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Chip-scale nanofabrication of single spins and spin arrays in diamond

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ABSTRACT

We demonstrate a technique to nanofabricate nitrogen vacancy (NV) centers in diamond based on broad-beam nitrogen implantation through apertures in electron beam lithography resist. This method enables high-throughput nanofabrication of single NV centers on sub-100 nm length scales. Secondary ion mass spectroscopy (SIMS) measurements facilitate depth profiling of the implanted nitrogen to provide three-dimensional characterization of the NV center spatial distribution. Measurements of NV center coherence with on-chip coplanar waveguides suggest a pathway for incorporating this scalable nanofabrication technique in future quantum applications.

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Solid-state spins are attractive for quantum information processing in part because mature nanofabrication techniques developed for the semiconductor industry can be used for their production. Among single-spin systems the negatively-charged nitrogen vacancy (NV) center in diamond stands out because of its individual addressability,¹ optical spin polarization, and millisecond room-temperature spin coherence.² These properties have generated intense interest in the use of NV centers for quantum device applications in dipolar-coupled quantum registers³ and hybrid quantum computing architectures.⁴⁻⁸ Many of these architectures present exacting device requirements that necessitate the development of methods to place NV centers in diamond on 10 nm length scales. Nitrogen implantation into high-quality chemical vapor deposition (CVD) diamond is a promising method for engineering NV centers and has been used to fabricate single-qubit^{9,10} and two-qubit^{3,11} NV center devices. Proposals for nanometer-scale, spatially controlled NV center fabrication for quantum device applications have commonly relied on serial nitrogen implantation with scanning probe microscope apertures^{12,13} and ultracold ion sources.¹⁴ We demonstrate an alternative approach for spatially controlling NV center formation by using electron beam lithography resist masks to pattern ion implantation. This approach offers high spatial resolution and high throughput: resist aperture arrays are scalable to 10 nm aperture diameters and 30 nm aperture pitches¹⁵ and are patterned at a rate of 10^3 per second. This nanofabrication technique makes possible the production of dense, large-scale NV center arrays in diamond for spin-based quantum computing architectures.

The nanofabrication technique described in this letter relies on nitrogen implantation into masked, high-purity CVD diamond substrates. SIMS measurements showed the intrinsic nitrogen content of the substrates to be $\sim 7 \times 10^{15} \text{ cm}^{-3}$. Before masking, a 10 nm SiO_2 layer was deposited on the diamond surfaces to mitigate ion channeling during implantation. Positive electron beam lithography resist, approximately 300 nm thick, was applied to the samples and resist apertures were patterned using a 100 kV electron beam lithography system. The minimum aperture diameter achieved after resist development, as measured with a scanning electron microscope (SEM), was ~ 30 nm (Figure 1a). Aperture diameters smaller than 30 nm are possible, but require thinner resist masks.¹⁵ This spot size,

in combination with ion straggling during implantation, determines the lateral NV center placement accuracy of this technique. After development the apertures were implanted with 20 keV $^{15}\text{N}^+$ ions, which enabled isotopic tagging of the implanted nitrogen to distinguish it from ^{14}N in the diamond substrate.¹⁰ ^{15}N has a low natural abundance (0.4%) and a distinct nuclear spin ($I = 1/2$) from ^{14}N ($I = 1$). Monte Carlo simulations using the Stopping Range of Ions in Matter (SRIM) program¹⁶ for these implantation conditions predict a lateral straggle of 9 nm. Following implantation, the samples were annealed in Ar at 850° C to induce vacancy diffusion to form NV centers and then annealed in O₂ at 420° C to reduce photochromism.¹⁷ Diffusion of nitrogen atoms, a potential source of NV center placement uncertainty, is expected to be negligible under these annealing conditions.¹⁸ Further process details are provided in the Materials and Methods.

