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**SPECIAL TOPIC — Structures and properties of materials under high pressure**

# Low-temperature photoluminescence study of optical centers in HPHT-diamonds

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The properties and creation of optical centers in diamond are essential for applications in quantum technology. Here, we study the photoluminescence (PL) spectroscopy behavior at low temperatures of diamond subjected to electron irradiation and annealing heat treatment. Through temperature variation testing, it was found that the $NV^-$ center intensity of diamond with a nitrogen content of 150 ppm before treatment is insensitive to the experimental temperature, but significantly increases with decreasing temperature after treatment, showing sensitivity to temperature. In addition, the H3 center also shows an increasing trend with decreasing temperature. The results of annealing diamond with a nitrogen content of 730 ppm showed that even at a low temperature of 93 K, no $NV^-$ centers were detected, but there were a large number of Ni-N related centers, especially NE8 centers. Our findings can promote a deeper understanding of the behavioral characteristics of HPHT-diamond optical centers in low-temperature environments.

**Keywords:** high temperature and high pressure, diamond, optical centers, photoluminescence

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## 1. Introduction

Diamond is an extreme material with excellent physical and chemical properties. It has long been the focus of researchers.[1,2] Recently, significant research and development have focused on the optical centers in diamond. Optical centers influence the optical and electronic properties of diamonds, some of which have great potential in quantum mechanical applications.[3,4] Many optical centers are nitrogen-related, such as the NV ($NV^0$ and $NV^-$), H3 ($N_2V$), and N3 ($N_3V$) centers.[5,6] Of these optical centers, the negatively charged nitrogen-vacancy ($NV^-$) center is among the most widely studied solid-state systems for applications in quantum technologies, and it has the potential to influence significant breakthroughs in computing, simulation, communication, and sensing.[7,8] In addition, H3 centers have also attracted significant interest due to their non-blinking red and green photoluminescence, and excellent single-photon emission properties.[9,10] These properties of diamond are related to its unique lattice and optically active defect centers. Various applications require optical centers with different characteristics and their suitable concentrations or total amounts. In addition, examining the behavior of optical centers under environmental changes such as magnetic fields, temperature fields, and stress fields can contribute to their development and application in emerging fields such as high-precision detection and biotechnology.

High temperature and high pressure (HPHT) treatment is an effective method for regulating material properties.[11,12] Among them, HPHT heat treatment (annealing) has been shown to significantly alter the optical properties of diamond, thereby increasing its potential use in a variety of applications.[13] Diamond has extreme characteristics such as high hardness, high breakdown voltage, radiation resistance, and resistance to strong acids and alkalis, which make it suitable for use in extreme environments. Diamond with quantum spin characteristic optical centers is considered the best material for solid-state single-photon sources.[14] Some applications require diamond optical centers to operate at special ambient temperatures. Therefore, it is necessary to study the temperature behavior of optical centers in diamond.

In the present study, we conducted temperature-dependent photoluminescence (PL) tests on diamond before and after electron irradiation and subsequent annealing treatment, and studied in detail the behavioral characteristics of optical centers in HPHT-diamond under different conditions and their changes with test temperature. The results can provide

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deeper insight into the properties of optical center transformation in the HPHT diamond lattice.

## 2. Experimental details

The synthetic diamonds used in this study were grown from seed using the temperature gradient method (TGM) in Fe–Ni–C environments at 5.8 GPa and 1400 °C using a China-type cubic anvil high-pressure apparatus (CHPA) (SPD-6×1400). Subsequently, we annealed these diamond samples using the high-pressure apparatus at 5.5 GPa. Each diamond crystal was embedded in powdered graphite, as shown in Fig. 1.

