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Invited Paper

680–890 nm spectral range of Nickel-Nitrogen and Nickel-Silicon complex single centres in diamond

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ABSTRACT

Fluorescent emitters in nanodiamonds are considered to be a valuable resource for emerging fields such as quantum communication, quantum photonics and biological imaging. In this paper we report a wide range of narrow bandwidth spectral emission lines arising from different color centers at room temperature. We associate the zero phonon lines, observed using confocal microscopy, with previously identified nickel-related centers in high pressure high temperature diamond, with a Si/Ni complex and silicon vacancy defects. In particular we show the first observation of a 850 nm emission in diamond with single photon signature, indicating the potential for diamond to harbor infrared single-photon sources. Index Terms—diamond defects, single-photon emission, confocal microscopy.

Keywords: confocal microscopy, defects in diamond, single-photon source

1. INTRODUCTION

Sources of single photons are relevant across many applications, as they can be used for quantum information processing, permitting long-range manipulation of quantum states for tasks such as quantum communication or distributed quantum computation.[1] Moreover, single-photon sources and in general quantum states of light have been considered to be a useful resource for establishing novel measurement methods in quantum metrology.[2,3] Defects in nanodiamonds (NDs) also offer the possibility of a remarkably efficient implementation of several methods in nanoscopy[4] and they can serve as bio-markers.[5] For a review of the use of diamond defects in super-resolution imaging see Castelletto *et al.*[6] Color centers in diamond are also prominent candidates to generate and manipulate quantum states of light, as they are in general photostable solid-state source of single photons at room temperature. For quantum information and quantum metrology applications the stability of defects in diamond constitute the main advantage over other systems. For biological applications the bio-compatibility of diamond is prominent and the emitters presented here are ideal not just for imaging in cells, but also for tissue.

Apart from the extensively studied optical center based on a substitutional nitrogen atom and a vacancy in the diamond matrix (NV center),[7,8] several other complexes such as silicon vacancy,[9–12] previously unexplored single centers (Cr-related),[13,14] a green emitter,[15] H3 and H4 defects[16] and the TR12 centre (interstitial carbon defect)[17] have been demonstrated to possess single-photon emission. In applications such as quantum information, metrology, nanoscopy and bio-marking, narrow bandwidth spectral emissions, high brightness and near-infrared emission are the properties underpinning ideal single-photon sources or more in general ideal fluorescent beads.

Nickel defects in diamond have been studied for many years (see Collins for a review[18]), particularly in HPHT diamonds grown using a Fe-Ni-C solvent/catalyst system at 1750 K.[19] From these investigations, several centers have been observed by Raman spectroscopy and electron paramagnetic resonance (EPR) in highly-doped diamond and their structures in some cases have been conclusively determined by EPR.[20] Nickel-nitrogen complexes are of particular interest to the quantum-communication community, since the NE8 center (a nickel atom surrounded by four nitrogen atoms) emits at 793.5 nm without phonon-interaction induced spectral broadening.[18,20] The first demonstration of this defect as a single-photon emitter utilized a natural diamond sample.[21] Later NE8 was created in thin diamond layers grown by chemical vapor deposition (CVD), by use of a nickel/diamond nano-particle slurry which was then seeded on top of a fused silica substrate.[22]

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These experiments triggered the search for superior single-photon sources compared to NV, for implementing quantum optics experiments and quantum cryptography.[23] Further attempts to create the center in NDs and in bulk diamond in a controllable manner were based on ion implantation of nickel into nitrogen-rich HPHT diamond, with subsequent annealing up to 1600°C, and by nickel implantation into CVD grown-nanodiamonds or on a silica substrate where the nanodiamonds were grown afterwards.[24] In this last case plasma-assisted diffusion of nickel from the substrate into the diamond nano-particle seeds the CVD diamond growth. However, although some nickel-related centers were captured as single-photon emitters from CVD sample growth, no single NE8 centers were observed.[25] Nickel and nitrogen ion irradiation of low nitrogen single crystal diamond[26] failed in demonstrating a controllable fabrication of nitrogen-nickel complexes and no evidence of NE8 at the single level was reported.

NE8 and other nickel-related centers were observed using cathodoluminescence in nickel doped single crystal diamond layers grown by microwave-plasma enhanced CVD, demonstrating the potential for a viable route to create NE8 centers in single crystal diamond.[27] Also in this case however single-photon emission was not obtained. Different doping methods to incorporate nickel-nitrogen complexes into nano and single crystalline CVD diamond films based on three different doping sources (gaseous nickelocene, nickel powder and nickel wire) have been used to date. From the examined experiments[22,27] reported in the literature the most promising technique to create NE8 and other related nickel-nitrogen complexes during CVD growth down to the single defect creation, seems to be the direct exposure of nickel wire or powder to the microwave plasma.