Photoluminescence measurements of masked, implanted samples demonstrated that the resist apertures were effective at spatially controlling NV center formation. The photoluminescence measurements discussed in this letter focus on a CVD diamond (sample A) patterned with a 60 x 60 array of 60 ± 10 nm diameter apertures (Figure 1b) with an array pitch of 1 μm (Figure 1c). This aperture diameter resulted in roughly one NV center per aperture for the 10^{12} cm^{-2} $^{15}\text{N}^+$ dose applied to sample A. A photoluminescence image of the patterned area shows the resultant spin array after implantation, annealing, and lithographic fabrication of a short-terminated coplanar waveguide (CPW) (Figure 1d). White contrast in the array comes from NV center photoluminescence; the individual white dots correspond to NV centers positioned with the resist apertures, while formerly masked regions of the sample showed no NV center emission. Roughly one third of the aperture sites produced single NV centers, confirmed by photon antibunching measurements (Figure 1e). Two single NV centers are circled in Figure 1d for reference. Brighter dots contain more NV centers—the distribution in the number of NV centers per site originates from the random incorporation of nitrogen atoms onto substitutional lattice sites¹⁹ and the diffusion of vacancies to these nitrogen atoms during annealing. The average number of NV centers per aperture site was determined by analyzing the minima of photon antibunching measurements²⁰ performed on 32 aperture sites. In cases where photon antibunching

measurements could not distinguish between 2 or 3 NV centers in an aperture site, photoluminescence intensity measurements were used to estimate the number of NV centers. On average 1.4 ± 0.2 NV centers formed per aperture, corresponding to a nitrogen to NV center creation efficiency of $(5 \pm 2)\%$. The distribution of NV centers formed per aperture shows reasonable agreement with a Poisson distribution (Figure 1f), as expected from the low NV center creation efficiency.

The depth placement of implanted NV centers, commonly inferred from SRIM simulations, is primarily determined by the nitrogen implantation energy. Using SIMS we have directly measured nitrogen depth distributions as a function of implantation energy. This technique relies on implanting ^{15}N concentrations well above the ^{15}N background of the CVD diamond substrates and well above the SIMS detection sensitivity for ^{15}N in diamond ($3 \times 10^{14} \text{ cm}^{-3}$). A $4 \times 4 \text{ mm}^2$ (100) CVD diamond with a ^{15}N background $< 10^{14} \text{ cm}^{-3}$ was exposed to broad beam nitrogen implantations with 10 keV, 20 keV, and 30 keV implantation energies (sample B). The implantations were performed sequentially in separate quadrants of the diamond with a 2 mm diameter ^{15}N ion beam aligned normal to the sample surface. SIMS measurements from sample B are plotted with SRIM simulations of the nitrogen depth distributions (Figure 2a-c). The simulations were normalized to the implantation dosages and were performed at a 1 degree tilt to match the sample miscut from (100) measured by x-ray diffraction. The SIMS measurements show a local maximum in nitrogen concentration positioned in good agreement with the SRIM simulations. However, the measured nitrogen distributions also extend deeper into the diamond than is predicted by SRIM, likely due to ion channeling.^{21,22}

To establish that the broad nitrogen depth distributions resulted from ion channeling we performed 20 keV ^{15}N implantations on a (100) CVD diamond (sample C) partially masked with an amorphous, 8 nm thick SiO_2 layer. The background ^{15}N concentration of sample C was $< 10^{15} \text{ cm}^{-3}$. SIMS measurements on sample C (Figure 2d) from the masked region (green squares) show reduced ^{15}N concentrations at depths $> 25 \text{ nm}$ compared to ^{15}N concentrations from the unmasked region (black squares). These measurements demonstrate that the increased dispersion in the ion beam angle resulting from the SiO_2 layer mitigates ion channeling in the diamond substrate. A similar reduction in the ion channeling was

obtained for a sample implanted 10 degrees from normal incidence (not shown). A thicker amorphous layer or a greater implantation angle could allow for greater control over channeling effects, although the increased lateral ion dispersion resulting from these methods must also be considered in the overall NV center placement accuracy.

