Long-lived NV$^-$ spin coherence in high-purity diamond membranes

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Abstract. The electronic spin associated with the nitrogen vacancy (NV) color center in diamond is an excellent candidate for a solid-state qubit functioning as a quantum register or sensor. However, the lack of thin film technologies for crystalline diamond with low impurity levels hampers the development of photonic interfaces to such diamond-based qubits. We present a method for manufacturing slabs of diamond of 200 nm thickness and several microns in extent from high-purity single crystal chemical vapor deposition diamond. We measure spin coherence times approaching 100 µs and observe increased photoluminescence collection from shallow implant NV centers in these slabs. We anticipate these slabs to be appealing as quantum memory nodes in hybrid diamond nanophotonic systems.

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1. Introduction

Solid-state systems provide a unique platform for quantum information processing given their practical scalability and connection to device physics and well-understood models within the context of condensed matter physics [1, 2]. Within the solid-state field, there has been much interest in the nitrogen vacancy center (NVC) in diamond due to its optical addressability and readout [3], high-fidelity state preparation [4] and long spin coherence time [5] with a controllable set of ancilla qubits [6]—all available at room temperature. To date, all reports of long coherence times of NVCs have been in bulk diamond samples, even the most recent advances using diamond solid-immersion lenses [7]. However, most photonic engineering of the optical photons emitted by NVCs, including zirconium solid-immersion lens [8], GaP cavities [9] and plasmonic resonances [10, 11] have used nanocrystalline diamond of lesser quality than bulk diamond. In order to engineer optical interfaces to useful NVC spin qubits, a requirement for quantum repeaters, increased coupling between the emitted photons and spins with long-lived coherence is necessary.

A promising path forward is to leverage the advances of metamaterials, specifically photonic band-gap engineered two-dimensional (2D) devices [12, 13] and apply these to diamond substrates. However, there are currently no thin film heterogrowth technologies for long spin coherence ultrapure diamond. Various approaches have been investigated, including 3D cavities carved using focused ion beams (FIB) [14], and combination of ion slicing and FIB [15, 16], as well as thin film heterogrowth with FIB [17]; however, none have shown reliable spin and spectral properties. Recently reported diamond membranes [18], formed through epitaxial growth, show photoluminescence (PL) spectra consistent with bulk defects, but these films do not yet exhibit excellent spin properties. In this paper, we outline a method for mass-producing diamond nanoslabs, down to 200 nm in thickness, with heights up to 10 µm and lengths exceeding 10 µm. We show that this procedure maintains the purity of near-pristine diamond samples, as evidenced by spin coherence times of single NVCs exceeding 100 µs in a nano-structured material.

2. Method

We start the diamond nanoslab fabrication using single crystal diamond plates (chemical vapor deposition growth, sourced from Element Six) with extremely low native nitrogen impurities (<5 ppb). The purity of this sample is confirmed using standard confocal microscopy and PL techniques (detailed below). The plate is then implanted with isotopically purified 15N ions at a
Figure 1. Fabrication of nanoslabs in ultra pure diamond. Single crystal diamond is implanted with $^{15}$N ions to a shallow depth of 10 nm (a). A thin film of Cr is deposited on the surface along with ZEP resist (b), which is subsequently patterned with a series of columns of 200 nm × 10 $\mu$m rectangles on the using electron beam lithography (c). Using chlorine ion etching, we transfer the mask from the resist into the Cr layer (d). The diamond surface is etched using an oxygen plasma, while the Cr is mostly resistant to the plasma. This results in the Cr mask pattern transferring to the diamond (e). Multiple layer angular redeposition of the Cr mask results in high aspect ratio nanoslabs. These slabs can be exfoliated from the diamond substrate and precisely positioned on another substrate (f).

fluence of $5 \times 10^9$ cm$^{-2}$ and accelerating energy of 6 keV, with an estimated mean implantation depth of 10 nm as simulated using Stopping Ranging of Ions in Matter (SRIM) software. The sample is then annealed for 2 h in high vacuum at 800 $^\circ$C to convert nitrogen defects to NV$^0$ and NV$^-$ color centers. A density of $\sim 2$ NV$^-$ centers $\mu$m$^{-2}$ is confirmed using confocal microscopy.

