Electrical and optical measurements of CVD diamond doped with sulfur

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Homoepitaxially grown chemical-vapor-deposition diamond to which H₂S was added during the growth was analyzed by electrical and optical measurements in order to investigate the nature of sulfur as a dopant in diamond. Hall measurements were carried out at low and high temperatures. In the low-temperature range (175-290 K), p-type conduction was found, with an activation energy of 360 meV. A hole concentration of 5.6×10^{12} cm⁻³ with a mobility of 270 cm² V⁻¹ s⁻¹ was measured at room temperature. On the other hand, at high temperatures (650-900 K), n-type conductivity was observed with a carrier activation energy of 1.55 eV. Using photoconductivity spectroscopy, four dominant ionization energies are detected at 371 meV, 479 meV, 1 eV, and 2 eV. Excited states of the two shallow levels were deduced from oscillatory photocurrent and photothermal ionization. The first ionization energy is attributed to the effect of an unknown boron contamination source, in agreement with the p-type conductivity measured. The origin of the level detected by Hall measurements at 1.55 eV and by photoconductivity at 2 eV is not clear yet, and it has been tentatively attributed to nitrogen impurities. The origin of the new acceptor state measured at 479 meV above the valence-band edge is also discussed.

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I. INTRODUCTION

Diamond is a promising semiconductor material with a large potential for electronic as well as biosensor applications. Intrinsic and *p*-type diamond (in which boron is the acceptor with an ionization energy of 370 meV) are already available with good quality. However, the question of finding a shallow *n*-type dopant for diamond is still open. In analogy to the conventional dopants used in Si, elements from group V (N and P) and VI (S) have also been studied in diamond. Phosporus and nitrogen are expected to be single donors on substitutional sites, while sulfur should behave as a double donor. Nitrogen has been well characterized as a deep donor with an electronic level at 1.7 eV below the conduction band.¹ The *n*-type doping of chemical-vapor-deposition (CVD) diamond by phosphorus was demonstrated by Koizumi and co-workers,² who added phosphine during the deposition in a microwave-assisted plasma system. Based on photoionization spectroscopy as well as optical absorption, it has been reported that P introduces a level about 600 meV below the conduction band.^{3,4} However, its thermal ionization energy is too high to use P as a conventional donor. Moreover, due to reasons that are not yet known, the carrier mobility measured in P-doped samples is still low, less than $250 \text{ cm}^2/\text{V s.}^5$ As in silicon,⁶ sulfur would be expected to be a double donor in diamond, but until very recently, efforts to obtain *n*-type sulfur doping in diamond were not successful. In 1999, Sakaguchi and co-workers⁷ reported sulfur doping in diamond. In their samples a thermal activation energy of 380 meV and a mobility at RT in the range 250 to 600 $\text{cm}^2 \text{V}^{-1} \text{s}^{-1}$ was measured. These results were very promising, due to the relatively low activation energy and the high carrier mobility. However, later Kalish et al.⁸ showed that in some of those S-doped layers the conductivity was dominated by unintentional doping by boron. Therefore, the *n*-type doping of diamond with S is still controversial. Very recently, in a theoretical study⁹ it is predicted that, when considered as an isolated substitutional impurity in diamond, sulfur would introduce a deep donor level around 1 eV. Other sulfur-related defects in diamond, such as sulfur-boron, sulfur-nitrogen, or sulfur-vacancy complexes are expected to have donor levels around 0.5 eV. No acceptor levels were reported in this case. However, not even the atomic structure of S in diamond is clear up to now. In this paper, we present our investigation in a CVD diamond to which sulfur was added during the growth. We have found that nonintentional boron doping introduces an acceptor level at 371 meV. A new acceptor level has been found at 479 meV. A donor level at 1.55 eV was detected by Hall measurements at high temperatures, and was confirmed by optical experiments.

II. EXPERIMENTAL

The 2- μ m-thick diamond was grown homoepitaxially at 830 °C on a high-pressure high-temperature synthetic Ib diamond (100) crystal by microwave-assisted CVD. Sulfur doping was achieved during growth using H₂S in the reactant gas, with a H₂S/CH₄ ratio of 1000 ppm. Secondary ion mass spectroscopy (SIMS) was used to measure the doping profile as well as the presence of possible contaminants. However, no sulfur was detected in these experiments, which had a lower detection limit of 2×10^{17} cm⁻³. The presence of boron was also investigated, but no boron was detected above the detection limit, 4×10^{16} cm⁻³. In order to characterize the electronic properties, Ti(200 Å)/Pt(100 Å)/Au(3000 Å) contacts in van der Pauw geometry for Hall or in coplanar geometry with a gap of 0.8 mm for photoconductivity measurements were evaporated. Contacts were annealed for 10



FIG. 1. Dark conductivity (σ_D) measured after annealing at 700 K (full line, state A) and after UV-illumination at 77 K for 10 min (dashed line, state B). In state A σ_D is activated with an energy around 1 eV. The higher conductivity in state B vanishes at T > 550 K.

min at 500 °C in formin gas to obtain ohmic behavior. Hall experiments have been performed at low (175-350 K) and at high (700-900 K) temperatures in high magnetic fields (2 T).

