Wide-Field Optical Microscopy of Microwave Fields Using Nitrogen-Vacancy Centers in Diamonds

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Spatial and spectral analysis of electromagnetic fields at microwave frequencies is crucial for many applications, spanning a wide range of disciplines. Examples include the study of quantum electromagnetic metamaterials,[2,3] nanomaterials,[4] magnonics,[5–7] integrated circuit analysis,[8] and biological sensing.[9] In the past decades, scanning probe microscopy,[10–12] superconducting quantum interference devices,[13,14] and atomic vapor cells[15,16] have been used to improve the sensitivity and frequency resolution of microwave microscopy. However, the requirements of a scanning mechanism, cryogenic cooling, and/or a relatively large sensor size limit their relevance for practical applications.

Here we demonstrate wide-field optical microscopy of microwave fields using ensembles of nitrogen-vacancy (NV) color centers in diamond. Owing to NV properties, the microwave field can be mapped onto the population of spin states of NV centers, which in turn can be read out optically.[17] The negatively charged NV center is a lattice defect in diamond, which consists of a substitutional nitrogen atom and an adjacent vacancy. The spin states of NV centers can be optically addressed and are sensitive to DC and AC magnetic fields.[18,19] To date, NV centers have been utilized in magnetometry,[20–23] nuclear magnetic resonance detection,[24–30] targeted cells imaging,[31] and intracellular thermometry.[32] Recently, the ability of NV centers to detect microwave magnetic fields has also been investigated.[33–38] Pulsed laser sequences[33–37] were used to measure Rabi oscillations between different spin ground states, and scanning probe techniques[37] were used to achieve nanoscale resolution. For many practical applications, however, wide-field imaging and a simple experimental procedure are beneficial. To accomplish this, we opt for continuous wave laser excitation[35–38] to measure the spin populations of NVs in the presence of microwave fields using a conventional inverted microscope. In this way, we demonstrate wide-field imaging of microwave fields over a 200 × 200 µm² area with submicrometer spatial resolution, and spectral analysis of the microwave field with a resolution bandwidth of 460 kHz (i.e., the linewidth of NV). Minimum detectable microwave power of tens of nanowatts and a large dynamic range of over 33 dB in microwave power is obtained. Importantly, by using a bias magnetic field we could control the microwave frequency that NV centers are sensitive to—via Zeeman effect—over a frequency range of 170 MHz (potentially up to 100 GHz [ref. [35]]). In addition, we demonstrate a high frequency sensitivity of 2.5 kHz Hz⁻¹/² for a single frequency-modulated microwave signal detection.

A schematic of our apparatus is depicted in Figure 1a (see Figure S1 in the Supporting Information for the detailed experimental setup). A diamond chip containing a thin layer of NV centers with NV density of 3–4 ppm and linewidth of 460 kHz (see the Experimental Section for detail) contributes to spatial and spectral resolutions and signal-to-noise ratio. This diamond chip is closely placed on top of a microwave circuit under investigation, allowing near field imaging of microwave fields. An inverted optical microscope is used to deliver green (532 nm) laser probe light and NV fluorescence (600–750 nm) is collected through the same objective. The collected light is filtered and focused onto an electron-multiplying charge coupled device (EM-CCD) for imaging. An electromagnet is used to provide a bias DC magnetic field B₀ that controls the frequency splitting υ₀ between the NV’s spin 0 ground state |0⟩₀ and the spin –1 ground state |–1⟩₀, according to υ₀ = D₀ − γ B₀. Here, D₀ = 2.87 GHz is the crystal field splitting and γ(2π) = 28 MHz mT⁻¹ is the gyromagnetic ratio. For simplicity, in this work we align B₀ with one of possible NV orientations and focus on states |–1⟩₀ and |0⟩₀ only. The spectral analysis of an unknown microwave field is accomplished by scanning υ₀ over a wide frequency range, by sweeping a voltage applied to the electromagnet, while monitoring the NV fluorescence intensity. As illustrated in Figure 1b, when the microwave frequency f₉RF is resonant with υ₀, the NV is driven from |0⟩₀ to |–1⟩₀ by the microwave magnetic field with polarization perpendicular...
to the NV axis.\textsuperscript{[44]} This results in a reduction in the fluorescence intensity, since $|{-1}\rangle$, has a higher nonradiative decay rate than $|0\rangle$.\textsuperscript{[39–41]} Therefore, the amplitude of microwave magnetic fields can be determined by measuring the fluorescence contrast with and without microwave signals. Each frequency in the microwave signal results in three dips in NV fluorescence intensity as a result of hyperfine interactions with the nuclear spin of the $^{14}$N in the NV center\textsuperscript{[39]} (Figure 1c).

