Origin, characteristics, and suppression of residual nitrogen in MPCVD diamond growth reactor

Yan Teng(滕妍), Dong-Yang Liu(刘东阳), Kun Tang(汤琨)[†], Wei-Kang Zhao(赵伟康), Zi-Ang Chen(陈子昂), Ying-Meng Huang(黄颖蒙), Jing-Jing Duan(段晶晶), Yue Bian(卞岳), Jian-Dong Ye(叶建东), Shun-Ming Zhu(朱顺明), Rong Zhang(张荣), You-Dou Zheng(郑有炓), and Shu-Lin Gu(顾书林)[‡]

School of Electronic Science and Engineering, Nanjing University, Nanjing 210046, *China*

(Received 16 December 2021; revised manuscript received 21 May 2022; accepted manuscript online 2 June 2022)

Unintentional nitrogen incorporation has been observed in a set of microwave plasma chemical vapor deposition (MPCVD)-grown samples. No abnormality has been detected on the apparatus especially the base pressure and feeding gas purity. By a comprehensive investigation including the analysis of the plasma composition, we found that a minor leakage of the system could be significantly magnified by the thermal effect, resulting in a considerable residual nitrogen in the diamond material. Moreover, the doping mechanism of leaked air is different to pure nitrogen doping. The dosage of several ppm of pure nitrogen can lead to efficient nitrogen incorporation in diamond, while at least thousands ppm of leaked air is required for detecting obvious residual nitrogen. The difference of the dosage has been ascribed to the suppression effect of oxygen that consumes nitrogen. As the unintentional impurity is basically detrimental to the controllable fabrication of diamond for electronic application, we have provided an effective way to suppress the residual nitrogen in a slightly leaked system by modifying the susceptor geometry. This study indicates that even if a normal base pressure can be reached, the nitrogen residing in the chamber can be "activated" by the thermal effect and thus be incorporated in diamond material grown by a MPCVD reactor.

Keywords: microwave plasma chemical vapor deposition, diamond, residual nitrogen, system leakage

PACS: 81.15.Gh, 81.05.ug, 07.30.Bx, 07.30.Kf **DOI:** 10.1088/1674-1056/ac7552

1. Introduction

Nitrogen is an important dopant in synthetic chemical vapor deposition (CVD) and high-pressure high-temperature (HPHT) diamond since it is both electrically (as donors) and optically (as emission centers) active.[1,2] However, due to the ubiquitous nature of nitrogen around us, unintentional incorporation of nitrogen in diamond is annoying sometimes because the incorporation is quite frequent and uncontrollable. Nitrogen could be incorporated easily by the air remnants on the chamber wall,[3] the residual nitrogen impurities in the gas precursors, or simply a system leakage,[4–8] whatever must be avoided in a controllable diamond fabrication technology.

The unintentional incorporation of nitrogen has been noticed by many previous literatures. Ashfold *et al.* have mentioned unknown level of nitrogen contamination coming from an imperfect vacuum or air impurities in the source gases, and have pointed out that using high purity source gases and a high-vacuum reactor could solve this problem.[9] However, Bolshakov *et al.* have observed obvious nitrogen vacancy (NV) luminescence when hi-purity hydrogen (9N) and methane (5N) were used as source gases. They have noted that the leakage could be a main source of the nitrogen contamination in the system, and the gas contamination due to leakage could be avoided by operating at atmospheric pressure.[10] Moreover, Tallaire *et al.* have detected nitrogen impurity of less than 1 ppm, attributed to the remnants of previous experiments.[11] More interestingly, Rabeau *et al.* have successfully fabricated the single nickel-nitrogen defects without deliberately adding nitrogen to the feeding gas. Nitrogen here is known to be present at a background level of $\sim 0.1\%$ from the gas feedstock or some residual leaks.[12] As can be seen, it is difficult to realize high-purity diamond CVD growth due to the unintentional nitrogen incorporation from complex origins. Thus, there remains a pressing need to know where the residual nitrogen comes from and how to deal with it.