Photoconductivity (PC) measurements were made between 77 and 290 K in vacuum, using a liquid-nitrogen cryostat. A helium cryostat was also used to cool the sample to 7 K. The electric fields applied during the experiments were in the range $10^2 - 10^3$ V/cm. Chopped (3.5 Hz) monochromatic light from a halogen lamp was used for optical excitation. The spectral resolution of the setup is about 0.4 meV in the spectral range 0.3 to 1.2 eV. The photoresponse is detected by a lock-in amplifier and normalized to the number of incident photons measured by a pyroelectric detector.

As will be shown below, the existence of a deep level that behaves as an efficient trap for carriers requires the use of UV-light pumping in order to fill shallow levels. Hall and photoconductivity experiments have been performed after annealing at 700 K for 10 min (annealed state or state A), after illumination with deep UV light (state B), or during continuous illumination with deep UV light (state C). A deuterium lamp was used as the UV-light source.

III. RESULTS

Typical dark conductivities (σ_D) measured after annealing the sample (state A) and after UV illumination at 77 K (state B) are shown in Fig. 1. In the annealed state, the sample is highly resistive, showing an activation energy of the conductivity close to 1 eV. However, after UV-light illumination for 10 min, the conductivity of the sample changes dramatically. Starting at low temperatures (77 K), the conductivity increases monotonously towards higher temperatures until 200 K. From 200 to 500 K, the conductivity shows some modulations that we attribute to thermally stimulated currents. When the sample temperature is above 500 K, the conductivity decreases and finally turns into the



FIG. 2. Electron density n_e and mobility μ_e measured by Hall experiments at high temperatures (700–900 K). n_e is activated with an activation energy about 1.55 eV. A low mobility is measured, varying from 1 cm² V⁻¹ s⁻¹ (900 K) to 4.5 cm² V⁻¹ s⁻¹ (750 K).

same activated increase as in the annealed state. This behavior can be obtained reproducibly by applying either UV illumination prior to the experiment or annealing the sample above 500 K. The conductivity results could be explained by the presence of some levels in the band gap that are filled at low temperature during UV illumination. The filling of those levels produces a change in the quasi-Fermi level position, which is shifted towards the conduction or valence band, and hence increases the conductivity of the sample.

Hall experiments were used to verify *n*-type or *p*-type conduction. Without UV-illumination prior to the Hall experiments, the resistivity was too high to be measured at low temperature. Therefore, Hall experiments were carried out at high temperatures in the dark. Below 700 K it was not possible to detect any Hall signal. However, above 700 K a clear *n*-type Hall signal was measured. In Fig. 2 the carrier density (n_{e}) and the mobility (μ_{e}) versus inverse temperature are shown. The electron density is thermally activated between 700 and 900 K with an activation energy of 1.55 eV. The electron mobility changes from about 4.5 cm²/V s at 750 K to $1 \text{ cm}^2/\text{V}$ s at 900 K. The conductivity measured by the Hall effect shows an activation energy of 1 eV, consistent with the results shown in Fig. 1. In this temperature regime, the transport should be mainly dominated by phonon scattering. However, the temperature dependence of the carrier mobility does not agree with the dependence expected for phonon scattering ($\mu \propto T^{-3/2}$). The origin of the low mobility of electrons is still not clear. It is well known that nitrogen introduces a donor level in diamond around 1.7 eV below the conduction band.¹ From our SIMS analysis, it was not possible to detect nitrogen (N) above the system sensibility, 3 $\times 10^{18}$ cm⁻³. However, nitrogen impurities with a concentration below 3×10^{18} cm⁻³ could generate the *n*-type conductivity that is measured. Therefore, it is not possible to conclude that the activation energy of 1.55 eV is produced by residual nitrogen contamination during the CVD growth.

