Isotropic Scalar Quantum Sensing of Magnetic Fields for Industrial Application

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Magnetic field sensors based on quantum mechanic effects are often susceptible to misalignments of the magnetic field or need advanced procedures to compensate for these. Also, the record breaking sensitivities reported for superconducting quantum interference devices and alkali vapor magnetometers come along with large and complex experimental setups. The nitrogen vacancy center in diamond can be used to design a simple, small, and robust sensor without employing microwave radiation. By using compressed nanodiamond particles, it is possible to eliminate the need of an alignment of the magnetic field and still obtain the absolute magnetic flux density in a single measurement. In order to demonstrate the capabilities of this approach, a centimeter-sized modified automotive demo board is employed as a complete sensor with a sensitivity of 78 μT/√Hz.

1. Introduction

Current magnetometry research on the negatively charged nitrogen vacancy (NV) centers in diamond focus mostly on experiments, where the spins are manipulated by microwave radiation. Since a strong microwave field is required, the experiments, where the spins are manipulated by microwave radiation, have a need for magnetic sensors. Electronic vehicles, has a need for magnetic sensors. The most common resistive magnetic sensors are based on anisotropy (AMR), giant (GMR), and tunnel magneto resistance (TMR), which are all strongly dependent upon the angle of the magnetic field just like Hall sensors. All these sensors are current driven and based on semiconductor technologies. Typically, the bandwidth of Hall sensors is below 250 kHz, while the bandwidth of AMR, GMR, and TMR sensors can span from DC to several MHz. The sensitivity of commercial sensors of these types can reach values even below 100 pT/√Hz, but they suffer from temperature drifts and hysteresis effects. For many industry applications, e.g., current measurements, the direction of the field is not important and only the magnitude is of interest. Especially, the automotive industry, which is challenged with the rise of electronic vehicles, has a need for magnetic sensors. Electronic vehicles feature currents up to 450 A which need to be measured
Figure 1. Energy scheme of the simplified seven states model of the NV center in case of a zero and non-zero external magnetic field. The excitation and the fluorescence are pictured by green and red arrows, respectively. Non-radiative transitions between the triplet and singlet states are represented by dashed arrows. Additionally, the zero field splittings $D_{gs}$ and $D_{es}$ are defined for the ground and excited states, respectively. The transition rate from the state $|i\rangle$ to the state $|j\rangle$ is given by $k_{ij}$. The states and transition rates for the zero-field case are labeled with an additional zero. The pumping parameter $\beta$ is linear dependent on the pumping power $P$ used for the excitation of the system.

within an accuracy of 0.5 %,[32] to give a precise estimate of the state of charge of the battery system.[33] Also small ripples with bandwidths up to 10 kHz need to be monitored. [34] At charging, the battery system current for slow and fast charging can reach up to 15 and 600 A, respectively.[35] To challenge these requirements, we propose an isotropic scalar sensing technique based on NV centers in a pellet of nanodiamonds. By using a microwave free approach, we simplify the sensor design significantly and can create cost-efficient devices small in size. To cover a broad range of applications and provide a robust design, we utilize the fluorescence quenching caused by off-axis magnetic fields up to $\approx 50$ mT as sensing signal.

To understand the physical limitations of a microwave-free NV-based sensor, we first considered a single NV$^-$ center. The fundamental dependency of the fluorescence from the magnetic flux density vector $\mathbf{B}$ of single NV centers under different magnetic field angles was examined. This dependency was already presented in the literature while the dependence on the excitation power was neglected.[36] The magnetic field dependent ground and excited states of the NV center can be calculated by using a simple Hamiltonian neglecting the hyperfine interactions:

$$\mathcal{H}_{gs,es} = hD_{gs,es}S_z^2 + g\mu_B\mathbf{B} \cdot \mathbf{S} \quad (1)$$

Where $D_{gs,es}$ is the zero field splitting of the ground and excited states, respectively; $g$ is the gyromagnetic ratio; and $\mu_B$ is the Bohr magneton. Since the NV center has a spin of $S = 1$, two state triplets and several singlet states are present. For simplicity, the singlet states are represented by a single state, giving seven level system, as shown in Figure 1.