To connect the depth distribution of nanofabricated NV centers with the SIMS implanted-nitrogen distributions, we performed continuous-wave electron spin resonance (ESR) measurements on sample A to spectroscopically probe the NV center hyperfine coupling. The AC magnetic field in these measurements was generated by applying microwave power to the short-terminated CPW,²³ shown in Figure 1d, which was lithographically aligned to the sample A spin array. ESR spectra of six individual NV centers showed hyperfine doublets with splittings of ~ 3.05 MHz (Figure 3a), indicating coupling of the NV center electronic spin to the spin- $1/2$ ^{15}N nucleus.¹⁰ The exclusive creation of ^{15}N NV centers from implanted ^{15}N is consistent with previous reports¹¹ and eliminates the possibility that NV centers were formed from vacancies created in the ^{15}N ion tracks diffusing to ^{14}N atoms in the diamond substrate.

We also performed room-temperature, pulsed ESR experiments to characterize the coherent spin dynamics of nanofabricated NV centers. The CPW facilitates coupling NV centers to a spatially-localized AC magnetic field to produce coherent spin rotations on nanosecond timescales;²³ Figure 3b shows 244 MHz Rabi oscillations performed on a single NV center. Figure 3c shows Hahn spin-echo measurements for two representative NV centers that yielded spin coherence (T_2) times of $4 \mu\text{s}$ and $22 \mu\text{s}$, respectively. These T_2 times are shorter than a previously-reported $T_2 = 350 \mu\text{s}$ for unmasked nitrogen implantation¹¹ and are likely a consequence of photochromism^{24,25} and spin bath decoherence.²⁶ Due to the low NV center creation efficiency, approximately 30 nitrogen ions were implanted in each aperture and were distributed over a volume expected to contain only 1 native nitrogen impurity based on measured ^{14}N concentrations.

The small electric and magnetic dipole moments of single NV centers often lead to kHz-scale couplings in NV center quantum computing architectures.^{3,6,7} Detection of these kHz-scale couplings will require extending the T_2 times of implanted NV centers to the millisecond timescales achieved for

native NV centers in isotopically pure diamond.² The results presented in this letter suggest several pathways for doing so. Lowering the implantation dosage to admit only two or three nitrogen atoms per aperture would result in spin environments similar to those of highly-coherent implanted NV centers. Recently demonstrated co-implantation techniques also hold promise for increasing the NV center creation efficiency to lessen spin bath decoherence in future devices.^{13,27} Finally, isotopically pure diamond substrates could be used to eliminate the ¹³C nuclear spin bath. Combining the high spatial resolution and high throughput of this NV center placement technique with millisecond spin coherence times would enable the production of coherent, few-spin quantum registers in diamond and deterministically-positioned single spins for hybrid quantum computing devices.

Materials and Methods. Resist Aperture Fabrication. Sample A was a 4.5 x 4.5 mm² (100) electronic grade diamond from Element Six. The sample was first cleaned in boiling nitric/sulfuric (3:2) acid to remove graphitic carbon from the sample. Evaporated Ti/Pt (100 Å/600 Å) alignment marks were fabricated by photolithography on the sample surface. The 10 nm SiO₂ layer was then deposited on the sample surface. The sample was coated with ~300 nm of positive electron beam lithography resist (ZEP520A) and 110 Å of thermally-evaporated Au. Aperture arrays were patterned using the 5th lens of a JEOL JBX-6300FS electron beam lithography system with a 500 pA beam current and a 100 kV beam voltage. The aperture arrays were patterned over areas of low surface photoluminescence determined by confocal microscopy measurements performed prior to SiO₂ deposition. The sample was developed and implanted with a ¹⁵N⁺ dosage of 10¹² cm⁻² by Cutting Edge Ions, LLC. The implantation energy was 20 keV and the implantation was performed normal to the sample surface. The resist layer was removed and the sample was annealed in an Ar atmosphere at 850° C for 10 minutes to induce vacancy diffusion to form NV centers. The SiO₂ layer was removed with hydrofluoric acid and the sample was annealed in O₂ at 420° C for four hours to oxygen terminate the sample surface to promote the formation of negatively-charged NV centers.¹⁷ In the final fabrication step a Ti/Pt (100 Å/900 Å) short-terminated CPW was fabricated on top of the NV array produced with the 60 nm diameter apertures.

