The Study of the Efficiency of Nitrogen to Nitrogen-Vacancy (NV)-Center Conversion in High-Nitrogen Content Samples

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1. Introduction

Color centers and other impurity-related defects are attracting a lot of attention in the past decades. In particular, the negatively charged nitrogen-vacancy color center in diamond (nitrogen-vacancy (NV)− center) is a subject of active research due to a large number of potential applications, including solid-state single photon sources,[1] qubits[2] and qubit registers,[3,4] quantum communication lines,[5,6] quantum teleportation,[7] and various sensing applications.[8] Sensing applications seem to be the closest ones to practical applications and at the moment cover high-resolution thermometry,[9] measurements of electric[10] and magnetic[11] fields (either locally[12] or with high sensitivity),[13] magnet resonance tomography,[14] sensitivity of strain,[15–17] rotation,[18] and acceleration.[19]

Utilizing a large number of color centers generally improves the sensitivity of a sensor. Nevertheless, the use of a large number of NV− can cause additional difficulties, such as diminished coherence properties. Additionally, as the lateral dimensions of the diamond samples increase, it becomes more challenging to uniformly excite and drive the centers.[20,21]

Thus, a lot of recent efforts were focused on growth optimization and post-growing processing of diamonds to maximize...
the number and efficiency of NV\textsuperscript{−} centers in various sensing applications.\textsuperscript{[15,22–25]}

There are a number of post-growth diamond processing techniques. The most popular techniques are implantation with He ions\textsuperscript{[26–28]} or protons\textsuperscript{[28]} or irradiation with electrons.\textsuperscript{[29–31]} The use of diamond plates with relatively high concentrations (\(\approx 100\) ppm) of nitrogen can be advantageous to plates with lower nitrogen content in some applications. These types of samples tend to have larger nitrogen to NV\textsuperscript{−} center conversion and smaller fraction of unwanted absorption compared to lower concentrations, and still have long enough coherence times for magnetometry.

In this article, we focus on the optimization of diamond post-growing electron irradiation followed by a simple annealing procedure, and try to optimize the yield of the NV\textsuperscript{−} center, as well as shot-noise limited sensitivity per unit volume of the crystal for static field (DC) magnetometry. In monocrystal diamonds, with about 100 ppm concentration of nitrogen, we observed a continuous increase in the conversion efficiency with electron dose irradiation, limited by the time available at the electron-irradiation machine.

2. Experimental Section

The NV\textsuperscript{−} center was a complex formed by a single substitutional nitrogen atom and an adjacent vacancy in the carbon diamond lattice. A nearby electron donor (presumably another nitrogen atom) provided the negative charge state of the center and also caused the NV\textsuperscript{−} center to have a triplet electronic configuration. Energy levels (Figure 1a) of the color center were located in a diamond bandgap and formed triplet grounds and excited states. Besides, there was an optically inactive singlet state, which is responsible for different fluorescence intensities for levels of excitation with different magnitudes of the magnetic quantum numbers. Thus, if components of the NV\textsuperscript{−} center ground state were mixed with microwave radiation, resonant to the \(|m_s = 0\rangle \leftrightarrow |m_s = +1\rangle\) transition (see Figure 1a), the fluorescence of the color center was lower than if the microwave radiation is absent or off-resonance. The resonance in the fluorescence spectrum with respect to microwave radiation frequency is typically called optically detected magnetic resonance and is the key feature enabling the use of the NV\textsuperscript{−} center.

The diamond samples (type Ib), enriched with nitrogen, were grown using a temperature-gradient, high temperature, high pressure (TG-HPHT) growth process\textsuperscript{[32,33]} at a pressure of 5 GPa and a temperature of 1500–1550 °C. Pure (99.9995%) graphite was used as a carbon source for growth. Alloy Fe–C was used as a solvent, while nitrogen was not artificially added. The residual air in the growing mix served as a rich enough source of nitrogen.

All samples were cut from the same diamond Ib crystal. Each crystal facet has its own surface energy and, during growth, different impurities have different capture rates for each facet. Thus the HPHT-TG process results in the sectorial structure of the crystal. Plates were cut from the growing sector \{111\} with crystallographic base surface orientation (100) and 1.5 × 1.5 × 0.290 mm samples were cut from the plates. Thus, all samples were cut from uniform \{111\} growth sectors and had the same sizes (with uncertainty less than 5%). All sample plates were measured to contain substitutional nitrogen (C centers, also known as P1 center) concentration of about \(1.5 \times 10^{19}\) cm\textsuperscript{−3}. The cutting was...