To simulate the fluorescence $\mathcal{R}$ of the NV center, the transition rates $k_{ij}$ between these seven states need to be calculated from the zero field transition rates $k^0_{ij}$. By summing over all light emitting transitions $|k\rangle \rightarrow |l\rangle$ at the steady state population of the states $n_k$ ($\frac{dn_k}{dt} = 0$), the fluorescence for a given magnetic field $\mathbf{B}$ and optical pumping power $P$ can be calculated.[36] The pumping power dependence of the fluorescence can be described by a saturation function

$$\mathcal{R}(B, \phi) = R_0(B, \phi) \frac{P}{P + P_{sat}(B, \phi)} \quad (2)$$

In this equation, $R_0(B, \phi)$ is the terminal count rate ($\mathcal{R}(P \to \infty)$) and $P_{sat}$ is the saturation power of the center. We define the magnetic contrast $C_{B,\phi}$ as the reduction of the fluorescence with respect to the magnetic field compared to the unperturbed system ($|\mathbf{B}| = 0$). For a magnetic flux density $B$ perfectly aligned with the NV axis ($\phi = 0^\circ$), the fluorescence is the same as for the unperturbed state of the system. Therefore, we can calculate the magnetic contrast also by changing the misalignment $\phi$ for a constant magnetic flux density $B$. Since there are always small perturbations present, near the level anti crossing points at $\approx 51$ mT and $\approx 102$ mT, the fluorescence will be reduced for the aligned case. So, the chosen magnetic flux density $B$ should be not too close to these values. This leads to the following definition of the
magnetic contrast:

\[
C_{B,\phi} = 1 - \frac{R(B, \phi)}{R(B = 0, \phi)} = 1 - \frac{R_{\phi}(B, \phi)}{R_{\phi}(B = 0, \phi)} \frac{P + P_{sat}(B, \phi = 0)}{P + P_{sat}(B, \phi = 0)}
\]  

(3)

When transferring the definition of the magnetic contrast to an ensemble of NV centers, we need to take into account that if one NV direction is aligned with the magnetic field, the three other possible NV orientations are not aligned. Therefore, the magnetic contrast is calculated by the summed fluorescence \(R(B, \phi)\) of all orientations and Equation (3).

2. Results

In the following, we present magnetic field dependent measurements of the fluorescence of a single NV center, an ensemble of NVs in a bulk diamond, and a pellet of nanodiamonds.

2.1. Single NV Center

The magnetic field was produced by a neodymium magnet mounted on a rotation stage. The magnet position and angle \(\phi = 0^\circ\) was chosen, so that the magnetic field was aligned with the NV center axis and had a strength of 70 mT. At \(\phi \neq 0^\circ\), the eigenstates of the aligned system begin to mix. In general, this leads to a reduced fluorescence due to a rise of non-radiative transitions to the ground state. To investigate the effect of the pumping power \(P\), we swept \(P\) from 0 to 2 mW for each angle \(\phi\), while measuring the fluorescence. This measurement was repeated 100 times for each \(\phi\) to average over small deviations. Each fluorescence saturation measurement was fitted by Equation (2) to obtain \(R_{\phi}(\phi)\) and \(P_{sat}(\phi)\). The magnetic contrast depending on \(P\) and \(\phi\) was then calculated using Equation (3). The results of the measurements are shown in Figure 2. To validate our findings, we simulated the fluorescence behavior under the same circumstances as in our measurements, applying the methods from ref. [36] and using the simple Hamiltonian from Equation (1). Figure 2b shows the results of the simulation.