To characterize the efficacy of NV centers as microwave spectrum analyzers, the fluorescence is measured (averaging over a $5 \times 5 \mu m^2$ region at the central field of view) in the presence of known microwave signals with various frequencies and intensities. First, the apparatus is calibrated by detecting harmonic microwave signals of known frequency (in the range of 2.72 to 2.84 GHz) (Figure 2a). The calibration curve, shown in Figure 2b, features the linear dependence of $v_0$ on the voltage supplied to the electromagnet, with the slope $S = -18.12(6) \text{ MHz V}^{-1}$. From the fit, we also obtain the zero field splitting value $D_0 = 2.866(3) \text{ GHz}$ which is in perfect agreement with the literature value reported at room temperature.\textsuperscript{[42]} With this calibration, we obtain the spectra (blue lines in Figure 2c) of more complex microwave signals, and compared the results with the spectra (red lines) obtained using a real-time spectrum analyzer (RSA). Good agreement between the two sets of data is observed. The small discrepancy in the absolute spectral line position can be attributed to mechanical instability of the setup and hysteresis of the electromagnet.

Next, we demonstrate that NV centers can be used as a tunable microwave spectrum analyzer, whose sensitivity level and dynamic range can be adjusted by the probe laser power. As shown in Figure 2d, adjusting the laser power from 28 µW to 33 mW allows detection of microwave signals with powers in the range of $-50 \text{ dBm}$ to $-10 \text{ dBm}$ measured at the output of function generator. (The microwave frequency $f_{\text{MW}}$ is fixed at 2.834 GHz for this measurement. A detailed study of the dependence of the fluorescent contrast on microwave and laser powers is summarized in Figure S3 in the Supporting Information.) A logarithmic dependence of fluorescence contrast on the microwave power and a dynamic range of 33 dB of microwave power (from $-45 \text{ dBm}$ to $-12 \text{ dBm}$) are achieved by an intermediate probe laser power of 1.49 mW (black line in Figure 2d).

Figure 1. Experimental configuration and principle of optical microscopy and spectroscopy of microwave field using NV centers in diamond. a) Schematic of the experimental setup. b) Simplified electronic energy levels of negatively-charged NV center. The ground state is a spin triplet with a splitting $v_0$ between the spin-0 ground state $|0\rangle$, and the spin-(-1) ground state $|{-1}\rangle$, determined by crystal field splitting $D_0$ and Zeeman shift $\delta V$ induced by an external magnetic field $B_z$, which is aligned with the orientation of one class of NV centers. The nonradiative pathway (wiggly arrows) enables optical polarization and readout of the NV spin states. c) A frequency spectrum of a single frequency microwave signal obtained by scanning the external magnetic field $B_z$. When a microwave signal is on resonance with the splitting between $|0\rangle$ and $|{-1}\rangle$, three dips in fluorescence intensity will be observed due to hyperfine interactions with the $^{14}$N atom of NV center.

measured at the input port of microscope. We thus estimate the amplitude sensitivity of our apparatus to be $\approx 45 \text{ mT Hz}^{-1/2}$ (See Note I of the Supporting Information) for the $5 \times 5 \mu m^2$ region. At a higher laser power of 33.86 mW (red line), the fluorescence contrast linearly depends on the microwave magnetic field amplitude as shown in Figure 2d inset.

We image microwave fields by simultaneously measuring the fluorescence of NV center ensembles at all positions in the field of view of the microscope. The microwave structure (Figure 3a) consists of two 20 µm wide gold transmission lines realized on microchip coverslip, using standard microfabrication techniques. The gap between the two lines varies from 20 to 40 µm across the field of view. In this demonstration, the transmission line on the right (left) carries a $-10 \text{ dBm}$ microwave signal of radio frequency $R_F_1 = 2.78 \text{ GHz}$ ($R_F_2 = 2.80 \text{ GHz}$). The fluorescence contrasts indicating the microwave field amplitude are measured by tuning NV centers in resonance with $R_F_1$ and $R_F_2$ are shown in Figure 3b,c, respectively. The spectra of microwave fields at three positions indicated in Figure 3a are shown in Figure 3d–f. In these figures, the depths of resonance dips represent the amplitudes of microwave field: the larger the amplitude, the larger the dip. As expected, while the amplitude of the microwave field is almost identical in the gap between the two transmission lines, there are significant differences on either side of the transmission lines. Cross-talk between two lines is also observed, as expected, since the wavelength of the microwave (10.7 cm) signal used is much larger than the feature sizes of the circuit.

