Single photon emission and single spin coherence of a nitrogen vacancy centre encapsulated in silicon nitride

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Abstract

Finding the right material platform for engineering efficient photonic interfaces to solid state emitters has been a long-standing bottleneck for scaling up solid state quantum systems. In this work, we demonstrate that nitrogen-rich silicon nitride, with its low background auto-fluorescence at visible wavelengths, is a viable quantum photonics platform by showing that nitrogen vacancy centres embedded in nanodiamonds preserve both their quantum optical and spin properties post-encapsulation. Given the variety of high-performance photonic components already demonstrated in silicon nitride, our work opens up a new avenue for building integrated photonic circuits using solid state emitters.
Keywords

Colour centre, nanodiamond, silicon nitride, integrated quantum photonics, solid state quantum emitters, atom-like systems

Introduction

The ability to isolate and manipulate single atom-like systems in the solid state has heralded a new age of quantum engineering. Given the increased decoherence that these atom-like systems exhibit in comparison to real atoms, the primary motivation for sustained research interest arises mainly from the prospect of building scalable platforms, with efficient photonic interfaces using nanofabrication. As the semiconductor industry has repeatedly shown, integrated chip-scale platforms can vastly exceed the complexity and connectivity of discrete devices. Building on the progress in silicon photonics, manipulation of quantum states of light in chip-scale platforms has also grown in scale and complexity in the last decade. Adding solid state atom-like systems to these integrated quantum photonic platforms will help us realise deterministic single photon generators and on-chip quantum memories, two crucial components for photonic quantum technologies.

A variety of candidate atom-like platforms have been pursued for integration with on-chip waveguides. These range from InAs quantum dots in GaAs waveguides, colour centres in diamond and silicon carbide, 2D materials, dye molecules and rare earth ions in glass. Amongst these platforms the nitrogen vacancy (NV) centre in diamond is unique in possessing a long-lived (~ µs) spin coherence that can be optically manipulated and read-out at room temperature. Demonstrations of nuclear spin entanglement to error correct the NV centre spin and entanglement between distant NV centres show the possibility of engineering complex quantum information processing platforms around the NV centre. Pioneering experiments have relied on monolithic diamond samples with photonic interfaces provided by bulk optics, with limited scope for scalability. To realise the full potential of the
NV centre in quantum information processing, it is apparent that integrating NV centres with on-chip photonic waveguides and cavities is essential.

Diamond, which serves as the host for NV centres, is notoriously difficult to etch on account of its inert, rigid carbon lattice. While there have been demonstrations of nanophotonic devices in bulk diamond,\textsuperscript{12,13} using reactive ion etching in an oxygen plasma, it is difficult to foresee building large scale quantum circuits using this platform. An alternative approach relies on using NV centres located in nanodiamonds (size 10-20 nm) which are formed by milling bulk diamond. By embedding these nanodiamonds in high-index (relative to fuse silica) dielectric films, it would be possible to realise a photonic waveguiding platform. However, the dielectric material would need to satisfy the following criteria for enabling quantum information with NV centres: 1) Low absorption in the visible wavelength range 2) Low background fluorescence to preserve the NV single photon statistics 3) Preserve the intrinsic radiative quantum efficiency 4) Minimise any added dephasing effects on the NV electron spin and 5) Have established fabrication procedures for building photonic integrated circuits. Criteria 1-4 are essential for building a quantum photonics platform, while 5 is key for large scale quantum integration (LSQI).