For SEM imaging additional samples were prepared with identical procedures. The resist was coated with a thin layer of Au/Pd before SEM imaging.

SIMS Measurements. SIMS measurements were performed by the Evans Analytical Group, LLC. The samples used in this work were electronic grade (sample B) and IIIa grade (sample C) single crystal diamonds from Element Six. Sample B was implanted with ion dosages of 10^{12} cm^{-2} ($^{15}\text{N}^+$, 10 keV), $7 \times 10^{11} \text{ cm}^{-2}$ ($^{15}\text{N}^{2+}$, 20 keV), and $7 \times 10^{11} \text{ cm}^{-2}$ ($^{15}\text{N}^{3+}$, 30 keV). SRIM simulations used a density of 3.52 g/cm^3 and a displacement energy of 37.5 eV.²⁸ Sample B was etched in a Cl_2/Ar plasma to remove $\sim 750 \text{ nm}$ of diamond before implantation and SIMS measurements to confirm that the SIMS surface peak is a measurement artifact, as it appeared unchanged after etching. Atomic force microscope measurements of sample B showed a root-mean-square surface roughness of $\leq 3.5 \text{ nm}$. Sample C was implanted with $^{15}\text{N}^+$ dosages of $5 \times 10^{12} \text{ cm}^{-2}$ in the masked and unmasked regions. These 20 keV implantations were performed normal to the sample surface in a setup at Lawrence Berkeley National Laboratory.¹³

Photoluminescence and ESR Measurements. Photoluminescence measurements were performed using a home-built confocal microscope.²⁹ Photon antibunching measurements and photoluminescence intensity measurements used to determine the number of NV centers per aperture site were performed with circularly polarized light in zero magnetic field to couple the 532 nm excitation laser equally to all structural orientations of the NV center.

ESR measurements were performed with an $\sim 261 \text{ G}$ static magnetic field applied along the NV center symmetry axis on the $m_s = 0$ to -1 spin sublevel transition. All ESR measurements were performed at room temperature. Optical excitation with a 532 nm laser polarizes the NV center spin state into the $m_s = 0$ level of its spin-1 ground state as a consequence of the defect's spin-selective orbital relaxation.³⁰ Similarly, spin state readout after resonant manipulation was accomplished by monitoring the NV center photoluminescence intensity, which depends linearly on the population in the $m_s = 0$ state. Continuous-wave ESR experiments were performed with low power continuous optical illumination of the NV center and low power swept-frequency microwave current applied to the CPW. Pulsed ESR

experiments used a sampling scheme. After initialization into the $m_s=0$ spin state with a $2 \mu\text{s}$ optical pulse, the excitation laser was turned off for a fixed delay using an acousto-optic modulator. A microwave pulse sequence was then applied to the CPW. The microwave pulses were generated by gating a microwave signal generator with mixers modulated by an arbitrary waveform generator. The signal was amplified and passed through microwave filters and an isolator to optimize the pulse profiles at 2.14 GHz. After the microwave pulse sequences were applied, the NV center spin state was read out by turning the laser and photon counters on. Each data point represents an average of $\sim 10^5$ measurements. For Rabi measurements this sampling scheme was iterated with microwave pulses of progressively longer duration. For Hahn spin-echo measurements this sampling scheme was iterated with progressively longer pulse delays, τ , in the $(\pi/2 - \tau - \pi - \tau - \pi/2 - \text{echo})$ pulse sequence. The lengths of the $\pi/2$ and π pulses were roughly 10 ns and 20 ns, respectively.

