Zero-field magnetometry based on nitrogen-vacancy ensembles in diamond

Huijie Zheng,† Jingyan Xu,2 Geoffrey Z. Iwata,1 Till Lenz,1 Julia Michl,3 Boris Yavkin,3 Kazuo Nakamura,4 Hitoshi Sumiya,5 Takeshi Ohshima,6 Junichi Isoya,7 Jörg Wrachtrup,3 Arne Wickenbrock,1 and Dmitry Budker1,8,9,10

1 Johannes Gutenberg-Universität Mainz, 55128 Mainz, Germany
2 Chinese Academy of Sciences, Key Lab of Quantum Information, University of Science and Technology of China, Hefei 230026, People’s Republic of China
3 Institute of Physics, University of Stuttgart and Institute for Quantum Science and Technology IQST, 70174 Stuttgart, Germany
4 Application Technology Research Institute, Tokyo Gas Company, Ltd., Yokohama, 230-0045 Japan
5 Advanced Materials Laboratory, Sumitomo Electric Industries, Ltd., Hami, 664-0016 Japan
6 Takasaki Advanced Radiation Research Institute, National Institutes for Quantum and Radiological Science and Technology, Takasaki, 370-1292, Japan
7 Faculty of Pure and Applied Sciences, University of Tsukuba, Tsukuba, 305-8573 Japan
8 Helmholtz Institut Mainz, 55099 Mainz, Germany
9 Department of Physics, University of California, Berkeley, CA 94720-7300, USA
10 Nuclear Science Division, Lawrence Berkeley National Laboratory, Berkeley, CA 94720, USA

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Ensembles of nitrogen-vacancy (NV) centers in diamonds are widely utilized for magnetometry, magnetic-field imaging and magnetic-resonance detection. At zero ambient field, Zeeman sublevels in the NV centers lose first-order sensitivity to magnetic fields as they are mixed due to crystal strain or electric fields. In this work, we realize a zero-field (ZF) magnetometer using polarization-selective microwave excitation in a 13C-depleted crystal sample. We employ circularly polarized microwaves to address specific transitions in the optically detected magnetic resonance and perform magnetometry with a noise floor of 250 pT/√Hz. This technique opens the door to practical applications of NV sensors for ZF magnetic sensing, such as ZF nuclear magnetic resonance, and investigation of magnetic fields in biological systems.

I. INTRODUCTION

Negatively charged nitrogen-vacancy (NV) centers in diamond have garnered wide interest as magnetometers, with diverse applications ranging from electron spin resonance (ESR) and biophysics to materials science. However, typical operation of an NV magnetometer requires an applied bias magnetic field to non-ambiguously resolve magnetically sensitive features in the level structure. Such a bias field can be undesirable for applications where it could perturb the system to be measured, or, for example, create challenging crosstalk within sensor arrays.

Eliminating the need for a bias field would extend the dynamic range of NV magnetometers to zero field. Zero-field, NV-based magnetometry opens new application avenues, and makes these versatile, solid-state sensors competitive with other magnetic field sensors such as superconducting quantum interference devices (SQUIDs) and alkali-vapor magnetometers, because, despite the lower sensitivity of NVs, they offer additional benefits in their small size, high spatial resolution, capability of operation in large temperature and pressure ranges, and wide bandwidth.

The relative simplicity of NVs operated at zero field can readily complement existing sensors in applications such as zero- and ultra-low-field nuclear magnetic resonance (ZULF-NMR), tracking field fluctuations in experimental searches for electric dipole moments, and magnetoencephalography or magnetoencephalography.

Magnetically sensitive microwave transitions within NV centers can be probed using the optically detected magnetic resonance (ODMR) technique, which relies on detecting changes in photoluminescence (PL) while applying microwave fields to optically pumped NVs. At zero field, these transitions overlap, and shift equally with opposite sign in response to magnetic fields. Therefore, NV ensembles have been considered unusable as zero-field magnetometers, except in certain cases, for detecting AC fields in the presence of applied microwaves.