The fabrication process is summarized in figure 1. The implanted diamond plate is first cleaned and coated with an 80 nm layer of chromium (Cr) on the implanted side using an electron beam evaporator. We subsequently spin-coated ZEP-520A electron beam resist at 3000 rpm for 45 s, followed by baking on a hotplate at 180 $^\circ$C for 3 min to achieve a mask layer of 400 nm. Using electron beam lithography, we expose line array patterns with 200 nm width and 10 $\mu$m length at a dose of 300 $\mu$C cm$^{-2}$. After exposure, the sample was developed and the pattern was transferred to the Cr layer by reactive ion etch using a gas mixture of Cl$_2$ and O$_2$ (10 : 1). Employing this Cr hard mask, an O$_2$ inductively coupled plasma (ICP) etch process (3 mTorr, 100 W dc power and 1000 W ICP power, 60 $^\circ$C) was used to etch trenches in the diamond roughly 10 $\mu$m deep. The diamond surface was selectively protected by the Cr. This leaves nanoslabs with a large aspect ratio as high as 33, as shown in figure 2. We found the edge of the Cr mask eroded during the etch due to high plasma power, which resulted in a tapered cross-section on the slabs. To achieve straighter side walls, a two-stage etch was applied: after etching trenches of 2 $\mu$m depth, the sample was taken out and recoated from both sides with a layer of 20-nm-thick Cr at a 45$^\circ$ incident angle to protect the edges of the hard mask.

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Figure 2. (a) Scanning electron microscope image of nanoslabs attached to diamond surface. Note the smoothness of the wall surface on the 10 nm scale (inset). (b) Confocal microscope image of diamond wall surface. Imaging was done using a pump laser at 532 nm, collecting emission from 635 to 800 nm. Bright spots on the surface are single NVCs in the surface of the wall. Count rates are improved by almost 50% compared to emission from the bulk substrate due to effective index reduction at the nanoslab/oil interface.

The Cr mask was removed using a wet etch process. The diamonds were finally cleaned using a boiling mixture of perchloric, sulfuric and nitric acids (1:1:1 by volume).

3. Results and discussion

Given the density of defects within the implantation layer, it is likely that each slab contains more than one NVC. We begin by examining the slabs while still attached to the bulk substrate (figure 2). We confirm the presence of NVC using the standard confocal microscopy technique: the sample is illuminated with 532 nm laser light using a diffraction limited spot. The resulting fluorescence from the excited metastable triplet state (zero phonon line (ZPL) at 637 nm; phonon sideband (PSB) emission up to 800 nm) is focused into a single mode fiber and detected with Si avalanche photodiodes (figure 2). The bright spots within the confocal image are verified to be NVC using a combination of PL spectroscopy, which shows the characteristic emission spectrum (figure 4), and second order autocorrelation functions (figure 5). The autocorrelation function of the photon emission for a single center is confirmed using a fiber-based Hanbury–Brown–Twiss interferometer and measuring the arrival times of the photons. The dip at zero delay time, \( g_2(0) < 0.5 \), indicates emission from a single NVC. The bunching phenomenon, as seen in figure 5(c) when the \( g_2(0) \) value exceeds the steady-state rate of 25 Hz, is indicative of driving the NVC near optical saturation. We note that for a given alignment of the excitation and collection beam paths, the nanoslab emission rates show a factor of two increase from NVC in the bulk diamond (\( \sim 90 \) and \( \sim 45 \) kHz, respectively). We attribute this to the reduced effective index of refraction due to the nanopatterning of the slabs, similar to those reported in [19].