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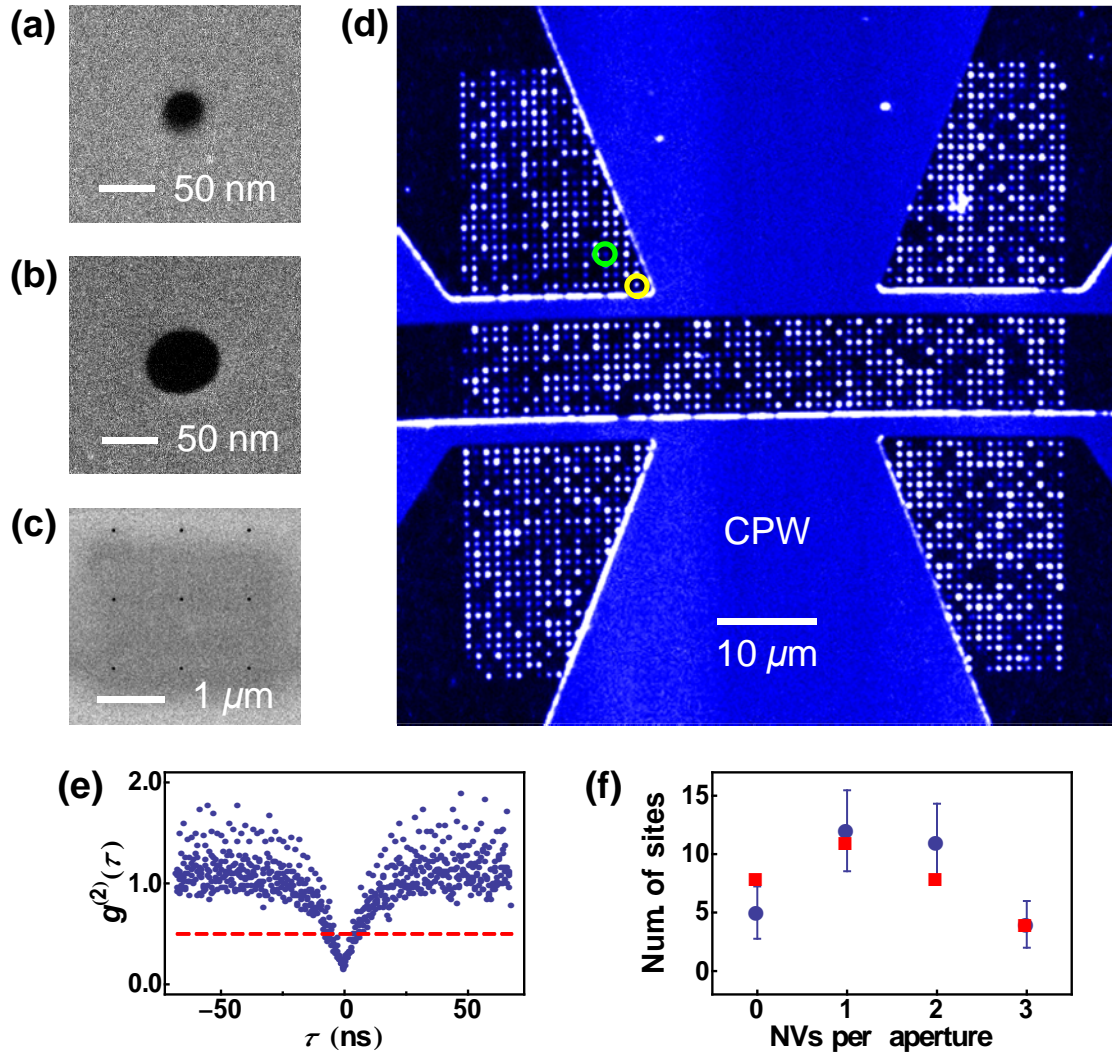