We overcome these complications at zero field by selectively driving resolved hyperfine transitions in NV centers in a 13C-depleted diamond with frequency-modulated, circularly polarized microwaves. This results in ODMR fluorescence with a linear response to small magnetic fields. We present such a zero-field NV magnetometer with a demonstrated noise floor of 250 pT/√Hz.

II. EXPERIMENT

A schematic of the experimental apparatus is shown in Fig. 1(a). Intensity stabilized green excitation light at 532 nm is used to optically pump the NV centers in diamond into a single spin projection (ms = 0) in the ground state. Microwaves (MWs) drive transitions from this ground state into the magnetically sensitive
spin states \((s=\pm 1)\), reducing the fluorescence resulting from the pumping cycle \[24\]. The diamond is glued to a parabolic light concentrator to collect fluorescence, which is focused through a filter and onto a photodetector (PD). The parabolic-concentrator arrangement was demonstrated to have over 60% collection efficiency in Ref. [1]. The signal from the PD is fed into a lock-in amplifier (LIA), which is referenced to the frequency modulation of the MWs. Three pairs of Helmholtz coils are wound onto a 3-D printed mount around the diamond, and their roll overlap and merge into four distinct features.

The NV spin Hamiltonian that describes the energy spectrum, and includes interaction with an applied magnetic field \(B\) and hyperfine interaction with the intrinsic \(^{14}\)N, is written as

\[
\mathcal{H} = D S_z^2 + E (S_x^2 - S_y^2) + g_e \mu_B B \cdot S + S \cdot A \cdot I - g_n \mu_N B \cdot I + Q I_z^2,
\]

where \(D = 2870 \text{ MHz}\) is the zero-field-splitting parameter, \(E\) is the off-diagonal strain- or electric-field-splitting tensor \[25\], \(S\) and \(I\) are the electron and nuclear-spin operators, \(g_e\) and \(g_n\) are the electron and nuclear-spin \(g\)-factors, \(\mu_B\) and \(\mu_N\) are the Bohr and nuclear magneton, and \(Q\) is the nuclear quadrupole-splitting parameter. The electron spin \(S\) and nuclear spin \(I\) are coupled via the diagonal hyperfine tensor,

\[
A = \begin{pmatrix} A_\perp & 0 & 0 \\ 0 & A_\perp & 0 \\ 0 & 0 & A_\parallel \end{pmatrix},
\]

where \(A_\perp = -2.7 \text{ MHz}\), and \(A_\parallel = -2.16 \text{ MHz}\) \[26\].

At low fields, the Hamiltonian is dominated by the 2870 MHz zero-field splitting, \(D\), between the \(|s, m_s = 0)\) and the degenerate \(|s, m_s = \pm 1)\) states. The hyperfine interaction with the nuclear spin of the NVs’ intrinsic \(^{14}\)N results in three hyperfine projections for each electron spin state, and so MW transitions between the \(m_s\) states are three-fold split [Fig.1 (b)]. These groupings of \(|m_s = \pm 1)\) states separate in energy with increasing magnetic field due to the Zeeman effect. At fields where the Zeeman shift results in degenerate hyperfine levels of the \(|m_s = \pm 1)\) states, anti-crossings between hyperfine states with the same nuclear spin projection occur due to the tensor \(E\).

These anti-crossings between hyperfine states are apparent in the ODMR Zeeman spectra of Fig.2(a). The anti-crossings for the transition energies at \(\approx 0.08 \text{ mT}\) correspond to interaction between the states \(|s, m_s = -1, m_I = \pm 1)\) and \(|s, m_s = +1, m_I = \pm 1)\). The bottom spectrum in Fig.2(b) shows strain- and electric-field splitting of the transitions \(|s, m_s = 0, m_I = 0) \rightarrow |s, m_s = \pm 1, m_I = 0)\) due to the interaction \(E\) between the upper states, while transitions \(|s, m_s = 0, m_I = +1) \rightarrow |s, m_s = \pm 1, m_I = +1)\) and \(|s, m_s = 0, m_I = -1) \rightarrow |s, m_s = \mp 1, m_I = -1)\) merge in the ODMR spectrum. In many diamond samples these features are indiscernible, because the transverse zero-field splitting \(E\) is larger than the intrinsic hyperfine splitting of the NV center. The well-resolved hyperfine structure in this diamond sample allows us to selectively address only the overlapping transitions that occur at \(\pm 2 \text{ MHz}\) from the central feature.