Some literatures have already reported the efforts on the inhibition of the residual nitrogen by tuning the processing parameters. Achard *et al.* have reported that higher substrate temperatures would help to suppress residual nitrogen.[13] While Nistor *et al.* have studied the regulation effect of hydrogen on nitrogen doped diamonds, and found that increasing the flow rate of hydrogen could effectively reduce the residual nitrogen content in the film.[14] Besides, Matsumoto *et al.* have successfully suppressed the residual nitrogen doping by adding oxygen, realizing Schottky and p–n diodes based on diamond.[15] With these methods, residual nitrogen in the chamber can be controlled to some extent.

From the above literature survey, at least three viewpoints could be summarized as follows: (1) the unintentional nitrogen incorporation is frequently observed and the origin is somehow complicated; (2) some efforts have been made to

[†]Corresponding author. E-mail: ktang@nju.edu.cn
[‡]Corresponding author. E-mail: slgu@nju.edu.cn

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control the residual nitrogen doping by improving the equipment and/or modifying the process; and (3) the growth of diamond is highly sensitive to the residual nitrogen, and even a small amount will have a significant impact on the properties of the resulted diamond material.[16,17] All of the above points require further investigation and we happened to have a set of proper samples to study these issues, which, we think, would advance the knowledge and understanding to these urging questions.

2. Origin of the residual nitrogen

The microwave plasma chemical vapor deposition (MPCVD) apparatus in our lab is of high standard for electronic-level diamond growth. When it operates normally, unintentional nitrogen can be ignored. Nitrogen content is below the detection limit of secondary-ion mass spectroscopy (SIMS).[18] However, recently, when we have done a set of growth experiments by varying the CH_4/H_2 ratio in gas phase, the resulted samples all contain considerable amount of nitrogen. Curiously, there is no obvious leakage since the base pressure can reach the normal value of 10^{-6} Torr. Also, we did not do any change to the gas precursors as used during the normal runs. It is thus quite confusing and interesting to see the origin of the unintentionally incorporated nitrogen.

Samples were grown on (100)-oriented type Ib chemical CVD diamond substrates ($3.0 \times 3.0 \times 1.0$ mm^3). Before deposition, all substrates were cleaned in a mixed acid solution ($H_2SO_4/HNO_3 = 1:1$) for 1 h at 300 °C for metal removal. After that, they were immersed in acetone and alcohol for organic removal, and finally rinsed with deionized water and dried with nitrogen.[19,20] High-purity methane (5N) and hydrogen (5N) were used as the reaction gases, respectively, without any additional purifiers. The flow rate of hydrogen was fixed at 500 sccm, while the methane-to-hydrogen (CH_4/H_2) ratio was kept at 1%, 1.5%, 2%, 3%, and 4%. The chamber pressure and microwave power for all samples were fixed at a range of 180 Torr and 4.2 kW, respectively. The substrate temperature was determined to be 850 ± 20 °C by an IR pyrometer. The deposition time for all samples were carried out for 1 h. The diamond substrate was placed in a square groove (side length 4 mm) 5-mm away from the center of the molybdenum support.

We have noted the unintentional nitrogen incorporation by seeing typical characteristics of nitrogen in the samples. Firstly, Fig. 1(a) shows the growth rate versus the methane flow, which has been enhanced significantly as compared to the ones grown at the same condition but without unintentional nitrogen previously (hereinafter, the counterpart). The growth rate is determined by measuring the film thickness by using a micrometer with a resolution of 1 μm. Secondly, the surface morphology [the inset of Fig. 1(a) and Figs. S1(a) and S1(b)] shows obvious stepped bunches for the samples yet quite smooth surface for the counterpart. The inset of Fig. 1(a) represents the typical surface morphology of the diamond films grown at the CH_4/H_2 ratio of 2% under the condition with nitrogen or not. Thirdly, Fig. 1(b) shows the photoluminescence (PL) spectra of all the samples taken from a 100× optical lens (numerical aperture: 0.9) in a confocal system excited by a 514-nm laser at room temperature. As shown in Fig. 1(b), a sharp peak at 552 nm is derived from the first-order Raman peak of the diamond, which is used as a reference to normalize all the spectra. Diamond substrate has no nitrogen related emission peak, while all of our samples can be clearly seen that there are two nitrogen-vacancy NV^0 and NV^- PL bands with zero-phonon lines at 575 nm and 638 nm, respectively, and the broadband at 668 nm is derived from the zero order phonon trace of NV^-.[21,22] Besides, the PL spectra of the samples for the counterpart show negligible NV center-related emission in Fig. S1(c), and details can be found in the supplementary material. All the above results indicate that considerable nitrogen has been incorporated in the films and its origin is not from the substrate or from the gas precursors. Assuming that the residual nitrogen is from these two ways, the counterpart should also contain nitrogen since we use the exactly identical conditions for the samples and the counterpart. Thus, the origin could be from a chamber leak or from the air remnants on the chamber wall.

