Surface conductivity enhancement of H-terminated diamond based on the purified epitaxial diamond layer

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Abstract:

Diamond-based semiconductor with high electrical conductivity is a key point in diamond device development. In this paper, a thin single-crystal diamond layer of high quality was epitaxially grown on a commercial tool-grade diamond seed by incorporating active O atoms from the typical growth environment. Subsequently the H-termination density was enhanced on the diamond surface by exposure to the pure hydrogen plasma, and the surface conductivity of H-terminated diamond was analyzed in detail. The thin epitaxial layers on the high pressure high temperature (HPHT) diamond seeds show lower resistance than the ones on the chemical vapor deposition (CVD) diamond seeds, which could be comparable with the lowest values reported. After the thin diamond layers were grown with and without addition of O_2, the carrier mobility in the conductive channel increase to almost 80 $cm^2 \cdot V^{-1} \cdot s^{-1}$ under O_2 contained condition, much higher than those without O_2 incorporation. The ionization scattering is dominant to the carrier mobility compared with the surface scattering. The higher carrier mobility is attributed to the lower impurity density in the epitaxial layer, which is because the active O atoms could purify the epitaxial layer by removing or reducing Si and N related impurities.

Key word: diamond; epitaxial growth; H-termination; oxygen addition; surface conductivity; electronic device

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Introduction

Diamond has been considered as one of the most promising wide band semiconductors. Its many excellent properties including the wide bandgap, highest thermal conductivity, high carrier mobility and so on, make it a very good prospect for high-frequency and high-power device application[1-2]. Electronic device research based on the H-terminated diamond is still a hot topic due to the unique 2-dimensional conductive channel on the H-terminated diamond surface[3-4]. This channel has been found to be P-type conductive with the surface resistivity of 10^4-10^6 Ω and carrier density of 10^{12}-10^{14} cm^{-2}[5-7].

Recent research has focused on device development using H-terminated diamond passivated by high-*k* dielectric materials, which makes the high-temperature and high-voltage operation of diamond devices possible[8-9]. However, until now, there are still two challenges to overcome for the diamond device development. One is the lack of a reliable supply of diamond material perfect enough for electronics. Although huge progress has been made in CVD synthesis, the background concentration of nitrogen and silicon is still hard to limit[10-11]. High purity single crystal diamond layers have always been obtained on the high quality single crystal diamond seed under the harsh growth conditions, which leads to a high production cost. For the electrical or even quantum application, requirement of high purity diamond crystal is an even greater obstacle: the background concentration of nitrogen and silicon to reach less than one part per billion[12]. Moreover, the surface conductivity of H-terminated diamond is restricted by the surface quality and conditions. Until now, many methods have been developed to improve the surface conductivity. The spontaneous polarization model was proposed to show that the carrier density in the channel on diamond surface was proportional to the density of the C–H dipoles, which could be improved by using (110) oriented diamond[13]. The different absorbates were introduced on the H-terminated surface to improve the carrier density including NO_2[14], WO_3 and ReO_3[15]. Until now, there are few reports on the carrier mobility improvement. Generally, the carrier mobility and the density in the conductive channel are contradictory. The carrier mobility will be limited by the carrier density improvement. Meanwhile, the 2-dimensional hole gas in the conductive channel is quite sensitive to the surface roughness, due to the surface roughness scattering for the H-terminated diamond or the interface scattering between the H-terminated diamond and the dielectric

layer[16], which will obviously decrease the carrier mobility. In fact, H-terminated diamond is often obtained by treating polished diamond with low roughness with a microwave hydrogen plasma treatment, which will make the surface rough again due to the anisotropic etching and limit the surface mobility improvement.

In this paper, we reported a method to obtain a high-performance conductive channel on the tool-grade diamond seeds by epitaxially growing a thin single crystal diamond layer of high quality. By incorporating O atoms in the growth environment, a thin single crystal diamond layer with thickness less than 1μm was obtained and no obvious impurities could be found by PL spectrum. Furthermore, the H-terminated diamond with high-density bonding H atoms was prepared by hydrogen plasma treatment for the short time after the epitaxial growth of single crystal diamond layer. The surface conductivity of the H-terminated epitaxial diamond layer especially for the carrier mobility was improved dramatically compared with the one without O atoms addition. The corresponding mechanism was investigated and analyzed in detail.