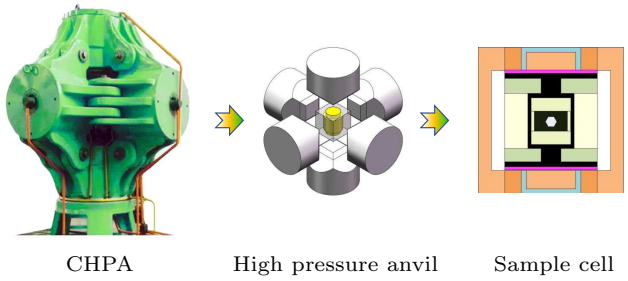



**Fig. 1.** High temperature and high pressure equipment and annealing sample cell.

After HPHT annealing, all samples were cleaned in an acid bath (sulfuric acid and nitric acid) at 300 °C before optical characterization to remove residual graphitic carbon from the diamond surface arising from the annealing processes. Finally, the samples were rinsed thoroughly with acetone, ion-exchanged water, and alcohol. The pressure was calibrated using the pressure-induced phase transitions of Bi, Tl, and Ba. The temperature was measured using a W–Re5/26 thermocouple, whose junction was placed near the sample.

The shape and color characteristics of the samples were observed using a Leica M205 C optical microscope. The infrared (IR) absorption spectra were measured using a Bruker Vertex70 V Fourier transform infrared spectrometer, with a spectral range from 400–4000 $cm^{-1}$ and a spectral resolution of 2 $cm^{-1}$ in the transmittance mode. Photoluminescence (PL) measurements were performed using a British Renishaw inVia Raman confocal spectrometer with laser excitation at wavelengths of 325 nm (He–Cd metal vapour laser), 488 nm (Ar-ion laser), and 633 nm (He–Ne ion laser).

## 3. Results

We conducted infrared tests on type Ib diamond before and after treatment, as shown in Fig. 2(a). Before treatment, the nitrogen content of type Ib diamond was mainly composed of C-center nitrogen (1130 $cm^{-1}$ and 1344 $cm^{-1}$).[15] The diamond was first irradiated with an electron beam with an energy of 3 MeV for 2 hours, followed by annealing treatment. The results showed that the infrared spectrum of diamond did not show significant changes, and the form of nitrogen was mainly composed of C-center nitrogen. The nitrogen content in the diamond before treatment was calculated to be about 150 ppm by infrared spectroscopy, and the nitrogen content after treatment was about 140 ppm. The results showed that there was no significant accumulation of nitrogen during irradiation and subsequent annealing treatment at 5.5 GPa and 1300 °C. To investigate the formation of $NV^-$ centers, we conducted PL spectroscopy tests at a low temperature of 93 K using 488 nm laser excitations on diamonds treated under different conditions, and the results are shown in Fig. 2(b). The type Ib diamond before electron irradiation and annealing exhibits strong Raman characteristic peaks. In addition, an $NV^-$ center with zero-phonon lines (ZPL) located at 637 nm was observed in the PL spectrum at a low temperature of 93 K (Fig. 2(b)(i)).[16] Figure 2(b)(ii) shows the PL spectrum of the electron irradiated crystal after annealing at pressure of 5.5 GPa and temperature of 1300 °C. It can be observed that the $NV^-$ center is significantly enhanced, indicating that electron irradiation can effectively generate a large number of vacancies, and annealing treatment causes a large number of vacancies to migrate and combine with nitrogen atoms to form $NV^-$ centers.[17,18]