Circularly polarized MWs drive a single electron-spin transition and remove the symmetric dependence of the feature composed of overlapping resonances with small magnetic field. The circularly polarized MWs are created using a printed circuit board (PCB) that follows the design of Ref. [27]. The board consists of two, 200 \(\mu\)m wires.
At fields where the transitions to states are labeled \( |m_s, m_f \rangle \) and overlaid with the simulation according to Hamiltonian of Eq. 1. Continuous-wave ODMR spectra for selected values of \( B_z \). At zero-field only the central transitions are split. The lower energy peak at \( \approx 2868 \text{ MHz} \) corresponds to the transitions \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = +1, m_f = +1 \rangle \) and \( |m_s = 0, m_f = -1 \rangle \rightarrow |m_s = -1, m_f = -1 \rangle \). The higher energy peak at \( \approx 2872 \text{ MHz} \) corresponds to the transitions \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = +1, m_f = -1 \rangle \) and \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = -1, m_f = +1 \rangle \).

FIG. 2. (a) ODMR spectra with linearly polarized MWs as a function of axial magnetic field, with transitions originating from all crystal-axis orientations. Those transitions corresponding to NVs oriented along the direction of the applied field are labeled \( |m_s, m_f \rangle \) and overlaid with the simulation according to Hamiltonian of Eq. 1. Continuous-wave ODMR spectra for selected values of \( B_z \). At zero-field only the central transitions are split. The lower energy peak at \( \approx 2868 \text{ MHz} \) corresponds to the transitions \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = +1, m_f = +1 \rangle \) and \( |m_s = 0, m_f = -1 \rangle \rightarrow |m_s = -1, m_f = -1 \rangle \). The higher energy peak at \( \approx 2872 \text{ MHz} \) corresponds to the transitions \( |m_s = 0, m_f = -1 \rangle \rightarrow |m_s = +1, m_f = -1 \rangle \) and \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = -1, m_f = +1 \rangle \).

In typical ODMR magnetometry, linearly polarized MW fields drive transitions between the \( |m_s = 0 \rangle \) and \( |m_s = \pm 1 \rangle \) states, decreasing the detected fluorescence with respect to MW frequency. At fields where the transitions to \( |m_s = \pm 1 \rangle \) states are well resolved, modulation is applied to the MW frequency, and the resulting PL signal is detected on a PD and demodulated on a LIA at the modulation frequency. The first-order harmonic output exhibits a linear response of PL to magnetic field. However, at zero-field, the \( |m_s = \pm 1 \rangle \) states are degenerate, and transitions to these states, including those from all crystal orientations, overlap, causing the LIA output to no longer exhibit a linear dependence with magnetic field, as described below.

In our experimental setup, we measure a field that is applied along the \( (111) \) direction. This field is equally projected onto the other crystal orientations, but a single-axis description is sufficient to understand how magnetic field sensitivity arises.

For a background PL of \( P_0 \), the detected signal from applying MWs at frequency \( \omega \) to a given transition can be modeled as,

\[
P = P_0 - \frac{A \left( \frac{\Gamma}{2} \right)^2}{(\omega - \omega_0)^2 + \left( \frac{\Gamma}{2} \right)^2},
\]

where \( P \) is the PL signal, and \( \omega_0, \Gamma, \) and \( A \) are the center frequency, the linewidth and the amplitude of this Lorentzian profile.