Materials and Methods

Materials

The commercial tool-grade HPHT Ib type and CVD single crystalline diamonds with roughness of below 5 nm were used as the seeds. The HPHT seeds were from the Sino-Crystal Diamond Co. Ltd and the CVD ones were from the Beijing Qite Diamond Tools co. Ltd in China. They are typical tool-grade diamond seeds. The gas sources during the treatment process included hydrogen, oxygen and methane and all of them were from Beijing Huanyu Jinghui City Gas Technology co. Ltd in China with purity of equal or greater than 99.999%. H_2SO_4 and HNO_3 were analytically pure from Beijing Chemical Works and the deionized water was home-made.

Preparation of conductive channel and epitaxial layer on diamond surface

Before being placed in the CVD chamber, the seed samples were boiled in the solution of H_2SO_4 mixed HNO_3 with volume ratio of 5:1 for 30 min. And then after being cleaned by deionized water, the samples were transferred into the microwave (MW) CVD chamber and the chamber was pumped to 10^{-4} Pa by using mechanical pump and turbo molecular pump. With addition of hydrogen and ignition by MW energy input, the samples were heated at 880 °C by plasma and the carbon source was added

to grow diamond epitaxially without intentional plasma etching. To compare the property differences, the HPHT and CVD samples were divided into 2 sets. Each set included one sample with introduction of oxygen and one without oxygen addition during the diamond growth processes. The detailed experimental design is shown in Table 1. The growth time of single crystal diamond layers for all samples was 15 min. After the epitaxial growth, the carbon and oxygen sources were switched off and the diamond was exposed in the hydrogen plasma at 800 °C for 5 min, to enhance the hydrogen atom concentration on the diamond surface[17]; this produced a smooth H-terminated surface with high quality[18].

Table 1 The experiment parameters of epitaxial growth of single crystal diamond layers on different diamond seeds.

Sample No.	Sample type	Temperature/°C	CH_4/sccm	O_2/sccm	Time/min
1	HPHT 1	880±20	3	0	15
2	HPHT 2	880±20	3	0.6	15
3	CVD 1	880±20	3	0	15
4	CVD 2	880±20	3	0.6	15

Characterization of conductive channel and epitaxial layer on diamond surface

Before the epitaxial growth of the single crystal diamond layer, the crystal quality of all the seeds was characterized by the X-ray diffraction rocking curve using a monochromized X-ray source of Cu-$K\alpha_1$ with a Ge (220) four-crystal monochromator. After the growth, the surface morphology of the thin single crystal diamond layers was observed by atomic force microscope(AFM). The differences between the epitaxial layers and seeds were recorded by a luminescence imaging system (DiamondView) provided by the Diamond Trading Company (DTC). In this instrument, samples were excited at wavelengths shorter than 230 nm by a filtered xenon arc lamp source. After hydrogen plasma treatment, the surface bonding characteristic of the thin grown layer was analyzed by attenuate total reflection (ATR) with the reflective crystal of Ge. The impurity content in the grown diamond layers was detected by photoluminance (PL) using a laser wavelength of 514 nm. To get the accurate thickness of the epitaxial diamond layer and the element distribution in the layer, time of flight secondary ion mass spectroscopy (TOF-SIMS) was used to test the trace element distribution using a 2keV Cs ion primary beam. The beam current of Cs source is 71nA

and it is constant during the whole test. The CN, O and Si ions were collected to show N, O and Si element distribution in the layers by Cs ion sputtering. The intensity collected is the ion beam current considering the matrix effect. Due to lack of standard sample, the absolute element content cannot be obtained. However, it can be used to compare the element content in the epitaxial layer grown with and without O_2 addition qualitatively. The sputtering zone size is 200μm and the depth profile of the element distribution was obtained by calculating the sputtering time and the depth of the sputtering zone. The surface conductivity of the H-terminated diamond layers after exposure to hydrogen plasma was obtained by Hall-effect test using four Au electrodes in the corners of the square samples. The surface conductivity was characterized by using Hall effect and the test for all samples was conducted with the magnetic field of 0.5T at room temperature. Based on the crystal quality, surface morphology, termination bonding and impurity content, the effect of O addition on the surface conductivity of H-terminated diamond was presented and explained.

Results

Crystal quality of diamond seeds before epitaxial growth

The rocking curves of HPHT and CVD seed samples were tested by XRD to show the crystal quality. Based on the tested data, the characteristic diffraction peaks of the seeds and the corresponding full width at half maximum (FWHM) of these peaks by fitting these peaks were listed in Table 2. For the two HPHT seeds, the feature diffraction angles were 59.89^{o} and 59.84^{o}, respectively, which are close to the standard diffraction peak of (004) plane of the perfect diamond crystal. Conversely, for the CVD, both diffraction peaks shift to the lower angles, which means there are some intrinsic tensile stress in the CVD seeds due to incorporation of impurities or other defects during the epitaxial growth process. The FWHM values of rocking curves of HPHT seed samples were 0.008 ^{o} and 0.011^{o}, which are common for the commercial HPHT Ib single crystal diamond seeds. By comparison, the CVD diamond seeds have a slightly larger FWHM. According to the Williamson-Hall method, the dislocation density in the crystals can be estimated from the FWHM of the characteristic diffraction peaks[19]. Although the dislocation density is also affected by other impurities and defects, this estimate qualitatively shows that the crystal quality of CVD seeds are not as good as the HPHT seeds, generally used as the cutting tools.