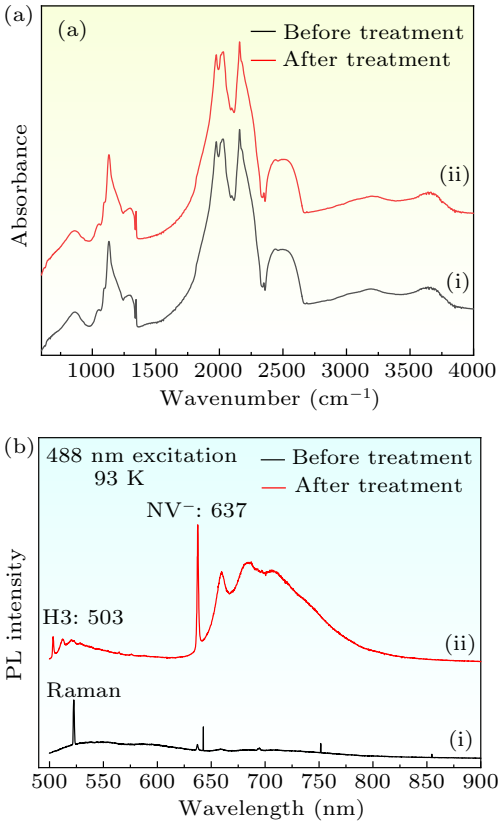



**Fig. 2.** (a) IR spectrum of diamond before and after treatment. (b) Low-temperature PL spectrum of diamond before and after treatment.

Figure 3(a) shows the PL spectra of type Ib diamond crystals before treatment at different test temperatures. It can be

seen that as the test temperature gradually decreases, the intensity of $NV^-$ centers tends to increase, but the change is slow. In addition, the peak of $NV^-$ center gradually shifts from 638.41 nm to 637.08 nm, shifting 1.33 nm towards the shorter wavelength direction (Fig. 3(b)). The results indicate that due to the presence of a small number of $NV^-$ centers in the diamond before treatment, the degree of peak intensity change caused by temperature changes is small, while the peak position shift is large.

We conducted electron irradiation on the diamond sample and annealed it at 1300 °C to increase the content of $NV^-$ centers. The processed diamond samples were subjected to temperature-dependent PL spectroscopy testing to observe the sensitivity of $NV^-$ centers to temperature again. PL spectra were collected from the temperature range of 290 K to 93 K, and the results are shown in Fig. 4(a). When the temperature gradually decreases from 290 K to 93 K, the intensity of $NV^-$ centers gradually increases, showing sensitivity to temperature.[19] The $NV^-$ center peak becomes narrower and sharper at a temperature of 93 K (Fig. 4(b)). At the same time, from Fig. 4(d), we found that the peak of $NV^-$ centers shifted to the left by 0.79 nm (from 638.27 nm to 637.48 nm). Compared to that before treatment of diamond, the change in peak value of $NV^-$ center is relatively smaller, and the peak intensity of $NV^-$ center is more sensitive to temperature. In addition, the H3 centers also exhibited similar changes, but the degree of enhancement is lower than that of $NV^-$ centers.

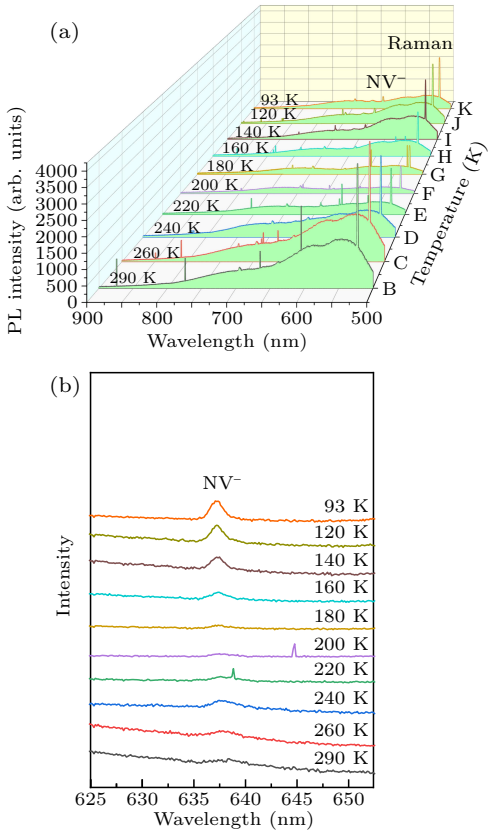



**Fig. 3.** (a) PL spectra of type Ib diamond before treatment at different measurement temperatures from 93 K to 290 K. (b) Peak shift diagram of $NV^-$ center peak.