2.2. Bulk Diamond

The sensitivity of an NV-based sensor increases with \(\sqrt{N}\), where \(N\) is the number of single NV centers. Therefore, a high-pressure, high-temperature (HPHT) single crystal diamond with a high density of NV centers was used to investigate the fluorescence behavior of an NV ensemble in bulk diamond under an external magnetic field. A home-build iron yoke with a copper coil was used to produce a magnetic field density \(B\) between 3 and 133 mT. The fluorescence over the full magnetic field range for different laser powers was measured. This measurement was performed for an aligned case, where the magnetic field was pointing along one NV direction, and for an about 20° (to the aligned case) misaligned case. In Figure 3b, the aligned case and in Figure 3d the misaligned case are shown. The magnetic contrast was then calculated using Equation (3) at \(B = 130\) mT. This contrast is plotted for both cases as inset in the graphs. Additionally, for the aligned case, the dip of the ground state level anti-crossing at \(B \approx 102\) mT was fitted with a Lorentzian function biased with a linear slope. The laser power dependency of the depth of the dip with respect to the fluorescence value at \(B = 0\) mT and the full width at half maximum (FWHM) of the dip are shown in Figure 3c.

2.3. Nanocrystalline Pellet

A pellet pressed from 25 nm diamond particles was used to perform pumping power and magnetic field dependent measurements on diamond powder. The experiment was designed similar to the bulk measurements, but due to the high attenuation caused by nanoparticle boundaries, a higher laser power was used. In Figure 4a, the magnetic field scans for different pumping powers for an arbitrary magnetic field orientation are shown. In the inset, the magnetic contrast defined in Equation (3) is plotted. Since the nanoparticles have no preferential orientation, NV centers are equally present in all directions. To prove this, the magnetic field was rotated over a range of 30°. In Figure 4b, the magnetic field dependency and, as inset, the magnetic contrast of different angles for a constant laser power of 50 mW are shown.
3. Discussion

The measurement of the fluorescence of a single NV center as a function of pumping power and magnetic field strength is qualitatively in good agreement with the simulation as shown in Figure 2. The lower measured magnetic contrast compared to the simulation can be associated with different mechanisms. One is the background fluorescence in our measurements, which was taken into account as an additional linear part in the fluorescence saturation fit using Equation (2). Since the background consists also partly of NV centers, the linear approximation will not eliminate all background signal which can lead to a reduced magnetic contrast. The second origin for the difference in the magnetic contrast is the transition rate of the NV center used in the simulation. The standard deviation of the inter-system crossing transition rates to the ground states ($k_j$) is especially high ($\approx 60\%$).[36] This can result in transition rates different to those present in the sample, which can be one of the reasons for the reduced magnetic contrast.

The measurements of the bulk diamond show the same saturation behavior of the magnetic contrast as single centers, but with a smaller absolute contrast. This can be mostly attributed to the fact that the measurements of the single NVs were background corrected since a confocal microscope was used. In the case of the bulk measurements, this was not possible and a large portion of NVs not in the focal point of the microscope contributed to the signal. The magnetic field is only aligned in the focal point of the microscope, due to the design-related field inhomogeneities of the yoke. The further an NV center is in the background, the more the magnetic field is misaligned. This leads to an increase of the overall fluorescence signal of the bulk diamond with increasing magnetic field strength above 50 mT, as it can be seen in Figure 3b and d. It is to mention that an increased quantum yield can cause a rise of the fluorescence for larger fields,[12] but
The nanodiamond pellet can be viewed as a cluster of numerous nanodiamond crystals, where the fluorescence signal will be an average over all possible angles of NV centers due to the random orientation of each nanodiamond. This results in the same fluorescence signal for an arbitrary magnetic field direction as long as the magnetic field strength is the same. Since most of the NV centers will not be aligned, the averaged signal has no features, similar to the misaligned case of the bulk sample as it can be seen in Figure 4a. The reflectivity of the pellet is much higher than that of the bulk sample, because on each nanodiamond surface light is reflected. This leads to a much smaller penetration depth of the excitation light compared to the bulk diamond. The flat signal for magnetic fields over 100 mT is a proof for the small detection volume, since for larger volumes the fluorescence would rise due to inhomogeneities of the field as seen for the bulk sample. Also, the achieved magnetic contrast of 12.5% is much higher as for the bulk sample. In addition, the magnetic contrast (inset Figure 4a) is not saturated for powers ten times higher than those of the bulk measurement. These two behaviors support the assumption that the penetration depth is lower and the reflectance is higher as for the bulk sample. To further compare the signal of the bulk and nanodiamond material, we measured the fluorescence signal with a confocal microscope in different depths with respect to the sample surface at constant excitation power. By integrating the fluorescence signal over the depth, we calculated an approximately 240 times stronger signal from the bulk sample compared to the nanodiamond pellet. To verify the isotropic sensing ability of the diamond pellet, an angle dependent measurement at a laser power of 50 mW was performed, as it can be seen in Figure 4b. The small decrease of the magnetic contrast of about 0.15 % over a \( \Delta \phi = 30^\circ \) can be attributed to the reduction of the pumping power due to the geometry of the magnet. As it can be seen in Figure 3a, the left arm of the yoke will be blocking the laser path with increasing \( \phi \), which leads to a reduced magnetic contrast.