At zero field, the PL signal is convolved with features due to strain and electric field, however, we can still approximate the signal with the Lorentzian form in Eq. 3. Focusing on the lowest-frequency peak of those shown in the zero-field trace of Fig. 2(b), two transitions, \( |m_s = 0, m_f = +1 \rangle \rightarrow |m_s = +1, m_f = +1 \rangle \) with amplitude \( A_+ \) and central frequency \( \omega_{0+} \), and \( |m_s = 0, m_f = -1 \rangle \rightarrow |m_s = -1, m_f = -1 \rangle \) with amplitude \( A_- \) and central frequency \( \omega_{0-} \), overlap such that \( \omega_{0+} = \omega_{0-} = \omega_0 \).
FIG. 3. (a) ODMR traces at fixed field with circular MW (left $\sigma^-$ and right $\sigma^+$). Here the peaks A and D (B and C) correspond to the transitions from $|m_s = 0\rangle$ to $|m_s = -1\rangle$ and $|m_s = 0\rangle$ to $|m_s = 1\rangle$ of on-axis (off-axis) NVs, respectively. (b) Fitted amplitudes of A and D in (a), as a function of the relative phase between two applied microwave fields. Blue crosses (amber circles) indicate amplitudes of A (D). Error bars on the data (1 standard error of the mean) are smaller than the symbol size. (c) ODMR spectra under linear, $\sigma^-$, $\sigma^+$ MW as a function of magnetic field. The color scale indicates the peak depth, in percent, relative to the off-resonant case.

Increasing the field $B_z$ along a single diamond axis, the central frequencies change by an amount,

$$\omega_{0\pm} = \omega_0 \pm g_e \mu_B B_z. \tag{4}$$

The effect of a small modulation of the MW frequency is described by the first-order expansion of $P$ for each transition around $\omega_0$ with respect to $\omega$. The resulting signal has linear dependence on magnetic field, and is a sum of the contributions from each transition. Using the relation in Eq. (4) we find that for small values of $B_z$,

$$\frac{\Delta P}{\Delta \omega} = (A_+ - A_-) KB_z + c, \tag{5}$$

where we have grouped various terms into the parameters $K = 8g_e \mu_B/\Gamma^2$ and $c = -8(\omega - \omega_0)(A_+ + A_-)/\Gamma^2$. When linearly polarized microwaves are applied, the transition probabilities for each $m_s$ state, and therefore the values of $A_{\pm}$, are equal. As a result, the change in PL is zero for small changes in magnetic field. However, circularly polarized microwaves can instead be applied, resulting in different transition probabilities so that the first term in Eq. (5) does not cancel out, resulting in magnetically sensitive changes in the PL. Therefore, to perform high-sensitivity magnetometry, we applied circularly polarized MWs and modulated the central frequency. Note here that the shape of the magnetometry signal is sensitive to the imperfection of circular MW polarization, which has varying effects for other crystal orientations, and to small detunings of the central MW frequency, which may arise from drifts in the diamond temperature. These effects can explain the asymmetry in Fig. 4 (a).

Contributions of the NVs oriented along other axes are also overlapping in the lineshape but they have a weaker dependence on the magnetic field (applied at an angle to these axes) and are suppressed when circular polarized MW are applied.

**IV. ALTERNATIVE MAGNETOMETRY METHOD**

As an aside, we mention that it is also possible to apply an oscillating magnetic field along the ⟨111⟩ direction in order to perform magnetometry with linearly polarized MWs. This oscillating field modulates the central frequencies of the two transitions as,

$$\omega_{0\pm} = \omega_0 \pm [g_e \mu_B B_z \cos(\nu t) + \omega_{mod}], \tag{6}$$

where $\omega_{mod} = g_e \mu_B \eta \sin(\nu t)$ and $\eta$ and $\nu$ are the amplitude and frequency of the oscillating field, respectively. The resulting PL is the sum of contributions from the relevant transitions, each described by the expansion of Eq. (3) in $\omega_{mod}$ around $\omega_{mod} = 0$. The magnetic dependence for small values of $B_z$, and $\eta \ll \Gamma/g_e \mu_B$ is,