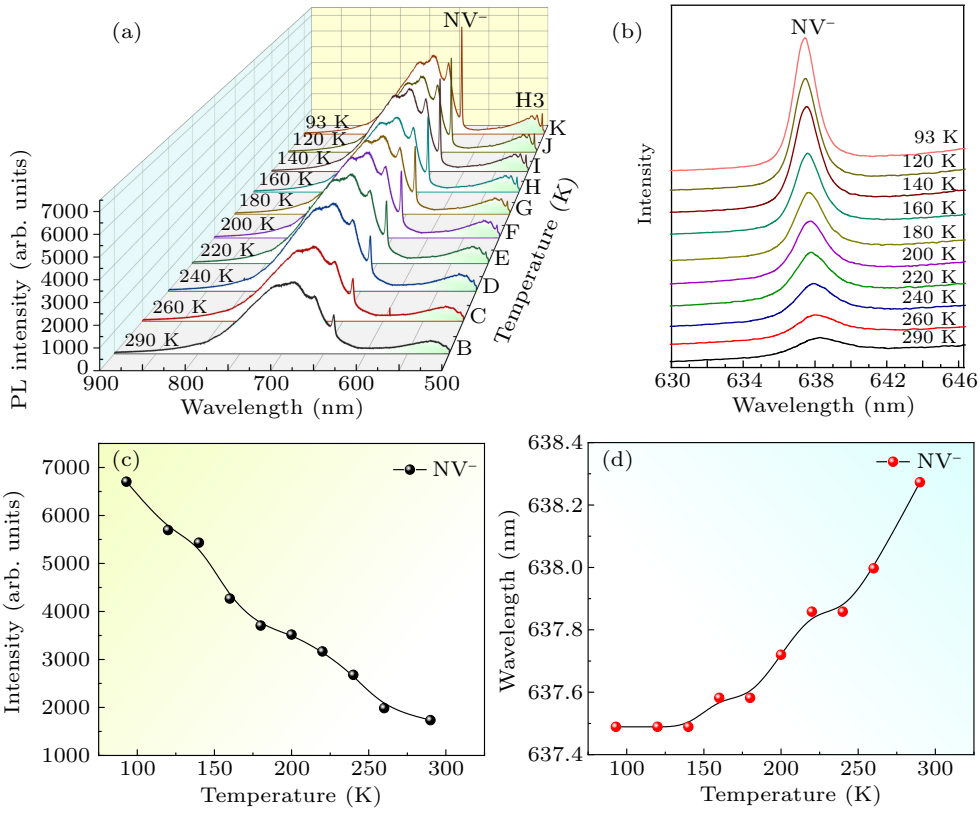



**Fig. 4.** (a) PL spectra of diamond at different measurement temperatures (from 93 K to 290 K) after 1300 °C annealing. (b) Peak shift diagram of $NV^-$ center peak. (c) Variation in NV center intensity with measurement temperature. (d) Variation of the $NV^-$ center peak position with measurement temperature.

We also annealed high nitrogen-containing diamond, which was grown in the same FeNi solvent metals at a temperature of 1380 °C and pressure of 5.6 GPa using the temperature-gradient method, with $NaN_3$ as the nitrogen source. Before treatment, the C-center dominated the spectrum. Based on the IR absorption spectrum of diamond (Fig. 5(i)), we calculated the total nitrogen content to be about 730 ppm. After HPHT annealing, the diamond crystal changed from the initial green to colorless, as shown in Fig. 5. The IR spectrum after HPHT treatment in Fig. 5(ii) reflects significant absorption, the dominant feature being two strong absorption peaks at 1282 $cm^{-1}$ and 1212 $cm^{-1}$. This absorption is due to the presence of nitrogen atoms in pairs (A-centers), and this defect was produced via the singly substituted nitrogen (C-centers) aggregates during the HPHT annealing.[20–23] After annealing, the concentration of A-centers has increased from 72 ppm to 730 ppm, whereas the concentrations of C-centers have decreased from 658 ppm to 0 ppm. According to the IR data, the treated diamond crystals are type IaA, with an aggregation of 100%.