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Figure 1. a) NV\textsuperscript{−} -center simplified level scheme. b) Absorption spectra of diamond plates at 77 K in logarithmic scale. Offset is added to simplify viewing the plot. c) Infrared absorption spectra for the plate with the largest irradiation dose before and after irradiation. Lines correspond to the know centers with wave numbers 1135, 1332, 1450, 1502, 1975, 2027, 2159 cm\textsuperscript{−1}. d) Comparison of the charged and non-charged NV lines in logarithmic scale.
performed using a homemade laser cutter based on InnoLas Laser (Nanio 532-18-γ) and then mechanically polished so that the average absolute values of the profile height deviations \((R_d\text{ coefficient of roughness})\) became smaller than 5 nm. The concentration of C center was measured using an infrared absorption method and a Fourier spectrometer Bruker Vertex 80v, combined with an optical microscope HYPERION 2000 equipped with an MCT detector with high sensitivity and 15X objective, which is able to collect a signal from 50 μm spot. To measure the concentration of C center we used features (peaks) in the 1100–1400 cm\(^{-1}\) region of IR absorption spectrum. Davies summarized calibration data for IR analysis.\(^{[34]}\) To correctly estimate C centers concentration, absorption in the 1100–1400 cm\(^{-1}\) region was decomposed on features connected with A, B, and C centers absorption. Spectra for three different model diamonds with A, B, and C centers only were used. The experimental spectra were mathematically decomposed on these three components with corresponding weights (see Figure 2b). Then calibration coefficients from\(^{[34]}\) were used to calculate C centers concentration using the following formula

\[
N_C[\text{ppm}] = (25 ± 2.5) μ_{1135}
\]

where \(μ_{1135}\) – absorption coefficient for C centers. Taking into account strong IR absorption, it was estimated that errors in nitrogen content calculation were correlated only with the calibration itself.\(^{[35]}\) Absorption spectra were taken from five different points of the samples. The difference in nitrogen concentration was less than 5%, which is smaller than the error of measurements. In contrast to the often used chemical vapor deposition (CVD)-grown crystal, TG-HPHT monocrystalline diamond had practically no vacancies after growth. Thus, the implantation of the vacancies is an essential step for the formation of the NV\(^−\) centers. In this work, electron-irradiation was utilized using linear electron accelerators with energy up to 3 MeV (LLC Velman, Novosibirsk) and 3.5 MeV INDUSTRIAC 3E1000M (TISNCM). In both cases, the energy of the electrons was chosen to be 3 MeV, while doses of irradiation \(d\) were varied in the range of \(1 × 10^{17}–5 × 10^{18}\) cm\(^{-2}\). The maximum dose was limited by the time, available at the electron accelerator. The irradiation was followed up by vacuum annealing at a temperature of 700–800°C for 2 h. According to\(^{[36]}\) during electron irradiation energy of an electron of 3 MeV and dose of \(1 × 10^{18}\) cm\(^{-2}\) in diamonds of type Ib with a concentration of C centers below 12 ppm about 9 ± 1 ppm isolated vacancies are formed, from which fraction of negatively charged vacancies is rising from 6% at \(n_v = 0.5\) ppm to 67% at \(n_v = 12\) ppm. The number of vacancies formed typically rises simultaneously with a rise in nitrogen concentration. The rise in the vacancy formation range is associated with the formation of the nitrogen + interstitial carbon atom (or two atoms) complex, which has a characteristic absorption line at 523.6 nm. Thus, for samples with \(n_v = 85\) ppm for dose \(2 × 10^{18}\) cm\(^{-2}\) \(≈ 30\) ppm (25 ppm of V\(^−\) and 5 ppm of V\(^0\)) and for dose \(5 × 10^{18}\) cm\(^{-2}\) \(≈ 55\) ppm (47 ppm of V\(^−\) and 8 ppm of V\(^0\)) of vacancies were detected.\(^{[36]}\) Therefore, the speed of the vacancy formation could be estimated as \(≈5.3\) and 9.7 cm\(^{-1}\) for V\(^−\) and V\(^0\) correspondingly.