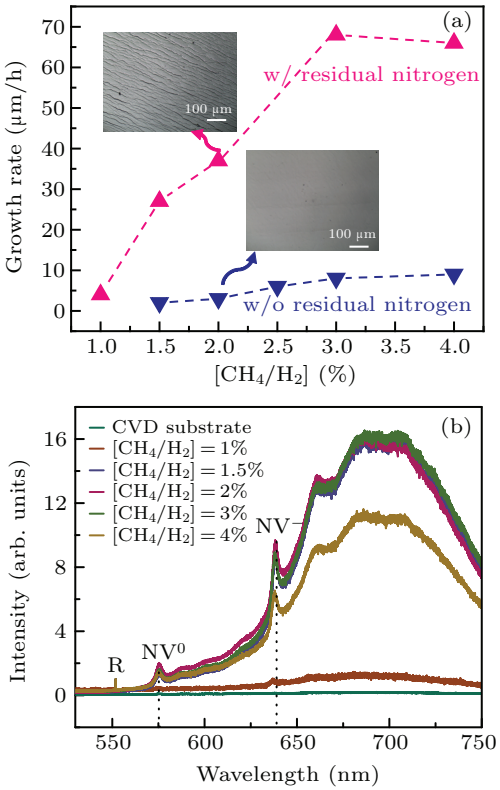


Fig. 1. Diamond films with an increase in the CH_4/H_2 gas ratio: (a) growth rate in the case with unintentional nitrogen introduction or not. Insets are the optical microscopy images for the 2% methane-to-hydrogen samples grown (800×600 μm^2); (b) PL spectra of the CVD substrate and diamond films with residual nitrogen excited by a 514 nm wavelength at room temperature.

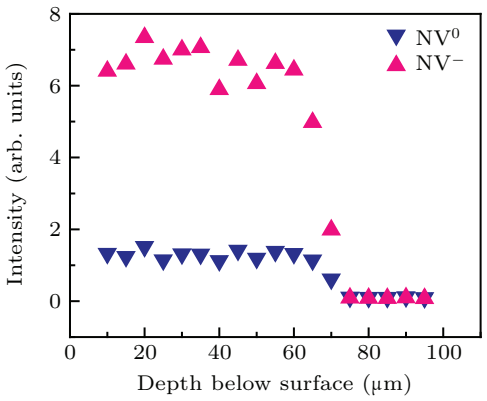


Fig. 2. NV^0 and NV^- density for the diamond with residual nitrogen at the methane-to-hydrogen ratio of 4% measured by 514 nm laser every 5 μm. The surface of this sample was particularly badly scratched and thus exhibited weak NV fluorescence.

Figure 2 shows the cross-sectional profile of the emission intensity related to the NV-centers. Detailed operational processes and description can be found in the supplementary material. The constant intensity of the NV-related emissions indicates a stable flow of nitrogen during the whole growth process. Supposing that the residual nitrogen comes from the chamber wall, the NV-centers emission should be increased with depth, since the total content of the residual gas during growth is fixed theoretically. Therefore, it is highly speculated that the residual nitrogen is from a chamber leakage rather than the air remnants on the chamber wall. But there still remains a contradiction. If the system leaks, why the base pressure can be reached. A quadrupole mass spectrometry system (INFICON, H200M) is thereby applied to detect the gas composition in the nominally evacuated system with a constant H_2 flow to simulate the environment before turning on the methane. Figure 3 shows the intensity of the detected species as a function of the microwave (MW) power from 1 kW to 4.6 kW. The detected species include nitrogen, oxygen, carbon dioxide, water, hydrogen and argon, which are quite consistent with the gas composition of air. In addition, the intensity of the residual gases increases with the MW power, indicating a higher chamber pressure when the MW power increases. Detailed analysis of gas composition can be found in the supplementary material. In our work, the substrate temperature is actually tuned by the microwave power and the pressure. Alongside increasing the plasma power, an increase in the temperature is intrinsically presented. Consequently, the above-mentioned contradiction can be understood. For the regular chamber evacuation before growth, the microwave power is off and the temperature is low to room temperature, leading to an undetectable leakage and a normal base pressure. However, when running the process, the MW power is set to 4.2 kW and the temperature is high to 850 °C. Based on the above results, the leakage at such a high temperature would be much more severe, and unintentional nitrogen incorporates in consequence. Checking the sealing ring of the quartz glass in the chamber, we have found that this is related to the thermal effect of the metal. The sealing metal is a lead ring, which has a low melting point of only 327 °C. If the seal is not protected properly, some areas may be over-heated to a high temperature, resulting in a melt and thus causing a small leak. In this way, some very minor spots of leakage, which cannot be easily found during the evacuation process, could be significantly "magnified" by the thermal effect.