In the low-temperature regime, the sample was too resistive to measure any Hall signal without UV illumination. Therefore, Hall experiments were carried out during or after UV-illumination. Under these conditions, the results clearly



FIG. 3. Hole density p_h and mobility μ_h measured by photo-Hall experiments at low temperatures (200–280 K). The carrier density is thermally activated between 210 and 285 K with an activation energy about 360 meV. The carrier mobility decreases from about 350 cm²/V s at 222 K to 250 cm²/V s at 290 K. Switching the UV illumination off causes a decrease of the hole density. However, the sign of the Hall coefficient is invariably *p* type.

show *p*-type conductivity. In Fig. 3 the dependence of the hole density (*p*) and the hole mobility (μ_h) versus inverse temperature is shown when the experiment is performed under UV illumination. The carrier density is thermally activated between 210 and 285 K with an activation energy of 360 meV. The carrier mobility decreases from about 350 cm²/V s at 220 K to 250 cm²/V s at 290 K. Switching the UV illumination off causes a decrease of the hole density, due to carrier recombination or carrier trapping in deep defects. However, the sign of the Hall coefficient is invariably *p* type. As is demonstrated below, this *p*-type conductivity is produced by nonintentional boron incorporation.

The normalized spectrally resolved photoconductivity measured in the annealed state is shown by curve a in Fig. 4. This measurement was performed starting from low energies towards high energies. Under this situation, an onset of photoconductivity below 2 eV is observed. N-doped diamond or Ib diamond exhibit an ionization energy around 1.7 eV due to nitrogen. In Fig. 4, the photoresponse spectrum of a Ib diamond is also shown. It can be seen that both spectra are very similar, and therefore it is possible that the absorption at 2 eV in our case is produced by nitrogen impurities. While measuring the photoresponse of curve a, the sample is necessarily exposed to light with an energy as large as the gap of the diamond. A redistributing of carriers is then observed. Curve b in Fig. 4 shows the new photoresponse of the sample obtained in a second scan in the same energy range. The absorption edge is now below 1 eV. This spectrum is very close to that of a polycrystalline CVD diamond that also exhibits an absorption edge close to 1 eV (see Fig. 4). In polycrystalline CVD diamond this absorption was attributed to the effect of the grain boundaries.¹⁰ However, the sample was grown homoepitaxially onto a Ib monocrystalline diamond substrate, and in principle grain boundaries are not expected during the diamond growth. After annealing the sample all the carriers are trapped in the deep state at 2 eV



FIG. 4. Normalized spectrally resolved photocurrent measured at 300 K after a thermal annealing at 700 K. In curve (a), the first scan after the annealing (from low energies to higher ones), an optical ionization threshold at 2 eV is observed (compare to the spectrum of the Ib diamond). Curve (b) shows the photoresponse measured after curve (a). Band to band illumination produces a filling of centers with an ionization energy of 1 eV. These centers could be produced by the presence of grain boundaries (compare with the spectrum of the poly-CVD diamond).

(curve a). Band to band injection of electrons is produced when the sample is illuminated with an energy higher than the band gap. Then, electrons can be trapped in the level at 1 eV, and therefore an absorption at 1 eV is observed (curve b).

After illumination of the sample with strong UV light (state B), the photoconductivity spectrum is completely different, as shown in Fig. 5. The absorption edge is now below 0.4 eV. The photoresponse also shows clear maxima and minima that are due to oscillatory photoconductivity. The minima in the photoresponse are produced by a fast thermalization of photoexcited carriers into the valence or conduc-



FIG. 5. Normalized spectrally resolved photocurrent measured at 100 K after 10 min UV-illumination. The photocurrent minima are due to lifetime modulations associated with the shallow level at 371 meV that is introduced by nonintentional boron contamination (the spectrum of a IIb is also shown for clarity). A new acceptor state is observed at 479 meV.



FIG. 6. (a) PC measured at 70 K after UV illumination. Two photoionization energies are detected at 374 and 479 meV. (b) PC after anneling the sample to 180 K. (c) Annealing the sample at 210 K empties the shallower level that can not contribute to the PC. (d) After cooling down to 10 K the broad absorption between 400 and 450 meV disappears.

tion band and their capture into an excited state by emission of a cascade of *n* LO phonons. In the spectrum of Fig. 5, five cascades (a-e) corresponding to five different excited states can be observed. The resulting excited states are at 240, 290, 304, 348, and 363 meV below the ground level. From the analysis of the cascades a LO phonon energy of 155 (± 5) meV is obtained. An optical ionization threshold at 371 meV is clearly deduced from Fig. 5. The photoresponse in the energy range below 371 meV is due to photothermal ionization (PTI) of carriers. Two single maxima are observed at 348 and 363 meV, in agreement with the results of the oscillatory photoconductivity. The energies of the ground state as well as of the excited states prove that boron impurities are dominating the photocurrent response.¹¹ This result confirms that the acceptor level measured by Hall at 360 meV above the valence band is introduced by an unintentional boron doping (from SIMS experiments the boron concentration must be lower than 4×10^{16} cm⁻³). The optical-ionization thresholds at 1 and 2 eV have now vanished. Probably they are masked by the very high photoresponse of boron.