The three most viable dielectric thin film platforms for building quantum photonic circuits encapsulating NV centres hosted in nanodiamonds are: Titania (TiO\textsubscript{2}),\textsuperscript{14} Aluminum nitride (AlN)\textsuperscript{15} and silicon nitride (Si\textsubscript{3}N\textsubscript{4}). Amongst these, Si\textsubscript{3}N\textsubscript{4} is the most attractive, given the impressive performance already achieved in integrated photonics, such as broadband frequency combs\textsuperscript{16} and integrated frequency converters,\textsuperscript{17} and being mature in terms of foundry compatibility.\textsuperscript{18,19} Unfortunately, stoichiometric silicon nitride (Si\textsubscript{3}N\textsubscript{4}) has broad auto-fluorescence around the zero phonon line of the NV centre (637 nm), which adds background noise and makes it challenging to observe the quantum signature (antibunching) of the NV centre single photon emission.\textsuperscript{20} Therefore, for silicon nitride to be a viable quantum photonics platform compatible with NV centres, this background fluorescence needs to be suppressed. In this paper we build on the idea of using non-stoichiometric films\textsuperscript{21,22}.
to minimise the background fluorescence. We show that nitrogen-rich amorphous silicon nitride films ($\alpha$-SiN$_x$) can serve as a viable photonic platform for interfacing with NV centres located in nanodiamonds by demonstrating single photon emission and spin coherence in nanodiamonds that are capped with 100 nm of $\alpha$-SiN$_x$. In contrast to probing the film for encapsulated emitters, here, we characterise the optical and spin properties of the same single NV centre(s) (identified using fiduciary markers) before and after film deposition allowing us to unambiguously demonstrate that the NV centre survives the plasma deposition process and that nitrogen-rich $\alpha$-SiN$_x$ provides a quantum photonics platform compatible with single atom-like systems.

**Low fluorescence $\alpha$-SiN$_x$ film deposition and characterisation**

Increasing the nitrogen content of $\alpha$-SiN$_x$ films helps reduce the background photoluminescence (PL) emission. The key question to address is whether the background PL can be reduced sufficiently to clearly observe single photon emission statistics from an NV centre, encapsulated with $\alpha$-SiN$_x$. In addition, given the prevalence of nitrogen (N) atoms in $\alpha$-SiN$_x$, one needs to measure the spin properties of the NV centre as well to ensure that the electron spin coherence is not significantly reduced after film encapsulation. To study the effect of $\alpha$-SiN$_x$ on encapsulated NV centres, we start by characterising the background PL emission from $\alpha$-SiN$_x$ with varying nitrogen content.

To achieve this, 300 nm amorphous silicon nitride films ($\alpha$-SiN$_x$) were deposited on fused silica substrates using plasma enhanced chemical vapour deposition (PECVD) with a gas mixture of ammonia, silane and nitrogen (NH$_3$/SiH$_4$/N$_2$) and alternating the plasma excitation frequency between high frequency (13.56 MHz) and low frequency (50 kHz) to minimise the internal mechanical stress of the material. The fused silica substrates were cleaned with acetone, isopropanol and deionised water and exposed to an oxygen plasma
Figure 1: a) Refractive index (n) and extinction coefficient (k) of α-SiN\textsubscript{x} films with increasing ammonia (NH\textsubscript{3}) to silane (SiH\textsubscript{4}) gas ratios (r) deposited using plasma enhanced chemical vapour deposition (PECVD). The values are extracted from a Cauchy model fit to the ellipsometry data b) Integrated photoluminescence (PL) spectrum for the samples prepared in (a) plotted as a function of gas ratio. The orange and green lines represent the PL of a stoichiometric Si\textsubscript{3}N\textsubscript{4} film deposited using low pressure chemical vapour deposition (LPCVD) and background noise measured using a bare fused silica substrate respectively. The insets show representative PL spectra (blue) for α-SiN\textsubscript{x} samples with low (left) and high nitrogen content (right). Here, the background signal from bare fused silica is shown in green for reference. The vertical error bars were obtained by averaging integrated values for repeated measurements. The horizontal error bars correspond to the uncertainty in the mass flow controller.
to remove organic contaminations before film deposition. The $\alpha$-SiN$_x$ films were deposited varying the NH$_3$ to SiH$_4$ gas flow ratio $r = [\text{NH}_3]/[\text{SiH}_4]$ from 0.6 to 3.0 while keeping the total gas flow constant as well as the chamber pressure at 1.0 Torr and the substrate temperature fixed at 300° C.

After deposition, the refractive index $(n + ik)$ of the deposited films was extracted from a Cauchy model fit to the ellipsometry data, plotted in Fig 1(a). As shown in Fig 1(a), the refractive index of the films decreases with increasing nitrogen content, in good agreement with previous work. The refractive index determines the mode area for a guided mode in the $\alpha$-SiN$_x$ film. Having a higher refractive index allows for tighter modal confinement and larger field overlap with a dipole (in this case an NV centre) located in the centre of the waveguide. This ensures that a large fraction of the emission is funneled into single-mode waveguide.

