ppm)	Hydrogen
	$C_3N_6H_6$	Ti		
D-1	–	1	≤ 1	×
D-2	–	–	~ 200	×
D-3	0.06	–	~ 800	√
D-4	0.09	–	~ 1600	√
D-5	0.09	2	~ 150	√
D-6	0.09	3	≤ 1	√

3.4. PL spectral analysis of synthetic diamonds

By performing photoluminescence analysis on these diamonds, it was found that the peak at 638 nm corresponding to the NV center was not detected in the spectrum of Fig. 6(a) of diamond IIa. Figure 6(b) depicts Ib diamond with a peak at 638 nm and an associated peak at 660 nm. In Fig. 6(c), where hydrogenous diamond with high nitrogen content is presented, both peaks at 638 nm and an associated peak at 660 nm showed an enhanced trend, while in the spectrum in Fig. 6(d) of hydrogenous Ib diamond, the peaks at 638 nm and 660 nm showed a slightly stronger trend than the Ib diamond. This effect could be ascribed to the co-doping of nitrogen and hydrogen atoms in diamond. More specifically, the simultaneous presence of the two impurity atoms could facilitate the production of vacancy defects inside the crystal.

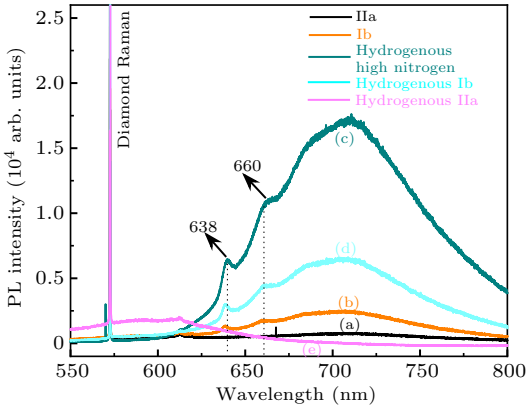


Fig. 6. PL spectra of the different types of diamonds.

Therefore, when the nitrogen content in Ib and hydrogenous Ib diamonds is similar, a slightly stronger NV center in hydrogenous Ib diamond is extracted. When a nitrogen getter is added to the synthesis environment to synthesize hydrogenous IIa diamond, it can be seen that the peaks at 638 nm and 660 nm in the curve in Fig. 6(e) did not appear. Moreover, the disappearance of the peak envelope between 620–770 nm indicates that the NV center content inside the crystal is extremely low.

4. Conclusions

In this work, the growth of hydrogenous diamonds with nitrogen concentrations ranging from $\leq$ 1 ppm to 1600 ppm was reported by adjusting the Ti/$C_3N_6H_6$ ratio in a high-temperature and high-pressure synthesis chamber. The experimental results confirmed that the hydrogen in the sp^3 hybrid C–H bond did not cause discoloration on the diamond, and Ti primarily facilitated nitrogen removal without affecting the presence of hydrogen. Additionally, from the PL results, it can be concluded that compared to the presence of a single nitrogen atom, the presence of hydrogen impurities in the synthesis environment will facilitate the increase in the NV center concentration in high-nitrogen-content diamonds. Our work paves the way toward regulating the content of a specific impurity in a multi-component co-doping system under high-pressure conditions. The proposed experimental regulation idea can be leveraged for the commercial production of industrial large-sized single-crystal diamonds with different properties in the future.

Acknowledgements

The project was supported by the National Natural Science Foundation of China (Grant Nos. 12274373 and 12004341), the Open Project of Inner Mongolia Key Laboratory of High-pressure Phase Functional Materials, Chifeng University (Grant No. cfxygy202301), the Science and Technology Project of Xilinguole Province (Grant No. 202209), and the Natural Science Foundation of Henan Province (Grant No. 242300421155).

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