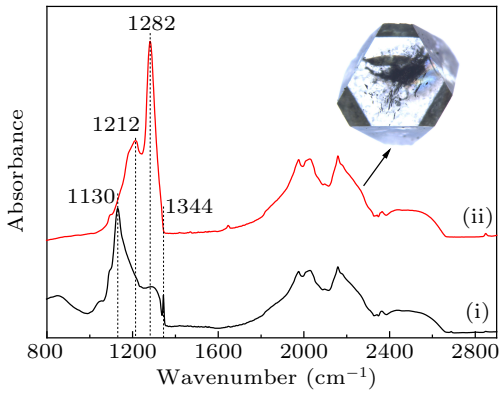



**Fig. 5.** IR absorption spectra of a synthetic N-rich diamond before (black curve) and after (red curve) HPHT treatment performed for 30 min at a pressure of 5.5 GPa and a temperature of 2000 °C. After treatment, C-centers are no longer detected by IR spectroscopy. Almost complete aggregation of C-centers into A-centers has been achieved.

Figure 6(a) shows the PL spectra of the high-nitrogen type IaA diamond samples after annealing treatment with 325 nm laser excitation at 77 K. The peak located at 496.7 nm is caused by the S3 center, which is thought to be an optical analogue of the Ni-related NE1 paramagnetic center (N–VNi–N).[24] The peak at 476.5 nm is attributed to NE2 centers, which contain one Ni ion and three nitrogen atoms (N–VNi–$N_2$).[25] The 528 nm peak could possibly be related to a defect containing a Ni atom bound to the A-center nitrogen.[26] The 483 nm and 488 nm centers that appear here have extremely weak intensity and are difficult to observe. We speculate that we speculate that this is because most nickel ions are transformed into Ni–N complexes. Therefore, after the synthetic diamond containing nickel and nitrogen is annealed by HPHT, the isolated nickel ions gradually weaken or even disappear. Annealing the type IaA diamond also produces absorption centers with ZPLs at 454 nm. The structure of 454 nm defects is still unknown, and they are detectable in the absorption spectrum of type IaB diamond after electron irradiation and annealing at 1650 °C.[27] They have also been found in the irradiated-type IaAB diamond crystals from the Mir pipe.[28]

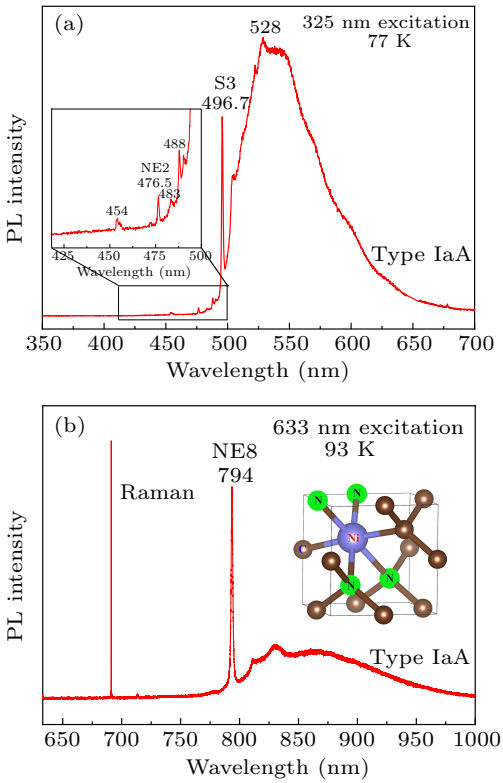



**Fig. 6.** PL spectra of high-nitrogen type IaA HPHT-treated diamonds measured at (a) 77 K with 325 nm laser excitation and (b) 93 K with 633 nm laser excitation. The inset in (a) shows partial spectral amplification at 425–500 nm.