To measure the background PL spectrum of the different $\alpha$-SiN$_x$ films, we use a standard confocal microscope setup (shown in Fig 2(a)). In Fig 2(b), we plot the integrated PL spectra for different films as a function of gas ratio. For reference, we also plot the integrated PL of stoichiometric Si$_3$N$_4$ deposited using LPCVD and the PL (background noise) from a bare fused silica substrate. For comparison, the integrated PL spectrum obtained from the fuse silica substrate (green) and the stoichiometric Si$_3$N$_4$ films on Si (orange) are also shown. Two representative spectra, corresponding to low and high nitrogen content are shown in the figure inset. As Fig 2(b) demonstrates, the background PL emission can be reduced by almost two orders of magnitude, whereas the refractive index of the films drops by less than 5%, making it feasible for $\alpha$-SiN$_x$ to be used as a quantum photonics platform.
Figure 2: a) Confocal microscope setup used for characterising the optical and spin properties of NV centres (red path indicates fluorescence). b) Large area confocal fluorescence scan of a bare fused silica substrate coated with NV centres. Pixel colour indicates the strength of the PL signal (cf. colourbar). The large bright spots, shown with square boxes, correspond to fiduciary markers (shown in the inset) used to identify the positions of the NV centres (indicated with hexagons). c) The confocal scan of the same area after the sample has been coated with 100 nm of low-fluorescence nitride. The positions of the markers and the NV centres characterised in this work are again indicated. A high-resolution scan around one of the capped NVs is shown in the inset.
Characterisation of NV centres before and after silicon nitride encapsulation

Rather than blindly probing the encapsulated film for single emitters, we borrow techniques from localisation microscopy to record both the optical and spin properties of the same NV centre before and after nitride film encapsulation. Localisation helps us in two regards: by returning to a pre-characterised NV centre as opposed to measuring many random NVs, we can better understand the effects of film encapsulation. It also provides unambiguous proof that the NV centres isolated in nanodiamonds can survive nanofabrication processes, in particular, exposure to plasma chemical vapour deposition as direct bombardment of NV centres with ions in sub-micron volumes is known to cause their destruction. To achieve localisation, we use a set of fiduciary markers (crosses) fabricated using electron-beam lithography. By performing a scanning confocal map of the sample, the spatial coordinates of the PL can be registered with respect to the fiduciary markers. A representative scan, on a bare fused silica substrate spin-coated with nanodiamonds, is shown in Fig 2 (b). High purity nanodiamonds are used with low nitrogen content to ensure a high probability of containing single NVs. The fiduciary markers are indicated by rectangular boxes. Each cell, labelled with four fiduciary markers spaced 20 \( \mu m \) apart, is assigned a unique reference code to allow instant localisation. An single NV centre would appear on this map as a Gaussian spot, with size determined by the point-spread function of the microscope. To confirm the identity of a single NV centre and avoid NV ensembles and other point defects in the nitride film, an intensity auto-correlation \( g^{(2)}(\tau) \) is performed at selected locations in the confocal map. To characterise the sample, this process is repeated by stitching together multiple confocal scans. This is realised by positioning a piezo stage on top of a 3-axis stepper motor. The piezo stage provides the spatial resolution necessary for performing localisation, whereas the stepper motor driven stage permits the large sample travel necessary to map a sample with \( \sim \) mm dimension. To understand the effect of the \( \alpha\)-SiN\(_x\) films on the optical and spin
properties of the nanodiamonds, we pick four representative NV centres (A-D, labelled by hexagons in Fig 2(b)).

We encapsulate the sample with a low fluorescence nitride film ($r = 3$ in Fig 1). The confocal PL map of the same area on the capped film is shown in Fig 2 (c). For ease of comparison, the location of the fiduciary markers and the four pre-characterised single NV centres (A-D) are shown. It can be clearly seen, from the four bright spots, that the NV centre fluorescence is preserved after film encapsulation. The positions of the NV centres are also identical with respect to the markers pre- and post- film deposition, which confirms that the nanodiamonds have not been disturbed during the process of NV pre-characterisation, moving the sample from the lab to the cleanroom, film deposition and post-deposition characterisation back in the lab. A zoomed-in scan of the emission of NV D after silicon nitride deposition is shown in the inset. The nitrogen-rich silicon nitride films show observable bleaching effects. We do not fully understand the source of this, but currently believe it is due to the presence of unpassivated charge traps in the amorphous nitride matrix, which are quenched by laser exposure.