The spectrum in Fig. 5 is not really identical to the spectrum obtained in a boron-doped diamond and it is better understood if two distinct shallow levels are taken into account, one at 371 meV (due to the boron contamination) and a second level at 479 meV. For comparasion, the spectrum measured in a IIb diamond (with a boron concentration of 10^{17} cm⁻³) is also shown in Fig. 5. To verify the existence of two distinct levels it is necessary to study both levels isolated from each other. In order to study the deeper level (E_x) alone the sample was annealed at different temperatures after the UV illumination. Here we will assume that the deeper level is also an acceptor state, as it will be demonstrated. By choosing the right annealing temperature, the holes from the shallower level will be removed while the deeper level is still occupied. Therefore, only holes in the deeper state will contribute to the photocurrent. The result of this experiment is shown in Fig. 6, where the energy range from 0.3 to 0.6 eV



FIG. 7. Photocurrent measured after UV illumination and annealing at 220 K. The first scan (curve a) shows that the boron level at 370 meV has vanished. A new scan in the same conditions (curve b) shows that the boron level has been populated again by holes. That is a clear indication that the level at 479 meV is an acceptor state. A schematic of the filling of the boron level with holes from the E_X level is shown in the inset.

is shown in more detail. After UV illumination at 77 K for 10 min, the sample was annealed at different temperatures. All the measurements were done at 77 K in the dark. In Fig. 5, in addition to the spectra measured after annealing at 180 and 210 K, also the photoresponse measured without any annealing is included. As can be observed, after annealing at 180 K the photoresponse still exhibits the contribution from both levels, but the complete spectrum is lowered by a factor of 5. This reduction is due to the fact that the annealing produces a thermal emission of most carriers from both levels. On the other hand, after annealing at 210 K the photocurrent spectrum is dominated by excitations at energies $h\nu > 479$ meV. The photoresponse in the energy range below 479 meV is due to PTI of holes. A broad peak can be observed between 405 and 450 meV, as well as a single maximum at 459 meV. These absorption features probably represent optical transitions from the E_X ground state into excited states, from where the holes are then thermally emitted into the valence band. We have confirmed the excited-state nature of these absorption features by cooling down the sample to 10 K. At this temperature, the holes cannot be thermally transferred from the excited state to the valence band, and therefore PTI of holes is not allowed. As expected, the PTI peaks associated with the 479 meV level vanished at low temperatures (see curve d in Fig. 6).

In order to verify the acceptor nature of the 479 meV level we have performed the following experiment. The sample was annealed at 210 K for 20 min, and a first scan of the photoresponse was taken from low to high energies. The result is shown in Fig. 7 (curve a). As expected, the boron level at 370 meV vanished and as a first approximation it is not contributing to the photocurrent. However, oscillatory photocurrent is observed in the spectrum indicating that boron centers are contributing somehow to the photocurrent. A new scan (curve b in Fig. 7) shows that the boron level has been populated during the first measurement. This behavior can be



FIG. 8. Photoconductivity spectra and CPM spectra measured at 70 K under (a) illumination and (b) after illumination and annealing to 210 K. In case (a) the absorption is dominated by the boron level at 371 meV. Annealing the sample at 210 K empties the boron level, and therefore only the second level at 479 meV is observed. Dashed lines are theoretical fittings to the optical cross sections.

explained if the level at 479 meV is populated by holes. Once that those holes are excited to the valence band during the PC experiment, they can be trapped into the boron centers and from there contribute again to the photoresponse (see inset in Fig. 7). This result clearly proves that the new acceptor level at 479 meV is an acceptor state in diamond.

The study of the shallower boron level isolated from the deeper one requires a different approach. In the situation with two acceptor levels separated by 100 meV, a fast trapping from the shallower level into the deeper one could occur. This could explain the lower contribution of the boron level to the photoresponse. Under UV-illumination, a constant refilling of the boron level is provided, and the trapping effect should be reduced. In Fig. 8, the photoresponse of the sample measured under UV illumination is shown. It is clear that the effect of the UV biasing is to enhance the contribution of the shallower level compared to the deeper level.