Table 2 The diffraction angles and the fitted FWHM of rocking curves for different types of seed samples tested by XRD

Sample No.	Sample type	Diffraction angle/(°)	FWHM of rocking curve/(°)
1	HPHT 1	59.89	0.008
2	HPHT 2	59.84	0.011
3	CVD 1	58.68	0.039
4	CVD 2	57.56	0.029

Surface quality of the epitaxial layers after diamond growth

The surface morphology of each thin, epitaxially-grown diamond layers grown on the HPHT and CVD seeds were measured by AFM, as shown in Fig.1. The epitaxial layer grown on the HPHT diamond seed without added O_2 has a comparatively smooth surface. When O_2 was added, some etching dots appeared. The epitaxial single crystal diamond layers on the CVD seeds both show many parallel grooves resulting from the mechanical grinding process. The corresponding surface roughness of single crystal diamond layers on HPHT and CVD diamond seeds were 1.92nm, 6.27nm, 2.28nm and 1.29nm, respectively. All of these samples keep a low surface roughness, except that the epitaxial layer grown on the HPHT diamond seed with O_2 addition shows a little higher surface roughness up to 6 nm. So, after the epitaxial growth of thin layer, the surface roughness of the single crystal diamond doesn't increase much.

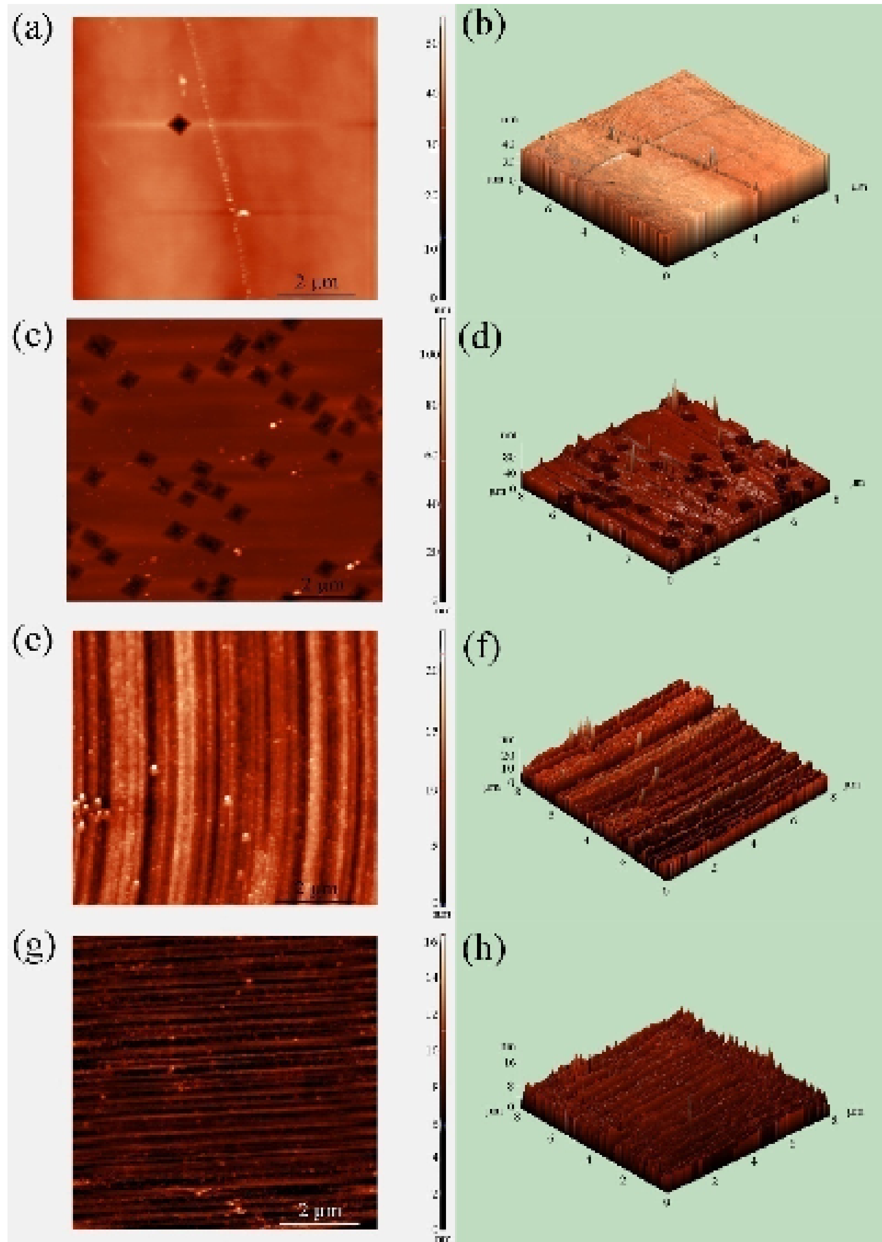


Fig.1 Surface morphologies of epitaxially grown single crystal diamond layers observed by AFM. Both epitaxial layers grown with and without O_2 incorporation on HPHT and CVD diamond seeds are shown. These are: (a), (b) without O_2 incorporation on the HPHT seed; (c),(d)with O_2 incorporation on the HPHT seed; (e),(f)without O_2 incorporation on the CVD seed; (g),(h) with O_2 incorporation on the CVD seed.

Impurity characterization of the epitaxial layers

To distinguish the epitaxial layers from the substrates based on the luminescence effect, the optical microscope images and DiamondView images were obtained, as shown in Fig.2. The growth side (left side) without O_2 addition shows the similar dark color with the HPHT diamond seed in Fig.2(a) and Fig.2(b). By contrast, the growth

surface with O_2 addition shows bright green fluorescence. Meanwhile, for the CVD, w incorporating O_2 changes the fluorescence from the orangy red to yellow. It has been reported that the orangy red fluorescence emanates from the emission of NV^0 center[20]. As the color changes to yellow, the concentration of nitrogen decreased. Also, the green or blue color is associated with the high purity diamond or the H3 center[21]. That means higher purity and fewer impurities of thin grown layer on both the HPHT and CVD seeds were improved by the O_2 addition.