To quantify the effects of the nitride film on NV spin and optical coherence, we study the properties of isolated NV centres before and after film capping. We start by characterising the effect on optical properties. Fig 3 (a) plots the emitted photon statistics of NV D before and after capping with silicon nitride. To characterise the photon statistics, the NV centre is excited by a continuous wave (CW) 532 nm pump laser and the pump-filtered emission is collected into a 50:50 fibre coupler and split between two single photon-counting avalanche diodes (SPADs). By measuring the intensity autocorrelation between the two detectors, $g^{(2)}(\tau) = \langle I(t)I(t+\tau)\rangle/\langle I(t)\rangle^2$, we can demonstrate that the emission is anti-bunched ($g^{(2)}(0) < 1$). The photon statistics, displayed in Fig 3 (a) are not corrected for dark counts in the detectors and the fluorescence of optics in the beam path. We fit an uncorrected $g^{(2)}(0) = 0.22$ for NV D measured on the bare fused silica substrate. Following encapsulation by a 100 nm low fluorescence silicon nitride film ($r = 3$ in Fig 1), we repeat
Figure 3: Optical characterisation of a representative single NV centre (NV D) measured before and after encapsulation with a low-fluorescence nitride film. a) Intensity autocorrelation ($g^{(2)}(\tau)$) measurements showing a $g^{(2)}(0)$ less than 0.5 i) before encapsulation is preserved ii) after encapsulation. b) Fluorescence lifetime measurements showing that the fluorescence lifetime ($\tau_2$) is reduced by a factor of 3, from i) 19 ns to ii) 7 ns attributed to radiative rate enhancement by coupling to slab modes in the nitride film and increasing the refractive index around the NV centre. c) Diagram of the substrate i) before and ii) after film encapsulation.

the same measurement on NV D and find a $g^{(2)}(0) = 0.43$. Although the $g^{(2)}(0)$ measured here is slightly larger, the anti-bunched emission from a capped NV is clearly demonstrated. In addition, the $g^{(2)}(0) < 0.5$, which is the threshold for single emitter emission. To build a viable quantum photonics platform in silicon nitride, it is key that single emitters encapsulated in dielectric films can be identified and their emission statistics quantified. Our result provides a proof-of-principle demonstration of this idea.

In addition to measuring single photon emission, we would like to understand the effect of the encapsulated film on the fluorescence lifetime of the NV centre. Amorphous dielectric films generally harbour traps and surface states which affect the energy levels of near-surface NV centres. To measure the lifetime, a 532 nm pulsed laser with repetition rate of 10 MHz was used to excite the NV and the NV emission as a function of time was recorded using a SPAD connected to a time-correlated single photon counting system (PicoHarp), which was synchronised to the pulsed laser. In Fig 3 (b), the excited state fluorescence lifetime
of the emitter located on a bare fused silica substrate is shown. The emission is fit by a bi-exponential function, \( I(t) = A \exp(-t/\tau_1) + B \exp(-t/\tau_2) \), with a rapid \( \tau_1 = 1.17 \text{ ns} \) decay from the background attributed to Rayleigh and Raman scattering, followed by the slower NV fluorescence. The bare fluorescence lifetime is measured to be \( \tau_2 = 19.35 \text{ ns} \), which is characteristic of NVs in nanodiamonds. After the NV centre is encapsulated in silicon nitride, the fluorescence lifetime decreased by a factor of 3 to \( \tau_2 = 6.84 \text{ ns} \). This is expected as the higher refractive index surrounding the NV centre acts to increase the bare spontaneous emission rate by funneling the emission into slab waveguide modes supported by the nitride film. To confirm this hypothesis, we need to map the dipole orientation of the NV centre and calculate the mode overlap with the guided modes supported by the slab waveguide.