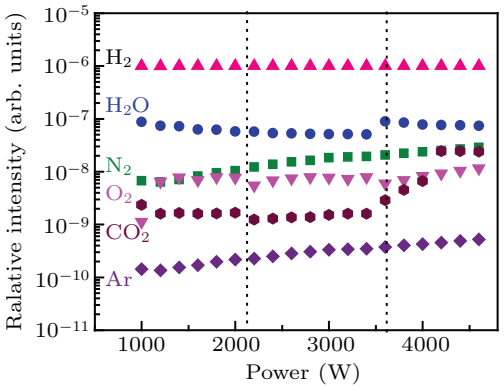


Fig. 3. The relative intensity of different active species detected by mass spectroscopy (MS) with power increasing before diamond growth. The partial pressure of every species is normalized by the total pressure in the reaction chamber.

To further confirm this leakage mechanism, when the system operates normally, we have simulated the air leakage gas environment in the chamber and have grown the diamond in that environment. A mixture of N_2/O_2 at the ratio of 4:1 have been introduced in addition to the methane and hydrogen for the diamond growth. Methane and hydrogen flow of 20 sccm and 500 sccm have been chosen here for all diamond films deposition due to its well balance of crystal quality and growth rate. Other parameters were kept no change. Two samples grown under the condition of alleged "small leakage" and "large leakage" were done by set the ratios of mixed gas to H_2 to 20 ppm and 8000 ppm, respectively. Figure 4 shows the results of the surface morphologies and the PL spectra. The sample grown with 8000 ppm mixed gas has typical surface morphology and PL spectrum of a nitrogen-doped diamond with an enhanced growth rate of 56 μm/h. Whereas the sample grown with 20 ppm mixed gas resembles the characteristic of non-nitrogen doping with a growth rate of only 4 μm/h. This is strongly different to the case for pure N_2 doping in diamond, where even as less as several ppm to 10 ppm N_2 can cause considerable incorporation of nitrogen [see Fig. S2]. This difference can be ascribed to the effect of oxygen co-existing in the leaked air, considered to have a great suppression effect on nitrogen incorporation in diamond.[23,24] Therefore, the thermal effect dependent leakage, which is caused by high gas temperature at the order of hundreds to thousands ppm, can be ascribed to the origin of obvious unintentional nitrogen in most MPCVD reactors.

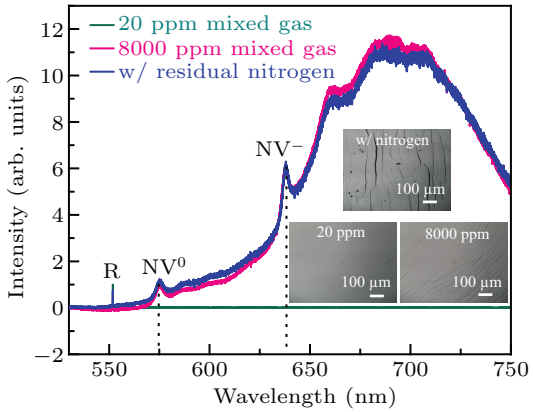


Fig. 4. PL spectra of the 20 ppm and 8000 ppm mixed gas as well as residual nitrogen doped diamond films with a 514 nm laser excitation source at a temperature of 300 K (inset are microscopy images of 4% methane-to-hydrogen diamond at 20 ppm, 8000 ppm mixed gas and residual nitrogen conditions (800×600 μm^2)).