It is apparent from these results that CVD offers an excellent basis on which the production and further studies of single color centers can be made. One difficulty facing all potential implementations is the limited understanding and repeatability of producing material with the desired color center for a particular application. Beyond the initial requirement of diamond containing a color center, it is also necessary for the concentration to be low enough for optical excitation of one and only one center in a given pump pulse. Here we report data from a suite of color centers incorporated in diamond films grown by CVD, including previously identified centers such as NE8, NE4 and silicon-vacancy (SiV) centers, as well as several additional defects which are likely nickel-related. Crucially some of these centers are shown to emit single photons. In this paper we show that in diamond grown with nickel powder by CVD microwave plasma on a silica substrate, other single-photon emission with narrow zero phonon lines (ZPLs) with peak wavelength from 711 nm until 900 nm, can be found and associated mostly to nickel-related centers. All these different ZPLs are present in the same sample.

The results summarized here therefore indicate that this production method represents a possible route for the mass-production of these centers in NDs for microscopy and metrology, and it could also be a useful resource for creating a wide range of single-photon emitting ZPLs in the red,near-infrared and in the infrared.

2. EXPERIMENTAL METHODS: CHEMICAL VAPOUR DEPOSITION FOR CREATING NICKEL-RELATED DEFECTS

The sample was created following the methodology outlined in ref. 22 and was characterized using a home-made confocal microscope. For the sake of summary, diamond films were grown on 131 cm^2 fused silica substrates using a 1.2 kW microwave plasma CVD reactor, with the chamber pressure maintained at 30 Torr in a 0.7% CH_4 in H_2 gas mixture. The substrate temperature was a constant 700°C during the growth period. Prior to growth, the fused silica substrates were seeded by exposure to a slurry consisting of nickel and diamond powder (< 10 nm) in an ultrasonic bath. Diamond films of thicknesses of $\sim 4\,\mu$m were then grown at a rate of $\sim 1\,\mu$m/h.

The centers were excited using a ~ 50 mW continuous-wave laser operating at 682 nm, or a laser at 665 nm with $\sim 260\,\mu$W power incident on the sample. The laser was focused onto the sample using a $100\times$ infinity-corrected oil immersion objective lens with a numerical aperture of 1.3 (Olympus) and luminescence was collected confocally through a pinhole. Several band-pass filters, at 740 ± 10 nm, 760 ± 10 nm, 780 ± 10 nm and 857 ± 10 nm, as well as a long-pass 830 nm filter, were use to identify specific spectral emissions and a spectrometer (Acton) with a cooled CCD (Princeton Instruments) was used to futher characterize the luminescence.

A Hanbury Brown and Twiss (HBT) interferometer with single-photon-sensitive avalanche photodiodes (Perkin Elmer SPCM-AQR-14) was used to measure the photon statistics. Photon counting and correlation was carried out using a time-correlated single-photon-counting (TCSPC) module (PicoHarp 300, PicoQuant

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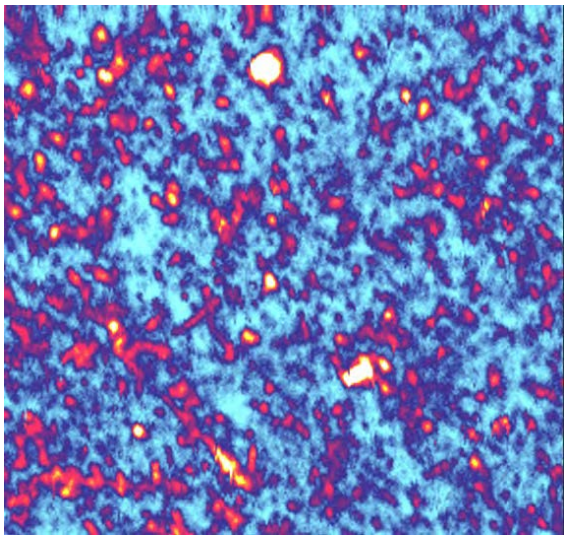

Figure 1. Confocal image of $30 \times 30\,\mu$m area of the sample detected using a 850 nm bandpass filter

GmbH). Diamond defects hosting single or more centers were identified using measurement of the second order intensity correlation function $g^{(2)}(\tau) =< I(t+\tau)I(t) > / < I(t)^2 >$, and the spectra measurement, to select only a single band emission lines. The normalized second order temporal correlation function $g^{(2)}(\tau)$ at zero delay time gives an indication of the number of emitters in a particular focal spot, since it represents the probability of detecting two simultaneous photons (where $\tau = 0$) normalized by the probability of detecting two photons at once for a poissonian photon source: an "antibunching" dip in $g^{(2)}(\tau)$ indicates sub-Poissonian statistics of the emitted photons and reveals the presence of a single quantum-system which cannot simultaneously emit two photons. In several cases we observed $g^{(2)}(0) < 0.5$, whilst the sample contained mostly particles with multiple centers $g^{(2)}(0) > 0.5$ or with high background.