In summary, the information of the magnetic field direction is therefore lost but isotropic sensing of the absolute magnetic field strength is achieved. The magnetic bandwidth spans between 3 and \( \approx 50 \) mT. For fields outside this range, the sensitivity is strongly decreased. In the stated bandwidth, the best sensitivities achieved in this study are 14 \( \mu \)T/\( \sqrt{\text{Hz}} \) and 28 \( \mu \)T/\( \sqrt{\text{Hz}} \) for the bulk sample and the nanodiamond pellet, respectively. Using the found insights, we designed a demonstrator based on the automotive industry certified E909.21 Elmos IC (AEC-Q100 certificated\(^6\)). Utilizing bulk and nanodiamonds, we achieved sensitivities of 32 \( \mu \)T/\( \sqrt{\text{Hz}} \) and 78 \( \mu \)T/\( \sqrt{\text{Hz}} \), respectively. The sensitivities of our measurements were calculated by integrating the spectral power density of the noise over the full bandwidth and dividing by the full bandwidth before taking the square root. The obtained sensitivity is equal to the standard deviation divided by the square root of the bandwidth, assuming that the noise floor of all measurements consists mostly of white noise. For the demonstrator, we modified a E909.21 evaluation kit by integrating a 520 nm laser diode with the sensing material (bulk diamond or nanodiamonds) in a small 3D printed housing in the design, shown in Figure 5. The difference of the sensitivities...
compared to our lab measurements can be attributed to the difference in collection efficiency of the fluorescence and excitation power density. We used no collection lens in the 3D printed housing, resulting in a smaller fluorescence collection. Also the focus lens of the laser could not be adjusted to reach the maximum magnetic contrast. The internal sampling frequency of the demonstrator is 125 kHz, while for one measurement, the signal of 63 cycles is used, resulting in a 2 kHz sampling. Due to internal waiting times, it is only possible to read out the demonstrator with 53 Hz. In a future design, it will be possible to use a 1 MHz internal sampling frequency and eliminate the waiting times. This would result in a bandwidth of up to 16 kHz, satisfying the needs of the automotive industry.\cite{34} It was already shown that all these measurements can be performed with the sensing material on an optical fiber.\cite{8,41,42} With this, the sensor can perform even in environments where the use of electronics is difficult. Additionally, the magnetic field dependency of sensors based on the spin mixing of NV centers like ours shows only small changes with temperature above 100 K,\cite{9} making them a good choice for harsh environments.

4. Experimental Section

Simulations: All simulations are based on a seven-level energy level structure following ref. \cite{36} and are shown in Figure 1. For the transition rates $k_{ij}$ from state $|i\rangle$ to state $|j\rangle$, the average values measured in ref. \cite{36} were used

$$
k = \begin{pmatrix}
0 & 0 & 0 & \beta k_r & 0 & 0 & 0 \\
0 & 0 & 0 & 0 & \beta k_r & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & \beta k_r & 0 \\
k_r & 0 & 0 & 0 & 0 & 0 & k_{37} \\
0 & k_r & 0 & 0 & 0 & 0 & k_{37} \\
0 & 0 & k_r & 0 & 0 & 0 & k_{37} \\
k_{71} & k_{72} & k_{73} & k_{74} & 0 & 0 & 0
\end{pmatrix} \tag{4}
$$

with $k_r = 65.925$ MHz, $k_{37} = 0.975$ MHz, $k_{72} = 0.725$ MHz, $k_{73} = 7.925$ MHz, $k_{32} = 53.325$ MHz, and $\beta$ the pumping rate, which is

proportional to the laser power. The states $|1\rangle$, $|2\rangle$, and $|3\rangle$ are the ground states with $m_s = 0, -1, +1$, respectively. The excited states are $|4\rangle$, $|5\rangle$, and $|6\rangle$ with $m_s = 0, -1, +1$, respectively. The state $|7\rangle$ represents all singlet states. The proportionality factor was calculated by fitting the power-dependent simulation to the measured fluorescence at $B = 0$ mT. All simulations were done for the time-independent steady state.