As discussed in the introduction, what makes the NV centre attractive as a quantum information platform is its use as a spin-photon interface via cycling transitions and electron spin dependent fluorescence. Therefore, it becomes critical to quantify the effects of the film encapsulation on the spin properties of the electron spin associated with an individual NV centre. To characterise the spin coherence, we perform a free induction decay measurement and extract the spin dephasing time \( \tau_{2*} \). The pulse sequence used to measure \( \tau_{2*} \) is shown in Fig 4 (b). A 532 nm laser is used to spin polarise the NV centre, initialising it in its \( |m_s=0\rangle \) ground state. By applying a pulsed microwave signal tuned to one of the spin transitions (in this case \( |m_s=0\rangle \) to \( |m_s=-1\rangle \)), we can couple the two spin states and resonantly transfer population between them by varying the duration of the microwave pulse length \( \tau \). The spin state can be read out by monitoring the fluorescence excited by a subsequent 3 \( \mu \text{s} \) laser pulse. Signal in the first 300 ns of the readout is integrated and normalised with respect to the average counts in the final 300 ns part of the laser pulse to recover the intensity contrast plotted. The NV centre located on a bare fused silica slide has a spin decay of \( \tau_{2*} = 0.45 \mu \text{s} \), consistent with values reported for nanodiamonds in the literature. After capping with the silicon nitride, we can observe coherent Rabi oscillations and extract
Figure 4: Free induction decay of a representative NV centre (NV D) a) before and b) after encapsulation with the silicon nitride film. The fitted Rabi decay indicates the NV centre in nanodiamond maintains electron spin coherence in the nitride-rich silicon nitride environment. The increased oscillation frequency suggests the film enhances the received microwave power. c) The diagram indicates the laser and microwave pulse sequence used to carry out this spin coherence measurement.
a spin decay time of $\tau_2^* = 0.29 \mu s$. Interestingly, the Rabi frequency ($\Omega$) of the NV centre increases by a factor of 1.5 after it is capped in nitride for the same applied microwave source power. We believe this is because the higher dielectric constant of nitride, compared to air in the bare fused silica case, increases the effective microwave field at the location of the nanodiamond. We need additional measurements to quantify this effect. Although in this work, we were experimentally limited to slow Rabi driving, we can apply dynamical decoupling pulse sequences to increase the effective spin lifetime,\textsuperscript{30} a key technique for quantum memories implemented with NV centres.

**Conclusions**

We have conclusively demonstrated that nitrogen-rich silicon nitride serves as a viable quantum photonic platform for building scalable spin-photon interfaces around NV centres in nanodiamonds. Our experiments demonstrate that both the optical and spin properties of individual NV centres are preserved after film encapsulation. Given the variety of high-performance photonic components already demonstrated in silicon nitride, our work opens up a new avenue for building efficient visible photonic interfaces to solid state emitters. While we have focused on the NV centre in diamond, the work can be easily extended to other quantum photonic platforms under investigation in the visible regime, including perovskite quantum dots\textsuperscript{31} and 2D transition metal dichalcogenides.\textsuperscript{32}

**Methods**

Fused silica wafers were sourced from MicroChemicals GmbH. Metal markers were patterned using a Raith Voyager and 50 nm gold lift-off in a thermal evaporator (Edwards). Nanodiamonds were sourced from Nabond and suspended in ethanol before syringe filtered and dropcast on the sample. $\alpha$-SiN$_x$ films were deposited onto solvent cleaned fused silica wafer
pieces using an Oxford Instruments PlasmaPro 100 PECVD chamber. Ellipsometry data was taken with a J. A. Woollam RC2 and fitted with a Cauchy model to extract the optical constants of the films. Photoluminescence spectra was recorded using Andor spectrometer Shamrock 300.

The NV centres were characterised using a home-built microscope 532 nm LaserQuantum GEM laser through a NA = 0.9 objective lens. An Excelitas SPCM AQRH 13-FC SPAD with 30 dark counts per second was used for the confocal images. Photon counting was performed using a UQDevices timetagger. The sample was manipulated using a closed loop Thorlabs NanoMax piezo stage and Thorlabs KCube driven stepper motors. Software was written in C++ to extract and synchronise time tags to piezo positions and parsed to a modified Qudi codebase. Autocorrelation measurements were performed by splitting the optical light through a 50:50 fibre coupler to a second Excelitas SPAD with 200 dark counts per second and recorded with a Picoharp 300. Fluorescence lifetime measurements were taken by adding a PicoQuant LDH P-FA 530B pulsed laser in to the excitation beam path, synchronised with a Picoharp 300. CW ODMR established the electron spin splitting using a Rohde and Schwarz SMB100A microwave source driving a custom loop antenna PCB. A small magnetic field using 3 perpendicular electromagnets was set to ensure spin states were well resolved. Rabi oscillations were programmed using a SpinCore PulseBlasterESR-PRO driving a AA optoelectronic AOM in the beam path and a microwave switch (Mini-Circuits ZASWA-2-50DR+) in the microwave path.

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