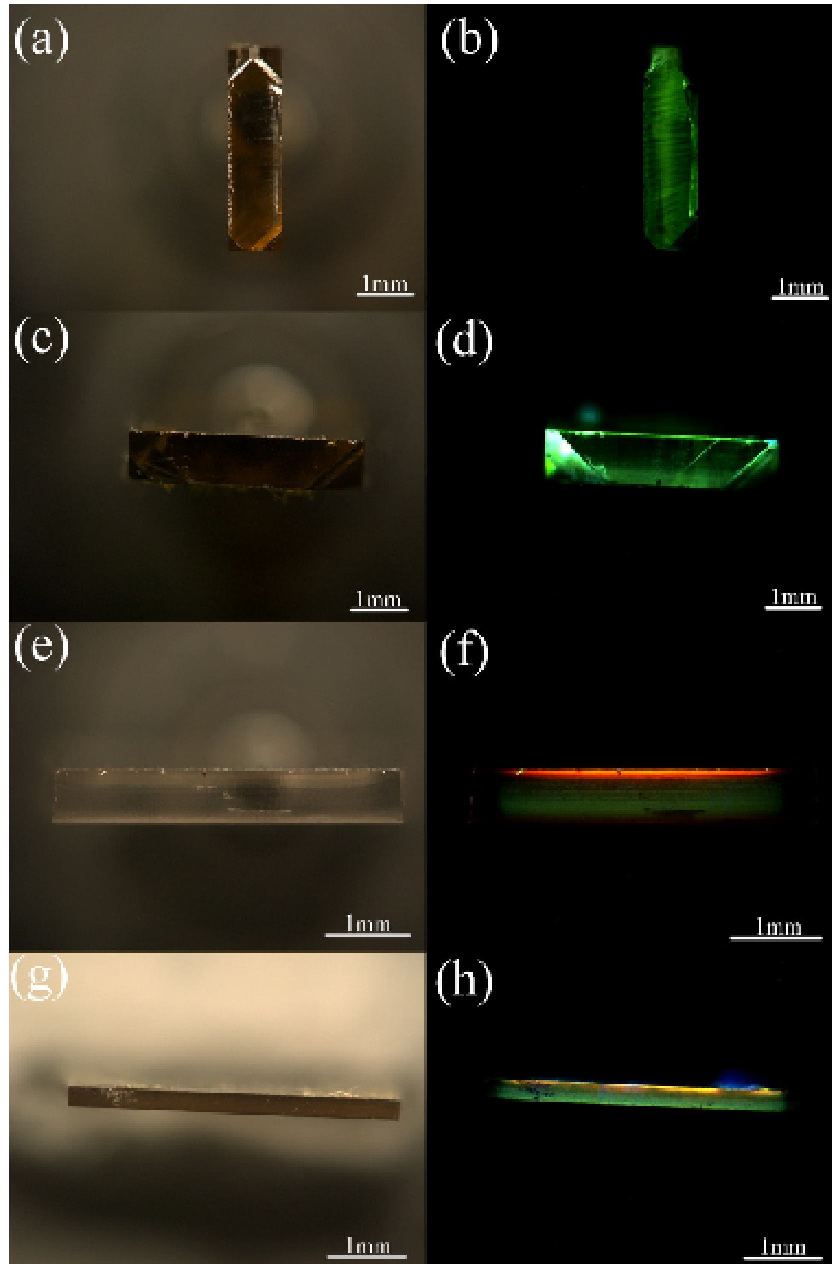


Fig.2 Optical microscope images and DiamondView images of epitaxially grown single crystal diamond layers were obtained. The fluorescence from the epitaxial layers grown with and without O_2 incorporation was compared. (a),(b) without O_2 incorpo-

ration on the HPHT seed; (c),(d) with O_2 incorporation on the HPHT seed; (e),(f) without O_2 incorporation on the CVD seed; (g),(h) with O_2 incorporation on the CVD seed.

To further compare the impurity concentration in the epitaxial layer, photoluminescence spectra were collected for the HPHT samples. We tried to test the epitaxial layer of single crystal diamond after growing for 15 min directly. However, there was no obvious change between the original diamond seed and the epitaxial layer. That is because the detected photoluminescence signal comes from several tens of micrometers beneath the sample surface, while the thickness of the epitaxial layer is too small. Therefore, most of the signal is from the diamond seed and the differences between the epitaxial layer and seed are hard to distinguish. In order to obtain spectra only from the epitaxial layer, the growth time was extended to 15 h and the thickness of the epitaxial diamond layer prepared with O_2 addition was about 30μm. Considering that the luminescence characteristics of the epitaxial layers on CVD seeds depends strongly on the crystal quality of substrates and sometimes, the lower quality of the CVD