$$\frac{\Delta P}{\Delta \omega_{mod}} = (A_+ + A_-) KB_z + c', \tag{7}$$
FIG. 4. (a) Detail of the LIA output around zero field (black line) with a dispersive curve fitting and a linear fit (red) to the data while modulating the frequency of the applied circularly polarized microwave at 3 kHz with a depth of 45 kHz. (b) Magnetic-field noise spectrum. Blue line indicates magnetically sensitive noise, amber line indicates magnetically insensitive at a MW frequency of \( \approx 2900 \text{ MHz} \) (average noise between 1 and 1000 Hz is 250 pT/\( \sqrt{\text{Hz}} \)), and electronic noise (average noise between 1 and 1000 Hz is 70 pT/\( \sqrt{\text{Hz}} \)). The photon shot noise limit of the magnetometer is indicated at 4 pT/\( \sqrt{\text{Hz}} \). The decrease in signal for frequencies above 1 kHz is due to the filtering of the LIA.

where \( K \) is as defined in Eq. \( 5 \) and \( c' = 8(\omega - \omega_0)(A_- - A_+)/\Gamma^2 \). The signal is detected on a LIA referenced to the field modulation frequency, \( \nu \). Since \( A_{\pm} \) do not cancel out in Eq. \( 7 \), there is linear magnetic sensitivity in the LIA output signal, which, for \( A_- = A_+ \), is insensitive to the detuning of the central MW frequency within the linear regime. The magnetic dependence, \( \Delta P/\Delta B_z \), reaches a maximum when \( \eta = \Gamma/2 \).

This method can be simpler to implement in some applications for DC measurements of small fields, since compensation coils, for instance, can be used to apply the modulation, and there is no need for circularly polarized microwaves. In certain DC and low-frequency applications such as for biomagnetic signals, this field modulation can be averaged out. Furthermore, employing an oscillating bias field relaxes constraints on bias stability, a concern for precision sensors.

V. MAGNETIC FIELD SENSITIVITY

To demonstrate the magnetic sensitivity of the frequency modulated zero-field magnetometer, we scanned a small magnetic field around zero. The derivative fluorescence signal as detected in a properly phased LIA output depends linearly on the field. The calibration signal is shown in Fig. 4(a) with modulation frequency 2.3 kHz, and modulation depth 40 kHz. The data near zero field are fit to a straight line in order to translate the LIA output signal to magnetic field. For noise measurements, the LIA output is recorded for 1 s while the background magnetic field is set to zero. The data are passed through a fast-Fourier transform and displayed in Fig. 4(b). For noise frequencies between DC-30 Hz we observe a \( 1/f \)-behaviour of the magnetic noise that we attribute to ambient noise, primarily arising from the compensation-coil current stability. While the noise floor at 250 pT/\( \sqrt{\text{Hz}} \) can be attributed to laser-intensity noise, a photon shot noise limit of 4 pT/\( \sqrt{\text{Hz}} \) is achievable. The peaks at 50 Hz and harmonics are attributed to magnetic noise in the lab as demonstrated by their absence in the magnetically insensitive spectrum, which is obtained operating at an off-resonant microwave frequency of 2900 MHz. The electronic noise floor was measured at \( \approx 70 \text{ pT}/\sqrt{\text{Hz}} \).

VI. CONCLUSION

We have demonstrated a NV-based zero-field magnetometer with a 250 pT/\( \sqrt{\text{Hz}} \) noise floor. This device can be useful in applications where a bias field is undesirable and extends the dynamic range of NV magnetometry to cover existing zero-field technologies such as SQUIDs and alkali-vapor magnetometers. ZF magnetometry with single NVs will be presented in a later publication. Improvements in the present technique will result in sensitivities that are useful for ZULF-NMR, and with further miniaturization, these zero-field diamond sensors can find use in biomagnetic applications such as magnetoencephalography and magnetocardiography.

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