The constant-photocurrent method (CPM) was applied to study the photoionization cross-section of holes in both levels. In this experiment the photocurrent is kept constant by adjusting the incident photon flux measured by a pyroelectric detector. In this way the quasi-Fermi level position and, therefore, the occupation of defects in the band gap remains constant. Then, the lifetime of photogenerated carriers is also constant and the absorption coefficient is given by¹²

$$\alpha_{\rm CPM}(h\nu) = \frac{\rm const}{\Phi(h\nu)},$$

where $\Phi(h\nu)$ is the photon flux necessary to keep the photocurrent constant. CPM measurements were performed under UV illumination and after annealing at 210 K. The results for those two sets of data are shown in Fig. 8, together with the photocurrent experiments described earlier. The agreement between both techniques is very good. Both CPM spectra were numerically fitted using theoretical formulas for the optical cross section developed by Inkson¹³ (dashed lines in



FIG. 9. Energy band diagram summarizing the position of the different states detected in the diamond sample. In the state A, the deep donor state (E_Y) pins the Fermi level at about 2 eV below the conduction band. Therefore, both acceptor levels at 371 meV (boron level) and 479 meV (E_X) are not populated by holes. The level at 1 eV (E_Z) , probably introduced by grain boundaries, is empty of electrons. This level is populated when electrons are excited from the valence band into the conduction band. Once in the conduction band electrons are trapped into the 1 eV state. Illuminating the sample with strong UV (state B) produces the population of the acceptor levels, and *p*-type conductivity is generated.

Fig. 8). The optical photoionization threshold energies deduced from the fit are 371 and 479 meV.

IV. DISCUSSION

Figure 9 summarizes the different states that have been detected in the diamond sample, and their energy position to the conduction and the valence band. The acceptor level at 371 meV has been attributed to a nonintentional doping by boron. However, the level at 479 meV above the valence band has never been reported in boron-doped samples to the best of our knowledge. The diamond sample analyzed in this work was grown under the presence of H₂S, and therefore some effect of S can be expected. An isolated substitutional sulfur (S_{sub}) is expected to be a donor in diamond. Based on ab initio calculations it was found that an isolated S_{sub} should introduce a deep donor level in diamond [1 (Ref. 9)]and 1.6 eV (Ref. 14)]. Other calculations predict a doubledonor behavior with levels at 0.15 and 0.5 eV.¹⁵ Moreover, sulfur complexes with other impurities (N or B) and with vacancies also introduces donor levels of 0.4-0.5 eV.9 Comparing these theoretical studies with our results, we did not find any donor level that can be clearly attributed to the sulfur. The deep donor level that we find at 1.55 eV could be introduced by N contamination (with a concentration below 3×10^{18} cm⁻³, that is the SIMS system sensibility) or it could also be the result of sulfur doping. The other deep level detected at 1 eV can be attributed to the effect of grain boundaries, although grain boundaries are not expected in homoepitaxial growths. The new level observed at 479 meV is an acceptor level, and therefore does not fit with any of the theoretical predictions. Our results clearly indicate that the doping of diamond with H₂S produces an acceptor level at 479 meV, instead of the expected donor level. New experiments such as very high resolution SIMS are planned in order to measure the real amount of boron and sulfur in our diamond. Probably those experiments, together with a theoretical analysis would provide some insights about the origin of the 479 meV acceptor level, and also would clarify the origin of the deep donor level at 1.55 eV and at 1 eV.

V. CONCLUSIONS

In summary, the electrical properties as well as optical absorption has been studied in a homoepitaxially grown CVD diamond to which H_2S was added during the growth. From spectrally resolved photoconductivity an acceptor level at 371 meV was detected, and it has been attributed to non-intentional boron contamination (SIMS experiments did not show boron above the system sensibility, 4×10^{16} cm⁻³). A new acceptor center at 479 meV is deduced from PC experiments, showing some excited states. The origin of this state is not yet well understood, although it can be produced by complex formation due to the presence of sulfur. To our knowledge, this new acceptor state has never been reported

in sulfur-free diamond samples. Hall experiments at low temperature showed *p*-type conductivity, with an activation energy of 360 meV, that it is explained by the boron contamination. At high temperatures, a *n*-type conductivity was measured with an activation energy of the electrons of 1.55 eV. The origin of this *n*-type doping is not clear yet, although it could be produced by nitrogen impurities (however, nitrogen was not detected by SIMS above the system sensibility for N, 3×10^{18} cm⁻³). More experiments are planned in order to clarify the origin of this deep donor level at 1.55 eV. Although in our study we did not succeed in finding a donor level clearly associated to sulfur, we are reporting the existence of an acceptor state that could be originated by the presence of sulfur, among other impurities (boron or nitrogen).

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