3. Characteristics of the residual nitrogen

Due to the doping source is "leakage" and the coexistence of oxygen, the behavior of such nitrogen doping should be different to pure intentional nitrogen doping. However, this issue is not addressed in literatures. Hence, in the following part, we will make some discussion on this.

In Fig. 1, we have already seen significant enhancement of the growth rate for the samples, which has been used as a sign for nitrogen incorporation. Such growth rate enhancement has been previously ascribed to nitrogen induced enhancement of H abstraction from diamond growing surface.[25] The density functional theory calculations show that the N atoms, if substitutional positioned within the upper diamond surface, will enhance hydrogen desorption by decreasing surface H abstraction energy from the growing surface, leading to a great improvement of growth rate.[26]

Besides, the growth rate is also elevated by increasing the methane/hydrogen ratio. It is easy to understand that more CH_4 leads to more rapid growth. However, nitrogen may also affect the dissociation of the CH_4. We have therefore examined the radicals and species of the plasma by the optical emission spectroscopy (OES, Jobin–Yvon iHR-320) equipped beside the chamber. The OES which contains the Balmer atomic hydrogen emission lines, the CN, CH and C_2 Swan system from the methane, hydrogen, and nitrogen plasmas is shown in Fig. S3 of the supplementary material. While Figs. 5(a) and 5(b) here show the extracted OES intensity of the $I(C_2)$ (516.4 nm)/I_β (486 nm) and I(CH) (431 nm)/I_β (486 nm) as a function of the CH_4/H_2 ratio. The trends of C_2 and CH are quite consistent with the growth rate, demonstrating that the nitrogen-induced high dissociation of the reaction gas CH_4 is responsible for the observed growth rate enhancement.

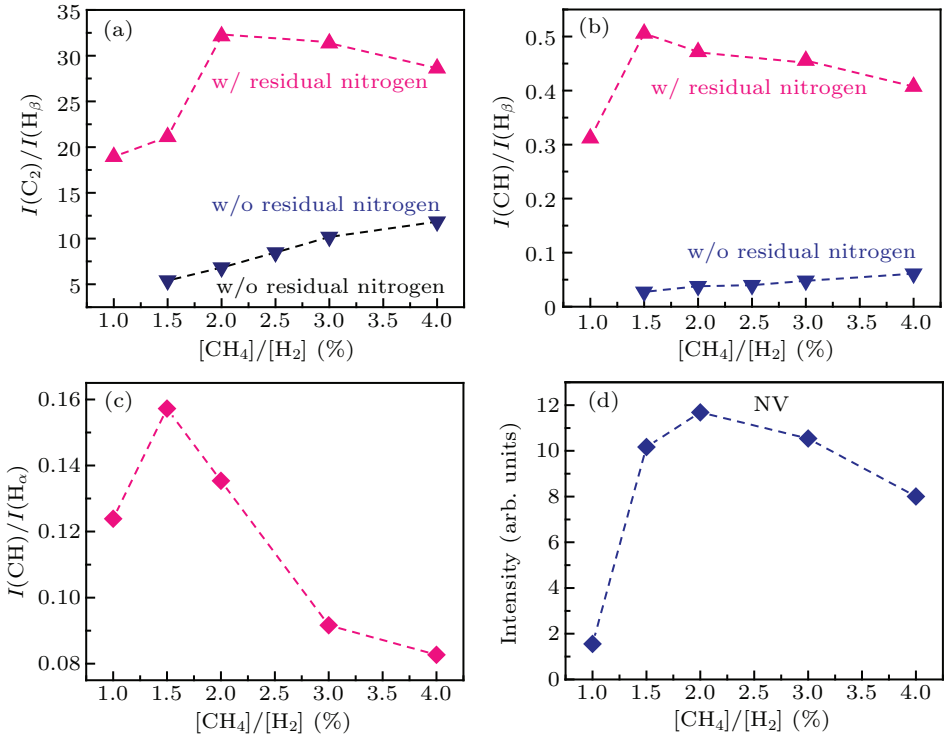


Fig. 5. Diamond films with an increase in the CH_4/H_2 gas ratio: (a) $I(C_2)$ (516.4 nm)/I_β (486 nm) ratio from OES with residual nitrogen or not; (b) I(CH) (431 nm)/I_β (486 nm) ratio from OES with residual nitrogen or not; (c) I(CN) (386 nm)/I_α (656 nm) ratio from OES with residual nitrogen. (d) The intensity of NV as a function of the methane to hydrogen ratio.