seed made understanding the chemistry useless, therefore, only the PL spectra of epitaxial layers on HPHT diamond seeds were compared. The corresponding PL spectra of epitaxially grown single crystal diamond layers with and without O_2 addition on HPHT diamond seeds were shown in Fig.3. It can be seen that without O_2 addition during the epitaxial growth process, the peaks at 572 nm, 575 nm, 613 nm, 637 nm and 737 nm were found obviously. These peaks at 572 nm, 575 nm, 637 nm and 737 nm are related to the intrinsic Raman excitation, $[N\text{-}V]^0$, $[N\text{-}V]^-$ and Si-V complexes, respectively[22]. The peak at 613 nm is often observed in the natural single crystal diamond, especially in untreated brown and pink diamond crystals[23]. To the best of our knowledge, there is no evident relationship between the peak at 613 nm and nitrogen content or its aggregate states in crystals. It should be a nickel-containing defect resulting from the HPHT growth process[23]. With O_2 addition, the grown thin layer shows typical characteristic of high purity single crystal diamond without any N-V and Si-V complexes. Note that there is still a weak peak at 613nm with O_2 addition in the epitaxial layer grown. It might be that the nickel-containing defect is sensitive to the photoluminescence effect[23].

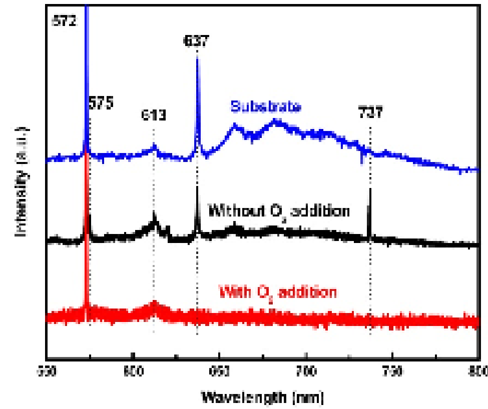


Fig.3 PL spectra of epitaxial single crystal diamond layers grown with and without O_2 addition on HPHT diamond seeds