The novelty of nanoslabs for quantum information and sensing purposes cannot be fully realized with the slabs attached to the bulk substrate. For example, patterning of the slabs
into planar 2D photonic crystals suitable for enhancing light–matter interactions is most straightforward from top-down, lateral lithography. To this end, we seek to remove the slabs from the bulk and verify that they behave similarly on heterogeneous substrates. First, we exfoliate the slabs from the surface using a syringe to fracture the slabs near the base. The slabs are then transferred from the surface of the diamond substrate to a glass substrate using a polydimethylsiloxane (PDMS) stamping technique. Here, a 1 mm thick square of PDMS is pressed onto the diamond surface with loose slabs whereby the tacky PDMS conforms to the slabs. Upon lifting the PDMS, the slabs are transferred to the polymer. Slabs are subsequently transferred from the polymer to a plasma-cleaned glass substrate by pressing the PDMS square onto the glass and applying slight pressure.

Confocal microscopy of the slabs on this substrate, however, did not show any isolated NVC. The reason for this absence could be twofold. First, $N^{15}$ ion implantation occurs within a shallow region, roughly 10 nm below the diamond surface, with a straggle (spreading of implant depth) of 10 nm. Under these conditions, the majority of NVC would be near the edges of the detached walls, where scattering is maximal, and not in the center where reflection dominates. Second, the shape and depth of the slabs could be leading to total internal reflection of fluorescence of NVC emission when viewed from the planar face of the slab. Note that in the vertical, attached geometry, the pump beam excites the NVC on the narrow edge (∼300 nm thick) and fluorescence occurs through the same side, increasing the out coupling by minimizing the index mismatch within the mode. Recent studies [20] have shown this collection technique to be near optimal.

In order to understand the absence of NVCs from the detached slabs (figure 3), we implanted them in a planar position with another, higher dose of $N^{15}$ ions (90 keV, $1 \times 10^{11} N cm^{-2}$) and intended to create NVCs 100 nm from the surface. Furthermore, we annealed the sample under the same conditions cited above. Imaging the reimplanted slabs showed the characteristic NVC spots, which in turn demonstrated the photon anti-bunching indicative of quantum emitters and the NVC fluorescent spectrum (see figure 4). We note that despite a 20-fold increase in ion implantation, we observed a low density of NVC compared to the bulk crystal. One possible explanation is that in-plane wave guiding of the fluorescence emission does not couple normal to the slab surface. This suppressed the planar-collected signal, except around rare surface defects such as the one central in the nanoslab indicated in figure 4. However, the observation of NVC in the exfoliated nanoslabs confirms that these materials, despite several processing steps, can support the quantum system of interest. Quantification and optimization of nanoslab fluorescence emission is on-going. It may be possible to employ near-field scanning optical microscope techniques, as recently demonstrated with diamond nanocrystals [21], in order to capture edge emission from a detached slab.

In addition to purely spectral qualities, a single NVC within the nanoslabs also exhibits spin coherence properties consistent with bulk implanted diamond samples under the same conditions [22]. Optically detected magnetic resonance of the ground state spin triplet ($S = 1$) is measured by sweeping the microwave frequency for a fixed microwave and laser pump power and collecting the ZPL and PSB emission. The spin and orbital energy diagram are well described in the literature [23, 24] and are omitted here for brevity. Due to the spin-dependent shelving into a dark metastable state, the mean fluorescence rate drops when the microwave frequency is resonant with the zero field splitting of the NVC ($D_{gs} = 2.875$ GHz). In order to separate the degeneracy of the $|S_z| = 1$ states, we apply a magnetic field of 43.7 G along the NVC symmetry axis, as estimated by the splitting of 245 MHz (figure 5(A)). Note that
Figure 3. Images of exfoliated nanoslabs. Slabs are removed from the bulk diamond substrate by abrasion with a hypodermic syringe and transferred to a glass slide (a) using a PDMS stamping technique. The diamond slabs did not show characteristic bright spots, indicative of NVCs, at first. Repeated implantation and annealing caused an accumulation of NVCs inside the slabs. The sample is then scanned over the laser spot of a confocal microscope to obtain a fluorescence image (b). Bright spots show the characteristic dip (c, d) in a Hanbury–Brown–Twiss measurement, indicative of quantum emitters with metastable $^3E$ excited states, whose lifetime is proportional to the dip width. Photoluminescence spectra (not shown) have similar characteristics to center in the bulk, as in figure 4.