Moreover, Figs. 5(c) and 5(d) show the OES intensity of the CN radicals and the NV emission intensity extracted from the PL spectra in Fig. 1(b). The trends of the two plots are quite similar, indicating that the NV centers in the films are mainly doped by the incorporation of the CN radicals. The intensity of the CN radicals in Fig. 5(c) is found to increase firstly then decrease as a function of the CH_4/H_2 ratio. In the presence of the residual nitrogen in the CH_4/H_2 mixture, the

CH_4 would react with N_2 to form CN radicals. In this way, more CH_4 will produce more CN radicals, leading to an increased intensity of the CN radicals. However, with the CH_4 concentration further increase, the intensity of the CN radicals tends to decrease. Y. Su and A. Tallaire *et al.* have reported that the O-related radicals decomposed from N_2O might limit the N-doping concentration in diamond films.[23,24] Therefore, combining with a leakage issue in our system, it is speculated that the decrease of the CN radicals as well as the NV emission in the growing gas mixture is due to the co-doping of oxygen. It is possible that more oxygen may participate in the gas phase reaction with the concentration of methane increase, thus limiting the incorporation of nitrogen into the crystal. In this way, a downward trend of the CN radicals and NV centers will be observed.

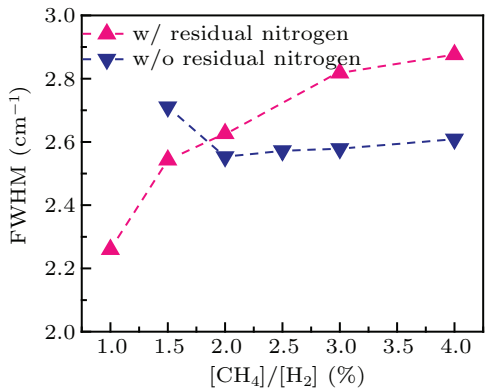


Fig. 6. FWHM of Raman scattering spectra with residual nitrogen or not as a function of the methane to hydrogen ratio.

As the growth rate increases, the crystalline quality may drop. Micro-Raman (Horiba JY HR-800) are thus used to characterize the crystalline quality. Figure 6 shows the extracted full-width at half maximum (FWHM) of the characteristic diamond peak at 1334 cm^{-1} from the Raman spectra [see Fig. S4] for all the samples and the counterpart. Generally, the FWHM monotonously increases, which accords well with the growth rate. The x-ray rocking curves (XRCs) also show the same trend [see Fig. S5]. Detailed analysis of Raman and x-ray diffraction (XRD) spectra can be found in the supplementary material. For the samples with unintentional nitrogen, the FWHM tends to increase as a function of CH_4/H_2 ratio, indicating that methane addition in the gas degrades the crystalline quality with much higher growth rate. This can be explained that at lower methane concentrations, the atomic hydrogen ratio in the gas is relatively high, and thus non-diamond species can be etched off, improving the quality of the diamond films.[27] For the counterpart, however, the FWHM shows only a slight increase as a function of the CH_4/H_2 ratio, indicating a weak dependence of the crystalline quality on the CH_4/H_2 ratio. The high FWHM of the sample at CH_4/H_2 ratio of 1.5% can be mainly attributed to an unduly etching effect of hydrogen at such low methane concentration. Despite the relative high fluorescence intensity of the NV center obtained at the CH_4/H_2 ratio of 1.5%–2%, the value of FWHM is low. This is a very interesting finding with residual gas during growth, and possibly ascribed to the oxygen presence which have a beneficial effect to etch away unwanted defects.[28]

4. Suppression of the residual nitrogen

As discussed above, the unintentionally incorporated nitrogen can significantly alter the properties of the diamond films. Moreover, the residual nitrogen doping is uncontrollable, leading to surface pollution inside the vacuum system and failure to achieve the results required by the vacuum process.[29,30] Therefore, a good background vacuum chamber system, which contains very little nitrogen, is always required.[31] However, sometimes the leakage is inevitable especially in the active gas mixture. So, researchers from different countries have come up with different solutions. Here, a novel method has been tried in this work, a deep hole (diameter of 2 mm) in the center of the susceptor is designed to suppress the nitrogen incorporation in the leaked chamber. The diamond substrate is placed in the same position as that without a deep hole. Detailed description of the design can be found in the supplementary material.