To determine the thickness of the epitaxial layer and the impurity element distribution, SIMS was used to test the CN, O and Si ion distribution in the epitaxial layer and the corresponding spectra of O, Si and N elements are shown in Fig.4. Both epitaxial layers grown with and without O_2 show an obvious, abrupt change in the N element distribution, which indicates the interface between the diamond seed and the grown layer. Based on the etching pit depth and measurement time, it can be obtained that the thickness of the epitaxial layers grown without and with O_2 are 1227nm and 543nm, respectively. The results of high-content N related impurities in the diamond seed and the decreased impurity concentration in the epitaxial layer correspond well to the PL spectra. However, the Si element distribution do not show any change in the depth for both layers, while the impurity content increases near the surface for the layer grown without O_2 incorporation compared with the layer with O_2 incorporation. For the O element distribution, the epitaxial layer grown in CH_4/H_2 environment also presents the sharp increase near the surface. The epitaxial layer grown in O_2 contained environment shows a little steady increase. The increase of Si and O content near the surface in the epitaxial layer obtained without O_2 addition indicates the impurities become rich at the later growth stage.

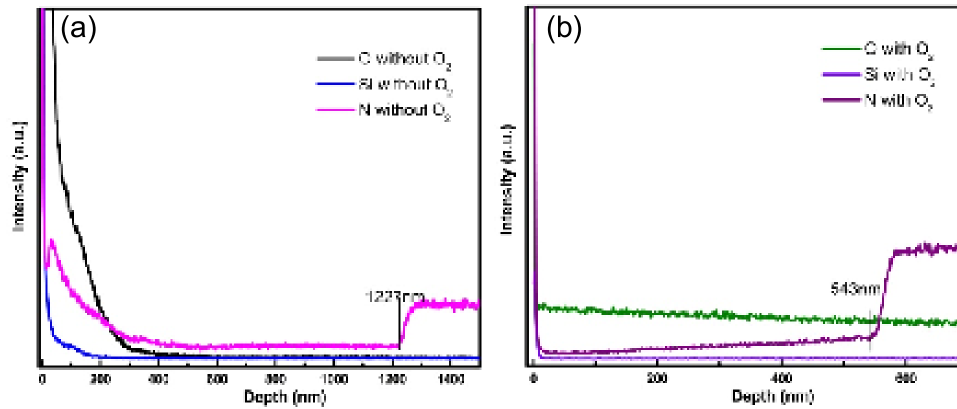


Fig.4 The impurity element distribution in the epitaxial layer on the HPHT diamond seed was tested by secondary ion mass spectroscopy (SIMS). The layers grown without (a) and with (b) O_2 addition were compared.

Surface bonding characteristics of the epitaxial layers

To increase the surface conductivity of the epitaxial diamond layer, the diamond was treated with pure hydrogen plasma after the epitaxial growth. Then the bonding characteristics of the treated diamond layer surface were measured by attenuate total reflection (ATR). After the treatment, C=O bonding corresponding to the bands near 1770 cm^{-1} (resulting from the oxidation of acid boiling) almost disappeared for all the samples, as shown in Fig.5. Meanwhile, the C-H stretching vibration bands corresponding to CH_2 and CH_3 groups can be observed clearly for the treated samples, indicating the formation of H-termination on the diamond surface. Generally, the bands at 2850 cm^{-1} and 2924 cm^{-1} are assigned to the symmetric stretching vibration of CH_3 and asymmetric stretching vibration of CH_2, respectively [24]. The C-H stretching mode is related to the reconstruction of diamond surface, resulting from the termination chemical reaction. Besides these peaks, the OH vibration bands at 1650 cm^{-1} and 3000-3700 cm^{-1}, corresponding to the stretching vibration and bending vibration of H_2O respectively, can also be found, which is because the ATR test was conducted in the atmosphere. For all the epitaxially grown single crystal diamond layers with and without O_2 addition on HPHT and CVD diamond seeds, they show the similar active groups and the corresponding stretching characteristics.