the optically detected magnetic resonance spectrum is only sensitive to the projection of the magnetic field along the NV axis to leading order for $B \ll 1000$ G. Effects of the transverse magnetic field can be estimated from other spin dynamics (described below).

Pulsed electron spin resonance measurements are preformed by tuning a microwave field on resonance with the $m_s = 0$–$1$ transition at 3.000 GHz and applying pulses of finite duration before looking at the transient fluorescence of the NVC immediately thereafter. Coherent Rabi oscillations between the two spin states show the presence of the hyperfine coupled $^{15}$N nuclear spin ($A_z \approx 3$ MHz, as seen in figure 5(B)) and also exhibit a decay due to a varying spin
environment or inhomogeneous microwave field. We fit the normalized fluorescence signal to the form: $S_{Rabi}(t) = \frac{1}{2} e^{-t/T_r} \left( \cos(\sqrt{\Omega^2 + \Delta^2} t) + \cos(\sqrt{\Omega^2 + (\Delta + A_z)^2} t) \right)$ and find a Rabi nutation rate of $\Omega = 19.2$ MHz, a marginal detuning ($< 1$ MHz), and a relaxation time of $T_r = 2.5 \mu s$ (figure 6, inset). (This modest relaxation time is most likely set by amplitude noise of the microwave amplifier [25].) By setting the $\pi$-pulse time to the first minimum of the Rabi oscillation, we can perform a spin echo measurement, where the long-time decay envelope reveals the coherence of the single electronic spin interacting with a magnetic spin bath. We observe the characteristic ‘revivals’ of the coherence [23] of a single NVC. (The emission from a single center is evidenced by the $g_2(0)$ autocorrelation function dipping below half the long-time rate of 25 Hz in figure 5(C).) We fit each revival peak to a Gaussian envelope and record the time for which each revival is maximal and find a revival period of $17.8 \pm 1.4 \mu s$ (figure 6). The $^{13}$C nuclear spins responsible for this behavior thus precess at a rate $\omega_C = 2\pi \times 1071$ Hz G, which corresponds to a net applied field of $52.4 \pm 4.0$ G. This reveals that the applied magnetic field is roughly $33.4^\circ$ tilted from the NVC axis.

The entire spin echo envelope (normalized) decays following $S(\tau)_{env} = e^{(\omega_C T_r)}$ with $T_2$ of $92.6 \mu s$. However, typical samples exhibiting modulations from the nuclear spin bath exhibit echo envelopes of the form $S(\tau) = S_0 e^{-(2\tau/T_2)^3}$ due to the non-refocused dipole–dipole
Figure 5. Optically detected magnetic resonance. (A) Optically detected magnetic resonance from NVC when pumped continuously with microwave and optical excitation. The peaks occur at 2.755 GHz (♦) and 3.000 GHz (◇) with an applied static magnetic field, indicating a field of 43.7 G along the NV axis. (B) Closer inspection under optimal excitation powers of the lower resonance peak reveals the hyperfine doublet due to coupling of the electronic triplet with the implanted $^{15}$N ion ($I = 1/2$). Due to the finite coherence time of this center, we are limited from full resolution of the line. Electron spin echo modulation (ESEEM) data (not shown) reveals the expected hyperfine at $\sim$3 MHz. (C) Second order autocorrelation function ($g_2(\tau)$) of the emitted photons as measured in a Hanbury–Brown–Twiss configuration. Note that the $g_2(0)$ value falls well below $1/2 g_2(\tau \to \infty)$, indicating a single emitter.