The surface morphology of the samples grown at the CH_4/H_2 ratio of 4% with a deep hole in the center of the susceptor or not are shown in the inset of Fig. 7. The sample grown with a deep hole has the same growth parameters (MW power, pressure, temperature, and time, etc.) with the one without a hole. It can be clearly seen that the sample with a deep hole has a much smoother surface, and no typical step bunching morphology is found. Besides, the modified susceptor geometry leads to the change of the growth rate to only 8 μm/h, showing a five-fold decrease. In addition, the PL spectra show that the NV-related emissions almost disappear, comparable to the PL of the substrate. Thus, it can be deduced that the characteristic behaviors induced by nitrogen impurity can be almost suppressed if a deep hole is designed in the center of the susceptor. However, when we use the OES to diagnose the plasma again, no obvious difference can be found, needing further investigation.

In this case, we have employed the COMSOL Multiphysics® to investigate the plasma characteristics in a cylindrical resonant cavity of the MPCVD system. It is noted that no obvious change has been observed in the center area of the plasma, but the electron behavior in the plasma just above the susceptor has changed a lot, with its intensity reduced by $\sim 40\%$ and its temperature decreased by ~ 0.02 eV, respectively. Meanwhile, simulation shows that the atomic hydrogen concentration increased slightly in the area near the center of the surface of the susceptor due to the reduction of electron temperature and density. Moreover, the concentrations of N and NH have also been found to decrease when a deep hole

was introduced into the susceptor. The molar concentration of N decreased more than an order of magnitude and the molar concentration of NH reduced by about 10%. Since the reduction of the concentrations of N and NH in the case with a deep hole, residual nitrogen can be effectively suppressed into the diamond film. It is believed that the high energy electron has a significant effect on the hydrogen desorption from the diamond surface during growth.[8,32–34] The change of the electron temperature and density near the substrate surface may have a great effect on the hydrogen coverage on the growing surface, changing the growth mode from mass transportation to surface kinetics. In this way, the N-related radicals formed at the center of the plasma is not easy to be incorporated into the diamond surface due to the high hydrogen surface coverage.

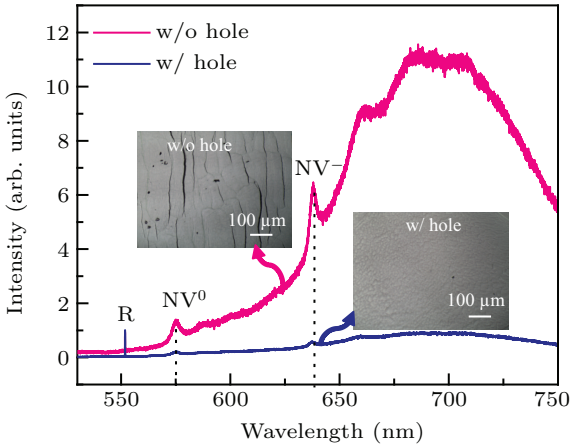


Fig. 7. PL comparison diagram on the samples with a deep hole or not. Insets are the microscopy images of the methane-to-hydrogen at 4% diamond with a deep hole or not (800×600 μm^2).

The detailed nitrogen suppression mechanism for the case with a deep hole by simulation will will be discussed in another work.

5. Conclusions

Due to the thermal effect, a minor vacuum imperfection could result in a significant unintentional nitrogen incorporation in MPCVD-fabricated diamond material. The doping behavior of the leaked air is somewhat different to pure nitrogen doping because of the co-existence of oxygen in air. With a well-designed susceptor geometry, the residual nitrogen incorporation could be readily suppressed in a slightly leaked system due to the modification of the plasma characteristic.

Acknowledgements

Project supported by the National Key R&D Program of China (Grant Nos. 2018YFB0406502, 2017YFF0210800, and 2017YFB0403003), the National Natural Science Foundation of China (Grant Nos. 61974059, 61674077, and 61774081), the Natural Science Foundation of Jiangsu Province, China (Grant No. BK20160065), and the Fundamental Research Funds for the Central Universities.

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