Owing to the narrow linewidth of its hyperfine lines, the NV center can effectively transduce small variations in the frequency of microwave signals to measurable changes in the fluorescence intensity. Thus, NV centers are promising candidates for low-noise frequency modulation spectroscopy.\textsuperscript{[41]} In this work, we demonstrate the ability of NV centers to detect frequency-modulated (FM) microwave signals in real time using the same experimental apparatus. In general, an FM signal can be written as $y(t) = A_0 \cos(2\pi f_0 t + k_{FM} x(t))$, where $A_0$ is the amplitude of microwave carrier signal, $f_0$ is the carrier frequency, and $k_{FM}$ is the amplitude-to-frequency gain. $x(t)$ is a modulating signal, which, in its simplest form, can be written as $x(t) = A_m \cos(2\pi f_m t)$. $A_m$ and $f_m$ are amplitude and
Figure 2. Microwave spectroscopy using NV centers. a) Fluorescence contrast of NV centers as a function of voltages driving the electromagnet, in the presence of microwave signals having frequencies from 2.72 GHz to 2.84 GHz. This data set is used to calibrate the apparatus and b) to establish the relationship between the applied voltage and NV resonant frequencies. Each point in (b) is extracted by fitting measurements in (a) to three Lorentzians. The magnitudes of fitting error is less than 0.7 mV (smaller than the size of data points, see detail in Figure S2 in the Supporting Information). c) Microwave spectra acquired using NV centers (blue lines) and a real-time spectrum analyzer (RSA, red lines). In the upper panel, a simple harmonic microwave signal with frequency of 2.74 GHz was used, while in the lower one signal centered at 2.74 GHz with side bands separated by 10 MHz was used. The single-pass scanning data obtained by taking one EM-CCD frame at each frequency are shown in light blue, while three frames shift-averaged data are shown in dark blue. The resolution bandwidth of the RSA is 20 kHz. d) The fluorescence contrast of NV centers exposed to microwave powers from −50 to −10 dBm and probe laser powers of 137.2 µW (blue lines), 1.49 mW (black lines), and 33.86 mW (red lines). The thin lines in light colors are the measurement data, while the thick lines are the fitted curves. The microwave frequency is fixed at 2.834 GHz. Inset: same data with x-axis proportional to the amplitude of magnetic field component of the microwave signal. The fluorescence data in this figure are averaged over a 5 x 5 µm² region near the central field of view.

Figure 3. Imaging the microwave field. a) A microscope image of the microwave structure with two gold transmission lines fabricated on a cover glass. The left and right line carries microwave signal at radio frequency RF₁ and RF₂, respectively. b,c) The images of fluorescence contrast of NV centers at frequency b) RF₁ = 2.80 GHz and c) RF₂ = 2.78 GHz. The RF intensity scale bars are in arbitrary unit. d–f) The microwave spectra from three positions indicated in (a). The powers of the microwave signal are −10 dBm.
frequency of the modulating signal, respectively. Therefore, the time-dependent frequency of the FM microwave signal can be written as $f(t) = f_0 + \Delta f \cos(2\pi f_m t)$, where $\Delta f = k_{\text{mic}} A_m$ is the maximum frequency deviation.

The principle of NV-based FM detection is depicted in Figure 4a. To achieve the best performance, the operating point is chosen by selecting the value of $B_0$ so that the FM carrier frequency $f_c$ corresponds to the position of maximum slope on the curve, indicated by the black vertical lines in Figure 4a. The time-dependent frequency component $\Delta f \cos(2\pi f_m t)$ of the microwave signal is then mapped to the fluctuation of fluorescence intensity. This fluctuation is finally recorded by the EM-CCD, and the original signal $x(t)$ is recovered.

We detected frequency-modulated microwave signals with various frequency deviations $\Delta f$ and modulating frequencies $f_m$. Figure 4b shows the results for a sine-wave with $\Delta f = 200$ kHz and $f_m = 0.1$ Hz. For this low frequency measurement, we take reference frames and detection frames alternatively (with same exposure time) to cancel out low frequency (1/f) noise, and obtain a sensitivity of $4$ kHz Hz$^{-1/2}$ at $f_m = 0.1$ Hz (Figure S4a, Supporting Information). Figure 4c–g show demodulated FM microwave signals when the signal $x(t)$ is a square wave with $f_m = 1$ Hz and $f_m = 20$ Hz, and $\Delta f$ varies from 100 to 300 kHz. The demodulated signals resemble the original modulating signals with the same frequency $f_m$ as $x(t)$, and the amplitude of the demodulated signal is proportional to $\Delta f$. The measurement of fluorescence fluctuations without FM is plotted in Figure 4h.