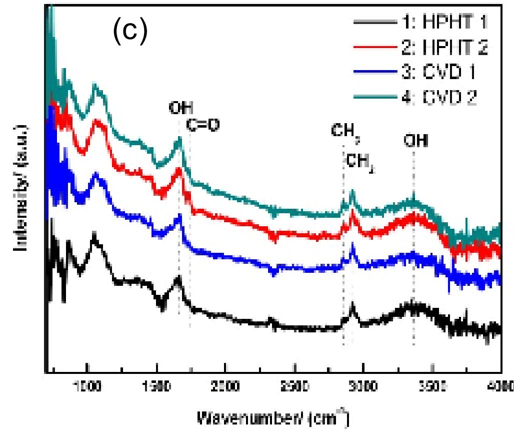
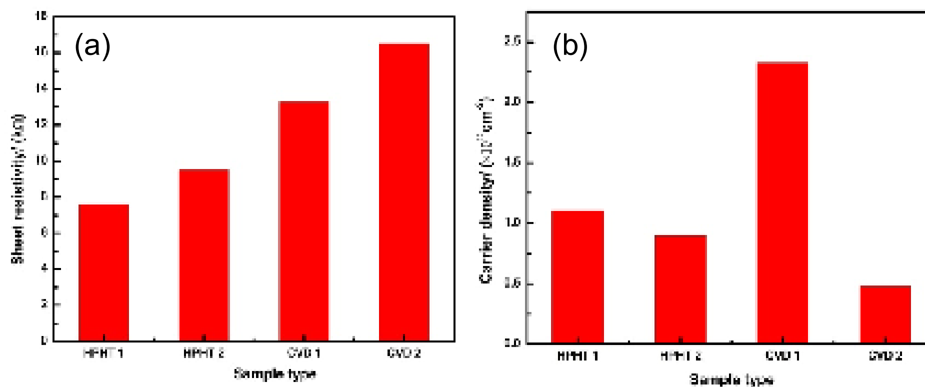


Fig.5 The FTIR-ATR spectra of epitaxially grown single crystal diamond layers with and without O_2 addition on HPHT and CVD diamond seeds

Surface conductivity of the epitaxial layers

After the hydrogen plasma treatment, the surface conductivity of the epitaxial diamond layers was tested and compared, as shown in Fig.6 and Table 3. The sheet resistivity of the epitaxial layer on the HPHT seeds is less than 10 kΩ, which could be comparable to the lowest values previously reported[25], while those on the CVD seeds present slightly higher values. Meanwhile, the carrier mobility of the samples shows distinctly different characteristics. For the epitaxial layer on the HPHT seeds grown without and with O_2 addition, the carrier mobility is 50.8 and 73.1 $cm^2 \cdot V^{-1}s^{-1}$, respectively. In contrast, the carrier mobility for the epitaxial layer on the CVD seeds are 20.1 and 78.6 $cm^2 \cdot V^{-1} \cdot s^{-1}$ without and with the addition of O_2 during growth, respectively. Note that the epitaxial single crystal layers on both HPHT and CVD seeds show a much higher carrier mobility when O2 was added during the growth than those without the O_2 addition. Accompanying the obvious increase of the carrier mobility, it is a decreasing trend in the carrier density correspondingly.



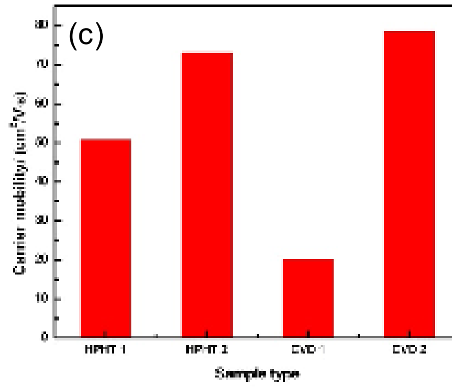


Fig.6 The surface conductivity of epitaxial single crystal diamond layers on HPHT and CVD diamond seeds was measured after hydrogen plasma treatment. Surface resistivity (a), carrier density (b), and carrier mobility (c) in the conductive channel were compared for the epitaxial layers grown with and without O_2 addition.

Table 3 The surface conductivity for different types of seed samples tested by Hall test

Sample No.	Sample type	With and without O_2 addition	Sheet resistance /kΩ	mobility /cm^2 $(V{\cdot}s)^{-1}$	density /cm^{-2}
1	HPHT 1	without	7.632	50.8	1.11E+13
2	HPHT 2	with	9.477	73.1	9.01E+12
3	CVD 1	without	13.25	20.1	2.33E+13
4	CVD 2	with	16.51	78.6	4.81E+12

Discussion

Until now, diamond based device development has been limited by the performance of the conductive channel. Recently, the surface conductivity of H-terminated diamond was improved by T Wade[25], et al. They reported that increasing the surface roughness is beneficial to enhance the diamond's surface conductance due to the carrier density increase in the conductive channel. However, the carrier density shows the counter trend. In our situation, it is exciting that a carrier mobility of almost 80 cm^2/Vs was obtained without further optimization on common commercial HPHT and CVD single crystal diamond made to be used as cutting tools. The thin epitaxial layers on the HPHT seeds show a lower resistance compared with the ones on the CVD seeds, which is associated with the crystal structure defects, such as the dislocations. Furthermore, the carrier mobility improvement of the H-terminated diamond was achieved by growing a thin high-quality epitaxial layer on both kinds of seeds by ad-