Figure 1. (a) SEM micrograph of an ~ 30 nm diameter resist aperture used to mask nitrogen ion implantation to spatially control NV center formation. (b) SEM micrograph of an ~ 60 nm diameter resist aperture. After fabrication, the NV centers resulting from this aperture diameter were analyzed in photoluminescence and ESR experiments. (c) SEM micrograph of an array of 60 nm diameter apertures. (d) Spatial photoluminescence image of an array of NV centers resulting from resist masking, implantation, and annealing. The white dots in the image result from NV center photoluminescence. A lithographic, short-terminated CPW is also visible. Two single NV centers used in further measurements are circled. (e) Photon antibunching curve from the NV center circled in yellow in Figure 1d. $g^{(2)}(0) < 0.5$, indicating it is a single quantum emitter. The data are uncorrected for background luminescence and dark counts of the photon detectors. (f) A histogram of resist aperture sites yielding zero, one, two, and three NV centers per aperture site (blue dots) determined by photon antibunching measurements on 32 aperture sites. The results are compared with a Poisson distribution with mean 1.45 (red squares).

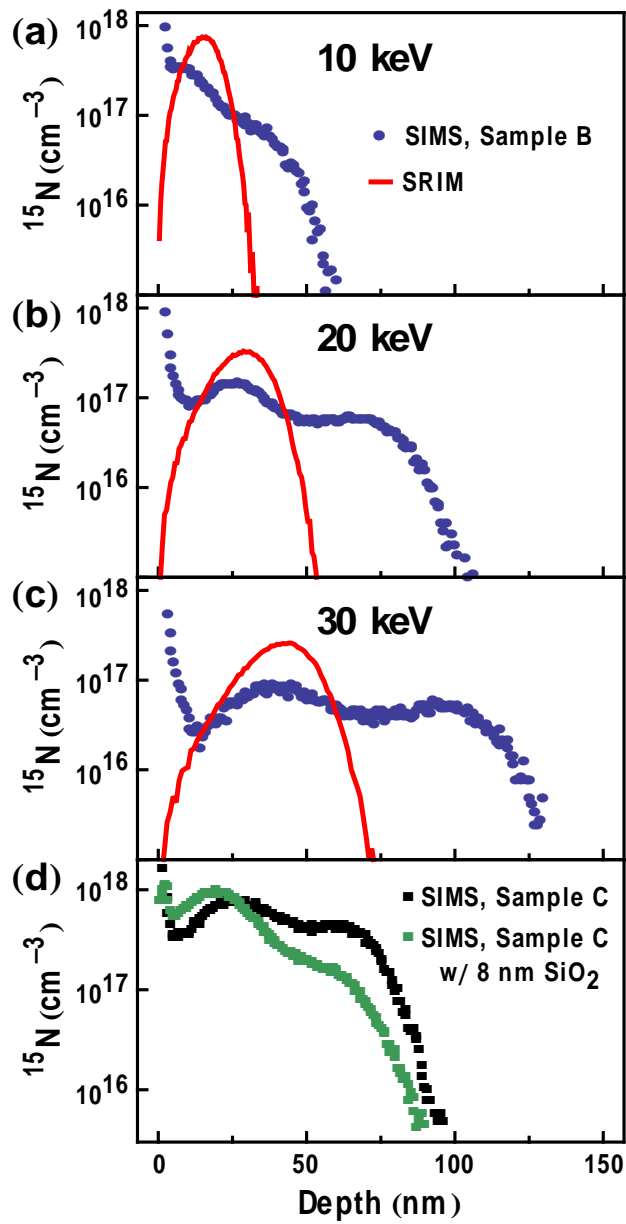


Figure 2. SIMS measurements of ^{15}N concentration versus depth (blue dots) and corresponding SRIM simulations (red lines) for a sample exposed to (a) 10 keV, (b) 20 keV, and (c) 30 keV ^{15}N implantations. The SRIM simulations were normalized to match the ^{15}N implantation dosage and were performed at a 1 degree angle to match the sample miscut. The measured ^{15}N concentrations that extend beyond SRIM predictions are a consequence of ion channeling. The sharp peaks in nitrogen concentration near the sample surface are an artifact of the SIMS measurements. (d) SIMS measurements for a sample half-masked with an amorphous 8 nm SiO_2 layer. SIMS measurements on masked portions of the sample (green squares) show a reduction in ion channeling relative to unmasked portions of the sample (black squares).