Sample Preparation: The single NV center was created by annealing a (100) chemical vapor deposition (CVD) diamond (element6, electronic grade) at 1200 °C. The bulk measurements were performed on a (100) HPHT diamond (element6, type Ib), which was treated as described in ref. \cite{43}. The nanodiamond pellet was prepared as follows: aqua regia cleaned MSY 0.0–0.05 nanodiamonds (ND) (Microdiamond AG, Switzerland) with a nitrogen content of around 100 ppm were cleaned in an initial step using air oxidation at 590 °C (1 atm) for 17 h to produce graphite-free oxidized NDs (ONDs). The diameter of the ONDs was below 30 nm according to dynamic light scattering investigations and scanning electron microscopy. 5 mg of the OND sample were pressed using a 3 mm diameter press cell container and a conventional KBr pellet stamp (PerkinElmer) applying a pressure in the kN range for 1 min. Afterward, the pellet was electron-irradiated (10 MeV, fluence $2 \times 10^{18}$ cm$^{-2}$). After the irradiation, the sample was post-treated by annealing in an argon stream (50 mL min$^{-1}$) for 5 h at 800 °C followed by a second air etching step at 620 °C. Further information about the characterization of the NDs and ND pellet can be found in ref. \cite{44}.

Experimental Setup: All measurements of the single NV center were done with a home-build confocal microscope equipped with a permanent neodymium magnet mounted on a x-y-z-ϕ stage. For the excitation and power dependency measurements a 532 nm laser (LaserQuantum gem532) was used. The fluorescence was collected through a ×60 1.35 NA oil objective and detected with two avalanche photo diodes.

The measurements of the bulk sample and the nanodiamond pellet were done with a home-build microscope, where the sample was mounted between a C-shaped iron yoke. Opposite of the opening the iron was wrapped with an enameled copper wire ($d = 1$ mm). The two yoke ends near the sample were fabricated conical to maximize the magnetic flux density at the sample. Magnetic flux densities between 3 and 130 mT could be obtained by currents up to 10 A. Due to the remanent field of the iron yoke, it is not possible to achieve magnetic flux densities below 3 mT. The iron yoke with the coil was mounted on a x-y-z-stage. On top of this positioning stage, a goniometer stage connected to a rotation stage was used. The rotation axes of all stages were running through the same point in the middle of the opening of the iron yoke, to ensure that only the...
direction of the field is changed. The maximum deviation of the magnetic field is about 10% in a 1 mm distance range from the center of the yoke opening.

The excitation was produced by a 520 nm pulsed laser diode (Osram PL520B) mounted on a self-made printed circuit board (PCB) driven by a fast laser switch (iC-Haus iC-HKB). The laser diode was collimated by a aspheric lens (f = 2.75 mm). A dichroic mirror (cut-on at 600 nm) was used to separate the excitation from the fluorescence. A plano-convex lens (f = 30 mm) focused the laser and collected the fluorescence of the sample. The position of the lens was optimized for maximum magnetic contrast. This was done separately for the bulk and nanodiamond pellet. Passing the dichroic mirror, the fluorescence was filtered by a 600 nm longpass and focused on a photo diode (Hamamatsu S5971). The photo diode was mounted on a self-made PCB together with a simple transimpedance amplifier (≈ 5 x 10^6 VA^-1). To measure the voltage provided by the transimpedance amplifier, a lock-in amplifier (Zurich Instruments MFLI) was used. The laser and photo diode were supplied by a shielded power supply.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

industry sensor, isotropic sensing, magnetometry, nanodiamond pellet, nitrogen vacancy center, quantum sensor

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