interactions of the nuclear spin bath [23]. We believe that in our case, we observe two distinct baths: $^{13}$C nuclear spins and another electron spin bath. The nuclear spin bath generates the collapses and revivals of the echo signal at the carbon Larmor rate, but the envelope is determined by other fluctuating magnetic fields. Following the standard semi-classical description of spin relaxation [26], we define $\tau_c$ as the correlation time of the fluctuating fields from this second source. In the limit that $\tau \gg \tau_c$, the envelope function is a single exponential $S(\tau) \propto e^{\tau/\tau_c}$ with $T_2^{-1} = \langle \omega_n^2 \rangle \tau_c$, where $\langle \omega_n^2 \rangle^{1/2}$ is the root-mean squared (rms) value of the fluctuating field. In estimating $\tau_c$ to be at most 1 $\mu$s, a small collection of electron spins
Figure 6. Electron spin echo revivals and Rabi oscillations of a single NVC in an attached diamond slab. The Rabi oscillations (inset) show coherent oscillations under an applied resonant microwave pulse. The envelope fits a single decaying exponential with a time constant of 2.5 µs. Given the Rabi nutation rate, we tune \( \pi \) and \( \pi/2 \) rotations to perform a Hahn echo experiment. We see the typical collapses and revivals of coherence associated with the NVC coupling to \(^{13}\text{C}\) spin bath, separated by twice the carbon Larmor frequency. From this we estimate \( B_\perp = 28.8 \) G. ESEEMs due to a strongly coupled \(^{15}\text{N}\) nuclear spin cause oscillations within the revival peak. The envelope encapsulating the echo revivals is fit to the model of an interacting spin bath: \( S(\tau)_{\text{env}} = e^{(-2\tau/T_2)} \) (green), yields a \( T_2 \) of 92.6 µs.

10–30 nm from the NVC could easily produce the rms fields on the order of 100 kHz in order to produce the observed \( T_2 \) value. As the implanted NVCs are < 100 nm from the surface that has undergone much reactive ion processing, it is entirely plausible that dangling bonds or trapped spin states on the surface of the diamond slab are the source of these fast fluctuations. This hypothesis deserves further study and can be tested along the lines of the spin echo double resonance experiments recently reported on NVCs [27], whereby one can actively decouple and recouple the bath spins from the NVC of interest.

When reporting a magnetic field in Hz, we implicitly use the relationship \( \omega_0 = 2\pi v_0 = \gamma_{\text{NV}} b_{\text{rand}} \), where \( \gamma_{\text{NV}} \) is the gyromagnetic ratio of the NV electron spin, and \( b_{\text{rand}} \) is the stochastic field arising from the fast spin bath.

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4. Conclusion and outlook

We have shown that nanoscale structures fabricated in high-purity single crystal diamond by electron beam lithography and oxygen plasma dry etching can exhibit long coherence times, approaching 100 µs, comparable to the coherence times seen in the host diamond material. Moreover, we have shown that nanoslabs of diamond can be removed and positioned on glass substrates for further processing, such as implantation or focused ion beam milling. The electron spin coherence time may be enhanced into the ms range using isotopically purified (12C) diamond [5] and with proper alignment of the external magnetic field. We note that the ~10 nm proximity to the surface indicates that structures on a much smaller length scale should also allow for a similarly long coherence time. This is an important finding, giving the tolerances needed in placement of the NVC with respect to photonic crystal nanocavities (PCNs) for simulating atom–photon interactions in the solid state. Furthermore, the diamond slab presented here is promising for post-processing into various structures such as PCNs to enhance optical transitions of the NV as quantum interfaces to NV spin qubits, magnetic or electric field sensors [28, 29] or spin-based frequency standards [30].

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