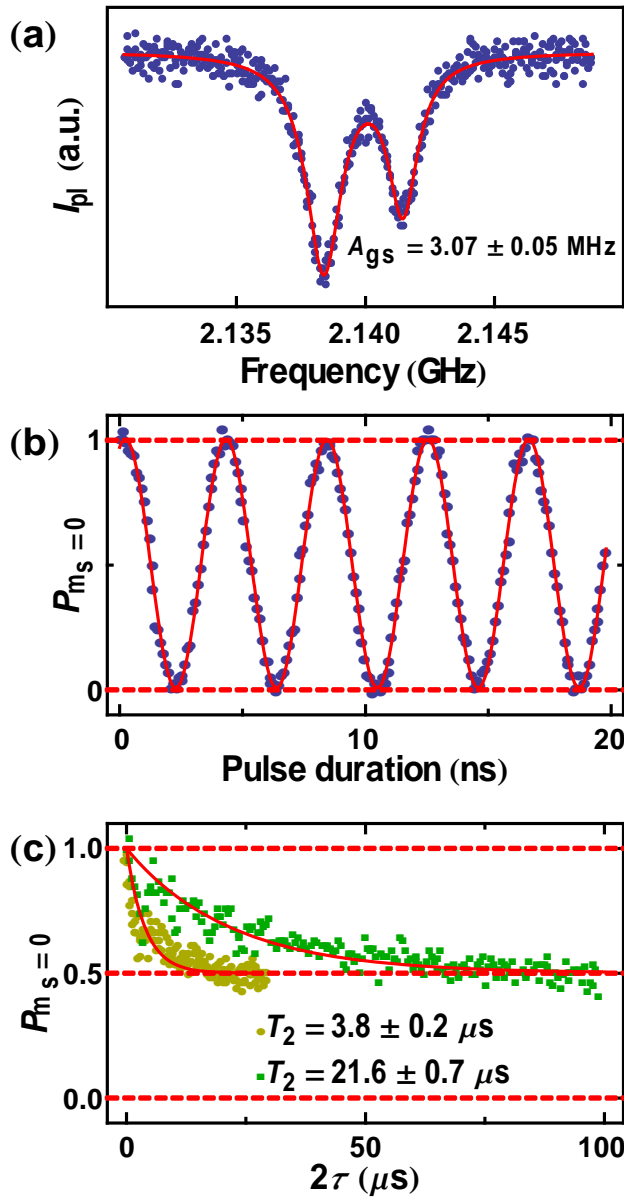


Figure 3. (a) Hyperfine spectrum (blue dots) for the NV circled in yellow in Figure 1d. Lorentzian fits (red line) to the two dips in photoluminescence show a splitting of 3.07 ± 0.05 MHz, indicating the coupling of the NV center electronic spin to a ^{15}N nuclear spin. (b) Rabi oscillations (blue dots) between the $m_s = 0$ and $m_s = -1$ spin sublevels for the single NV center circled in yellow in Figure 1d. The vertical axis corresponds to the $m_s = 0$ spin population, $P_{m_s=0}$, determined via photoluminescence intensity measurements. The intensity of $P_{m_s=0} = 1$ and $P_{m_s=0} = 0$ were calibrated from measurements of photoluminescence intensity after optical initialization and after an adiabatic passage,²³ respectively. The red line is a sinusoidal fit to the data yielding a Rabi frequency of 244 MHz. (c) Hahn spin-echo measurements of the NV center circled in yellow in Figure 1d (yellow dots) and the NV center circled in green in Figure 1d (green dots). In these measurements $P_{m_s=0}$ was calibrated from the photoluminescence intensity after optical initialization and after a microwave π -pulse. The solid red lines are exponential decay fits to the data.

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