dition of O_2 into the growth environment. On one hand, the thickness was limited to less than 1 μm, not increasing the surface roughness obviously. The low surface roughness was kept reducing the surface scattering by suppressing the fast step growth during the late growth stage. On the other hand, the thin epitaxial layers inherit the dislocations rooting from the diamond seeds, and the impurities resulting from the chamber or the bell-jar may be incorporated into the grown layer. Generally, a buffer layer should be introduced to improve the quality of the epitaxial layer for most of crystals synthesized by CVD[26-27]. In our situation, oxygen atoms were incorporated to the typical growth environment of diamond with active hydrogen atoms and the carbon source, and the epitaxial layer was purified. It is widely accepted that the quality of diamond films can be improved by active oxygen atoms rooting from the preferential etching of the non-diamond carbon[28]. Also, there are some reports on the quality improvement of the diamond by the oxygen plasma treatment [29] or oxygen addition into the hydrogen-methane plasma [30]. In our situation, we can find that nitrogen impurities in the grown layer under O_2 added condition shows larger reduction ratio compared with the one in no O_2 environment from SIMS. Meanwhile, the Si and O elements cannot be found in the epitaxial layer after O_2 addition, indicating that Si and O impurities resulting from the quartz bell-jar etching were removed completely. It means that oxygen addition can reduce and remove the nitrogen, silicon related impurities obviously. Although no clear evidence was found, it can be speculated that the OH and O radicals in the plasma will react with these impurity and form such nonreactive species, such as NO/NO_2, SiO/SiOH, H_2O and so on, as shown in red circle areas in Fig.8.

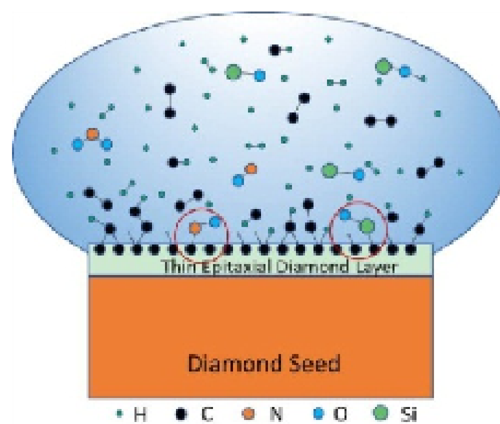


Fig.7 The purification mechanism of the epitaxial diamond layer in the O_2 contained plasma

After the hydrogen plasma treatment, the epitaxial layers on both HPHT and CVD diamond seeds with O_2 addition show much higher carrier mobility than those without O_2 addition. In fact, the carrier mobility in the conductive channel of H-terminated diamond surface is generally limited by the carrier scattering resulting from the surface roughness and the ionized impurity. The surface roughness of 4 samples was compared. Although the epitaxial layer grown with O_2 addition on HPHT seed present a little high surface roughness, it is still less than 10 nm, which will reduce the mobility slightly[16, 25]. Thus, the ionized impurity scattering will dominate the carrier mobility. Due to the oxygen atoms incorporation, the impurities in the epitaxial diamond layer were reduced dramatically. Correspondingly the mean free paths of carrier in the conductive channel will be increased, and the carrier mobility enhances. It can be speculated that the conductive performance of the H-terminated diamond layer would be improved further by optimizing the growth condition and treating the surface with some favorable absorbates such as NO_2[31], MoO_3[32], V_2O_5[33] and so on. It is also noteworthy that the purified thin epitaxial layer could be prepared using this simple method. It can satisfy the requirement of preparing high purity diamond and coupling light from the diamond to other solid-state photonics, which may be also promising in the quantum communication application.

Conclusions

In this paper, a low-cost method to develop the high-performance conductive channel on the diamond was proposed by growing a thin single crystal diamond layer on both HPHT and CVD tool-grade seeds with incorporation of active O atoms in the typical growth environment, and hydrogenating the epitaxial layer surface. The thin layers on the HPHT seeds show lower resistance compared with the ones on the CVD seeds, which is associated with the crystal structure defects, such as the dislocations. After the thin layers were grown on both the HPHT and CVD seeds with addition of O_2, the conductive channel on H-terminated diamond surface show much higher carrier mobility than those without O_2. The surface roughness of all samples is below 10 nm, which means the surface scattering resulting from the surface roughness, contributes to carrier mobility slightly. The carrier mobility is mainly affected by the ionization scattering stemming from impurities. The higher carrier mobility for the H-terminated diamond layer grown with O_2 addition is due to the lower impurity density

in the epitaxial layer, which is because the active O atoms could purify and repair the epitaxial layer by removing or reducing the Si and N related impurities.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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