The fluorescence contrast is an important figure of merit that determines the sensitivity and dynamic range for microwave field detection. As shown in Figure 2d, and discussed in detail in the Supporting Information, the fluorescence contrast is strongly dependent on the microwave and laser power used. In our experiments, the NV is continuously driven into the $|0_g\rangle$ state with an effective optical polarization rate $G_{\text{op}}$, determined by the power of the probe laser, while the applied microwave field drives the NV centers from $|0_g\rangle$ to $|1_g\rangle$, with a Rabi frequency $\Omega_{\text{MW}}$. The steady fluorescence contrast $C$ at resonance is given by

$$C = \frac{(1-\beta)\xi G_{\text{op}}}{(1+\beta)\gamma_1 + G_{\text{op}}} \times \frac{2\Omega_{\text{MB}}^2}{4\Omega_{\text{MB}}^2 + \gamma_1 (2\gamma_1 + G_{\text{op}})}$$

where $\xi = 1/12$ is the portion of NVs on resonance with the applied microwave signal (due to other NV directions and nuclear spin states), $\beta$ is the ratio of fluorescence intensity of $|1_g\rangle$ to $|0_g\rangle$, $\gamma_1$ is relaxation rate of ground state due to spin-lattice relaxation, $\gamma_1$ is dephasing rate of the ground state, all these parameters are material properties (i.e., not dependent on the laser power or microwave field amplitude). When the amplitude of microwave field is small ($\Omega_{\text{MB}}^2 \ll \gamma_1 G_{\text{op}}$), the contrast $C \propto \Omega_{\text{MB}}^2 / G_{\text{op}}$ assuming $G_{\text{op}} \gg \gamma_1$, which indicates a lower laser power should be used to measure a week microwave field. However, for a strong microwave field ($\Omega_{\text{MB}}^2 \gg \gamma_1 G_{\text{op}}$),
Experimental Section

Diamond Sample with NV Centers: The single crystal chemical vapor deposition (CVD) grown diamond chip (Element Six) consists of high purity diamond substrate (<1ppb N$_2$) on which an additional thin layer diamond is grown that is doped with N (natural abundance 99.6% $^{14}$N) during the CVD synthesis process. The thickness of the layer is 0.9 μm with a concentration of 10.5 ppm as determined by secondary ion mass spectroscopy. Post synthesis the sample was irradiated with 4.5 MeV electrons at a flux of $2.5 \times 10^{19} \text{cm}^{-2} \text{s}^{-1}$ for 10 h and then annealed at $800 \degree C$ for 8 h and $1200 \degree C$ for 1 h. The density of NV- is estimated as 3–4 ppm by measuring the optical absorption at liquid nitrogen temperatures. By integrating the absorption coefficient across defect’s zero-phonon-line, it can be converted into a NV concentration. $^{49}$ The CVD diamond chip is grown in the crystal direction of <100>, and the side surface of the diamond is <110>, and the NV centers are in direction of <111>.

Experimental Setup for Fluorescence Imaging of NV Centers: The detailed experimental setup is shown in Figure S1 (Supporting Information). A 532 nm probe laser (Coherent Compass 315M-100) is attenuated by neutral density filters (Thorlabs, FW2AND) to adjust the power as needed, followed by a beam expander, a convax lens, and an objective (Olympus SLMPan 50X/0.35NA or 20X/0.35NA as needed) providing wide field illumination. The laser power is measured by a power meter (Newport 1928-C). The fluorescence of NV centers is collected by the same objective, and filtered (532-nm Semrock StopLine notch filter, E-grade, and 632.8-nm Semrock EdgeBasic long-wavelength-pass filter) before it is sent to an EM-CCD (Hamamatsu C9100-13). The optical path for fluorescence is secured inside an inverted microscope (Olympus IX71) to reduce the noise from the environment. The diamond sample with NV centers is placed on the microwave structure fabricated on a cover glass on the microscope stage. The fabrication of the microwave structure is defined by photolithography, then electron beam evaporation titanium/gold layer, and lift-off. The bias magnetic field $B_b$ is provided by an electromagnet (APW Co. EM075-12-222), whose applied voltage is generated by a data acquisition device (NI DAQ USB-6361) with a home-made power amplification circuit (using TI LM358 Op Amp). This setup enables us to sweep the voltage applied on the electromagnet with resolution of 1 mV, resulting in the ability to tune the resonant frequency from 2.68 to 2.87 GHz. The frequency tuning sensitivity is measured to be $S = -18.12(6)$ MHz V$^{-1}$ (Figure 2b), and from this we obtain the frequency resolution of 18 kHz (assuming 1 mV voltage resolution). The microwave signals are generated by two function generators (HP ESG-3000A, and Rohde & Schwarz SMC100A), with a waveform generator (Agilent 33210A) for frequency modulation. The microwave spectra measured with NV centers are compared with a radio frequency real-time spectrum analyzer (RSA, Tektronix, RSA 3303B).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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M.Z. helped with detection noise reduction and analysis. A.S. and X.A. helped build and optimize the setup. All authors contributed to the discussion.

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