During irradiation, the diamond samples were maintained at room temperature with water cooling, thus the formation of NV centers during irradiation was strongly suppressed. Also, the range of electrons in the diamond at 3 MeV was around 4 nm,\(^{[36]}\) thus electrons pass through the plate without considerable loss of energy, and therefore the concentration of vacancies is expected to be uniform over the thickness of the sample. Figure 1c demonstrates the transformation of a nitrogen-related center before and after irradiation and annealing for diamond plate #4 with the highest dose of irradiation. After irradiation and annealing, 3 novel lines were observed in spectra which correspond to N\(^+\) defects (1332 cm\(^{-1}\)) and interstitial nitrogen (1450, 1502 cm\(^{-1}\)).\(^{[37]}\) Other lines at 1975, 2027, and 2159 cm\(^{-1}\) corresponded to the second-order lattice absorption of diamond and do not change. The reduction of 1332 cm\(^{-1}\) is related to the transformation of C centers into NV, N\(^+\), and interstitial nitrogen.\(^{[37]}\)

Concentrations of the NV\(^−\) centers formed after annealing were measured using the visible absorption spectrum (see Figure 1b), taken at 77 K using the method described in references.\(^{[32,33]}\) The concentration of NV centers was calculated using the following formula

\[
n_{\text{NV[ppm]}} = 0.332 I_{637}
\]

\[
I_{637} = \int μ(λ) dλ
\]

where \(n_{\text{NV[ppm]}}\) is the concentration of NV centers in ppm and \(I_{637}\) – the integral intensity of the NV zero-phonon line at

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**Figure 2.** a) Absorption spectra of diamond plates at 77 K recalculated into absorption coefficient. Offset is added to simplify viewing the plot. b) Portion of the infrared absorption spectra with fit of A, B, and C centers concentrations.
77 K in the absorption coefficient spectrum (see Figure 2a). The NV lines are the most pronounced in the visible spectra, while infrared spectra have a number of defects (see Figure 1c). The uniformity of the NV center was estimated by analysis of the fluorescent image, which has a fluctuation of intensity of about 8%, indicating quite good uniformity of the color centers distribution. The concentration of NV$^0$ is less than 0.5% of NV$^-$ (see Figure 1d).

To remove all types of surface contaminations, substrates were boiled in aqua regia and then washed in an ultrasonic bath in the surfactant solution, acetone, isopropyl alcohol, and deionized (DI) water. After washing, diamond substrates were blown dry with nitrogen and annealed in air for 20 min at 600 °C to burn out any residual non-diamond carbon.

Dephasing time $T_2^*$ of ensembles of NV$^-$ center was performed using a homemade confocal microscope at room temperature (similar to the one described in ref. [22]). A continuous-wave laser (Coherent Compass 300, wavelength of 532 nm) served as a source for the NV center excitation. Imaging was realized with the help of galvo mirrors (Cambridge Technologies). An Olympus 10X Plan Achromat objective lens with a numerical aperture of NA = 0.25 and a working distance of 10.6 mm was used to focus the excitation and collect the emissions from the diamond. The excitation power on the diamond sample was 8 mW. The collected fluorescence was coupled into an avalanche photodiode (PerkinElmer SPCM-AQRH-14-FC). A combination of an optical notch filter with a stopband centered at 532 nm and a longpass optical filter with a cut-off at 600 nm was used to remove the residual green excitation light and Raman signal from the collected emissions. Also, emissions were reduced by neutral density filters to achieve about 1–3 × 10$^6$ counts on the avalanche photodiodes. To manipulate the spin state of the NV$^-$ centers, a microwave field was supplied to the sample. The field was radiated by a Helmholtz resonance antenna, powered by an amplifier (Minicircuits ZHL-16W-43+), which was fed by a Stanford Research Systems SG384 generator.

The dephasing time $T_2^*$ was extracted from the dependence on the Rabi frequency of the optically detected magnetic resonance (ODMR) width (see Figure 3a). The ODMR spectra were recorded at different microwave powers and then fitted with a combination of three Lorentzian functions with fixed detuning between the function maximums of 2.2 MHz, corresponding to the well-known ODMR splitting line, due to the interaction of the NV$^-$ electron spin with nuclear spins of the nitrogen atom within the color center.[39] Rabi frequencies were measured using a standard technique; the centers were first initialized using green laser excitation, and then the microwave pulse of variable length was applied.[40] The microwave frequency was set to the central frequency of the ODMR line at 2795 MHz (see Figure 3a). The fluorescence of color centers was then measured in a 500 ns time window right after the microwave pulse. The resulting graph of fluorescence versus the length of the microwave pulse was then fitted with a product of exponential decay envelope and sin-shape. The fitted frequency of the sin-shape was then used as a Rabi frequency $F_R$.