Figure 6(b) shows the PL spectrum of type IaA diamond excited by 633 nm laser irradiation at liquid nitrogen temperature, 93 K. The diamond Raman peak is located at 691 nm. In addition, there is a strong peak at 794 nm, which corresponds to a Ni–N complex defect NE8 center.[29] Its structure consists of four nitrogen atoms surrounding a Ni ion ($N_2$–VNiV–$N_2$). For type IIa diamond, even if the annealing temperature is sufficiently high, it is difficult to form NE8 centers because of the lack of nitrogen impurities and the low nitrogen concentration due to high nitrogen aggregation. The results show that the formation of multi-atom complexes (such as NE1, NE2, and NE8 centers) strongly depends on the nitrogen concentration.

## 4. Discussion

Based on the above analysis of results, as the testing temperature decreases, the PL intensity of $NV^-$ centers in diamond usually increases. This is because the lattice vibration of diamond is weakened at low temperatures, and non-radiative recombination processes (such as phonon scattering) are reduced. More excitation energy is released through radiative

recombination (luminescence), thereby increasing the fluorescence intensity. Conversely, as the test temperature increases, atomic vibrations will also increase, and the energy of the system in an excited state rise. When the test temperature reaches a certain value, defects in the excited state will acquire sufficient energy, and the energy of the system reaches the crossing point of the ground state and the excited state. Therefore, the system can overcome the barrier and release energy in the form of phonon emission, ultimately returning to the ground state. The energy release through phonon emission leads to a decrease in the absolute intensity of PL. The absolute intensity $I$ of the ZPL at test temperature $T$ conforms to[30,31]

$$\frac{1}{I} = d_0 + e_0 \exp\left(-\frac{E_a}{kT}\right). \tag{1}$$

Here, $d_0$ and $e_0$ are constants, $k$ is the Boltzmann constant, and $E_a$ is the thermal quenching activation energy of zero phonon lines. As the temperature decreases, the spectral linewidth of the $NV^-$ centers ZPL will narrow. This is due to the weakened thermal motion of atoms at low temperatures, the weakened thermal broadening effect, and the more concentrated energy level distribution, resulting in a narrower spectral line width of ZPL. In addition, the ZPL peak of $NV^-$ centers will shift towards shorter wavelengths with decreasing temperature (blue shift).[32] The shift of the ZPL position is mainly influenced by the synergistic effects of lattice expansion and electron phonon coupling. At low temperatures, lattice vibrations weaken, electron phonon coupling weakens, resulting in an increase in energy level spacing and an increase in the energy of emitted photons, manifested as the blue shift of ZPL. The ZPL peak wavelength $\lambda$ of $NV^-$ centers can be expressed as a function of temperature[33,34]

$$\lambda(T) = \lambda_0 - \alpha T. \tag{2}$$

The PL intensity of $NV^-$ center increases when the environmental temperature decreases, with the ZPL peak shifting towards the blue and narrowing. These changes are primarily due to the reduction of non-radiative recombination at low temperatures, weakened electron-phonon coupling, and diminished thermal broadening effects. The PL intensity and peak variations of $NV^-$ center are influenced by a combination of nitrogen content, excitation wavelength, environmental factors, and measurement conditions. By optimizing these factors, the fluorescence properties of $NV^-$ center can be effectively tuned.

## 5. Conclusion

In summary, the $NV^-$ center in as-grown diamond with a nitrogen content of about 150 ppm shows no significant change in PL intensity as the test temperature decreases, demonstrating its stability with respect to the test temperature. However, after electron irradiation and subsequent annealing treatment, the intensity of $NV^-$ centers significantly increases with decreasing temperature, showing sensitivity to the test temperature. The results indicate that the content of $NV^-$ centers in diamond affects its sensitivity to environmental temperature. In addition, as the testing temperature decreases, the peak of the $NV^-$ center gradually shifts from 638.27 nm to 637.48 nm. For high-nitrogen-content (730 ppm) diamond, the low-temperature PL spectrum after annealing shows significantly different characteristics from that of low nitrogen content ($N = 150$ ppm) diamond, with no $NV^-$ centers but high-intensity NE8 color centers present. Our work provides important insights into the low-temperature properties of optical centers related to HPHT-diamond.

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