The power dependence of the extracted full width at half maximum (FWHM) $\Delta \nu$ was then approximated by a straight line and the value of the width at 0 power $\Delta \nu$ was extracted (see Figure 3b). The dephasing time $T_2^*$ was then calculated\[41\] as

\[
T_2^* = \frac{1}{\pi \cdot \Delta \nu}
\]

### 3. Results and Discussions

A summary of the measurement results of the nitrogen concentration, concentration of NV$^-$ and the corresponding dephasing time for various irradiation doses, is presented in Table 1. The table also contains the conversion efficiency for the substitutional nitrogen into NV$^-$ centers, which was estimated using the following formula

\[
\gamma = \frac{n_{NV}}{n_C}
\]

where $n_{NV}$ in the NV$^-$ concentration after the annealing procedure and $n_C$ is substitutional nitrogen concentration before diamond irradiation.

Figure 4 illustrates a correlation between irradiation dose and conversion of substitutional nitrogen centers into the NV$^-$ centers. The figure suggests a linear dependence of the conversion efficiency with no saturation observed. We should stress that the observed conversion efficiency of 37% ± 4% exceeds many recently published results, including detailed studies and

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**Figure 3.** a) ODMR spectra of the ensemble of NV$^-$ centers for a diamond plate with a dose of $5 \times 10^{18}$ cm$^{-2}$ versus applied microwave power. The Rabi frequency of the microwave transition varied in the 0.015–2.8 MHz range. b) Dependence of the ODMR resonance peak width $\Delta \nu$ on the Rabi frequency of the microwave transition for all samples used.
optimizations of the annealing procedure, while the annealing procedure in this work is rather trivial and is only the first step of much more advanced ones[31.42-44] This suggests that the irradiation dose for the high-concentrated samples has a very large impact on the final conversion efficiency and must be kept as high as possible. We could expect that the application of a more optimal annealing procedure may further enhance the conversion efficiency.

Also, despite rising conversion efficiency with the irradiation dose, we observe a slight drop in the dephasing time. Increasing the irradiation dose is expected to increase the conversion of substitutional nitrogen into NV centers, as observed. The dephasing time on NV center with 100 ppm concentration is typically limited by the concentration of the substitutional nitrogen, and is thus expected to increase as the nitrogen concentration decreases. But, experimentally the decrease in the dephasing time indicated that the NV–NV interaction started to limit the dephasing time and further confirm a high conversion efficiency for NV centers.[45]

The concentration of the color centers and the efficiency of substitutional nitrogen to NV− centers are not the only parameters that affect the sensitivity of a sensor. The other important parameter is the dephasing time, which allows a shot-noise limited estimate of potential sensitivity for DC magnetometry per unit volume of diamond[11]

$$\eta_{\text{DC}} = \frac{1}{\frac{g_{e}\mu_{B}}{\hbar}C\sqrt{n_{\text{NV}}T_{2}}}$$

where C — the contrast of ODMR resonance, $g_{e}$ — g-factor of an electron, $\mu_{B}$ — Bohr magneton, $\hbar$ — reduced Plank constant. Calculated this way $\eta_{\text{DC}}$ are summarized in Table 1 and presented in Figure 4b. We note that this sensitivity is a rather theoretical limiting factor, than a practically achieved sensitivity for ensemble-based sensors. The latter will depend on the collection efficiency of the setup, laser power used, absorption, uniformity of the fields, and so on.[46-51] It could be clearly seen that the sensitivity improves with dose even with a relatively simple annealing procedure. More advanced annealing procedures have the potential to further improve dephasing time and sensitivity. Moreover, the increased conversion efficiency from substitutional nitrogen to NV− centers solves one more issue—it reduces the fraction of unwanted absorption of the diamond sample by C centers, thus improving sensitivity per laser power.[46]

The conversion efficiency of C centers to NV− seems to vary a lot in the literature. In the case of high-nitrogen concentration (around 100 ppm) about 16% has been achieved[43] with the 3 MeV electron irradiation technique. Similar results were recently achieved with He ion implantation.[44] Sophisticated annealing procedures have been used in later cases. In the case of lower nitrogen concentration (about 3 ppm) about 15% conversion efficiency to NV− with a similar amount of NV0 has been reported with electron irradiation by 3 MeV electrons[31,42] In this case, nevertheless, considerable saturation of conversion efficiency was observed. In the case of nanodiamonds with similar nitrogen concentration (about 5 ppm) conversion efficiency of 25% has been reported using high electron energy of 10 MeV, again, with noticeable saturation. In our case, 37% ± 3.7% conversion efficiency with no saturation and using only 3 MeV electron conversion efficiency was observed, considerably overcoming all known previous results.

While this research only was done for one specific concentration on substitutional nitrogen centers, it is likely, that a considerable increase in electron irradiation dose may help samples with different, even with a relatively low concentration of

Table 1. Summary of the measurements.

| # | $n_{c}$ [cm$^{-3}$] | $d$ [cm$^{-2}$] | $n_{\text{NV}}$ [cm$^{-3}$] | $\gamma$ [%] | $T_{2}$ [ns] | $\eta_{\text{DC}}$ T [kHz × mm$^{-1}$] $^{-1}$ |
|---|---|---|---|---|---|---|
| 1 | 1.5 ± 0.15 x 10$^{19}$ | 1 ± 0.03 x 10$^{17}$ | 3 ± 0.3 x 10$^{16}$ | 0.2 ± 0.02 | 231 ± 12 | 1.09 ± 0.12 x 10$^{-12}$ |
| 2 | 1.5 ± 0.15 x 10$^{19}$ | 4 ± 0.12 x 10$^{17}$ | 1.4 ± 0.15 x 10$^{17}$ | 0.93 ± 0.1 | 228 ± 10 | 5.2 ± 0.6 x 10$^{-15}$ |
| 3 | 1.5 ± 0.15 x 10$^{19}$ | 2 ± 0.06 x 10$^{18}$ | 2.3 ± 0.23 x 10$^{18}$ | 15.3 ± 1.5 | 204 ± 10 | 1.35 ± 0.15 x 10$^{-13}$ |
| 4 | 1.5 ± 0.15 x 10$^{19}$ | 5 ± 0.15 x 10$^{18}$ | 5.6 ± 0.56 x 10$^{18}$ | 37 ± 3.7 | 197 ± 9 | 9 ± 1 x 10$^{-14}$ |

Figure 4. a) Correlation between C centers to NV− centers conversion efficiency and irradiation dose. b) Shot-noise limited sensitivity of a DC magnetometer versus irradiation dose. The line represents an eye-guiding fit function $0.38 + 2.2x^{-1} – 0.11x^{-2}$. 
nitrogen. One can speculate that in the case of lower nitrogen concentrations the irradiation dose should be even larger, since lower nitrogen concentration reduces the probability of nitrogen-vacancy collision on an annealing step. Nevertheless, previously, some saturation of conversion efficiency was observed for samples with lower nitrogen concentration,\textsuperscript{1,2} as well as a considerable fraction of NV\textsuperscript{0} center, while in the current work, NV\textsuperscript{0} was barely detectable. Thus, further research is necessary to confirm this hypothesis. Also, the variety of results on the conversion of nitrogen to useful color centers implies that a deeper understanding of the physics of NV\textsuperscript{−} centers formation is necessary.

4. Summary

The influence of the electron irradiation dose on the conversion of substitutional nitrogen into NV\textsuperscript{−} centers was investigated on an example of diamond plates with about 100 ppm of nitrogen (1.5 × 10\textsuperscript{19} cm\textsuperscript{−3}). A strong linear correlation between the dose of high-energy electron (3 MeV) irradiation and conversion efficiency of substitutional nitrogen to NV\textsuperscript{−} centers was found. No saturation was detected in the range of irradiation doses of 10\textsuperscript{17} – 5 × 10\textsuperscript{18} cm\textsuperscript{−2}, and the conversion efficiency was likely restricted by the available time on the electron accelerator. The annealing step was not optimized, and a simple one-step 800 °C annealing was used. The maximum achieved conversion efficiency was as high as 37% ± 3.7%. The dephasing times in the NV\textsuperscript{−} centers were measured for the same irradiation doses. The decrease in the dephasing time with the increase in the dose was found to be insignificant. The projected shot-noise limited sensitivity per unit volume of DC magnetometer for the plates thus was improved with irradiation dose and the best possible sensitivity with diamonds used was estimated as 9 ± 1 × 10\textsuperscript{−14} T [√Hz · mm\textsuperscript{−3}] \textsuperscript{−1}. Considerably improved conversion efficiency, compared to the best previous results, and potential performance in the magnetometry show that the physics behind NV center formation is still not completely understood and shows great promise to further enhance the power of NV/diamond-based sensing applications.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

Keywords

diamond, NV\textsuperscript{−} center, optically detected magnetic resonance

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