3. IDENTIFICATION OF SINGLE-PHOTON EMISSIONS

We investigated many emitters (>100) in the sample. By using different 10 nm bandpass filters, we identified bright spots most of the time associated to a single emission line on the confocal map. For more details see ref. 28. In Fig. 1 we show as example a confocal map used to identify emission around 850 nm. Of the characterized bright spots, approximately 38% corresponded to single narrow-emission lines, while others displayed broader emission of multiple emission lines in PL. Approximately 12–13% could be associated to single-photon emitters. In most cases the ZPLs associated with the centers show an emission bandwidth of about 1.5 nm (fitted using a Lorentzian lineshape), whilst in a few cases the resulting emission is broader with a full width at half maximum (FWHM) of ~ 10 nm. The statistics of ZPLs including broader emission lines is shown in Fig. 2.

The dominant ZPLs occur in the 718 nm, 754 nm and 800 nm region. In Fig. 3, we show examples of some of the ZPLs observed in the 800 nm region which possessed single-photon characteristics, which extends up to emissions at 850 nm. An interesting emission line, which corresponds to the first IR emission in diamond, showing single-photon emission, is reported in Fig. 4. Few emitters were found in two $100 \times 100\,\mu$m scans, indicating that these emitters are far less frequent than the red or near-infrared emission lines. In addition they displayed a higher degree of photoluminescence instability or blinking.

Here we focus on identifying some of the ZPLs exhibiting single-photon emission. To shed light on the wide variety of emission lines observed in the synthesized sample, we refer to previous investigations of HPHT synthetic diamond. HPHT diamond is usually grown from a melt such as Fe-Ni-C or Fe-Co-C, which acts as a solvent-catalyst for the diamond growth.

The photoluminescence (PL) spectra of synthetic diamonds containing high concentrations of nitrogen and nickel impurities have been examined in both as-grown samples and samples annealed at high temperatures.[19]

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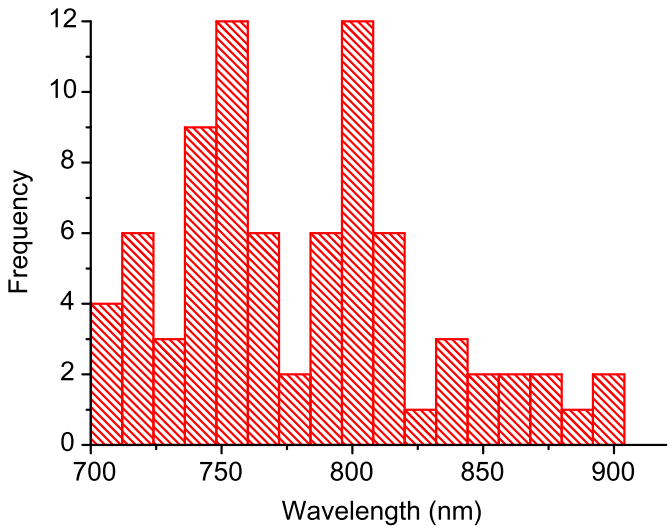

Figure 2. Statistical distribution of ZPLs in the sample.

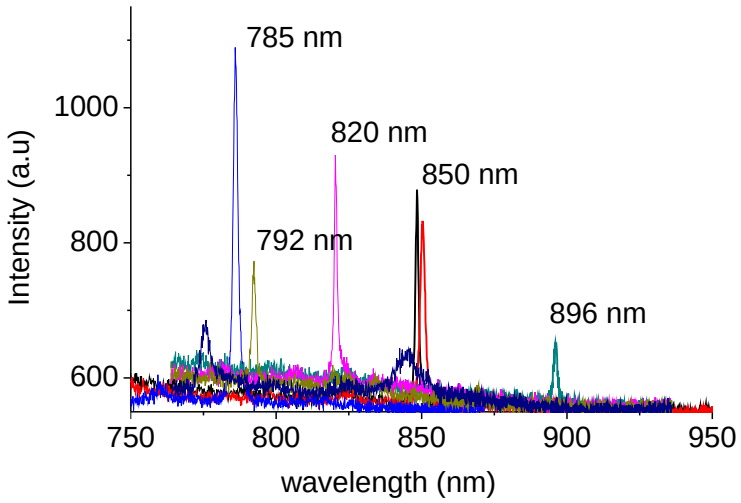

Figure 3. Typical emissions in the region around 800 nm corresponding to single emitters. The line at 792 nm can be associated with NE8, while emission at 780 nm has been found in Ni-Si complex.[24] The emission line at 820 nm is associated to a Nickel-related centre classified in group I (see below). Emission at 850 nm has never been reported before.

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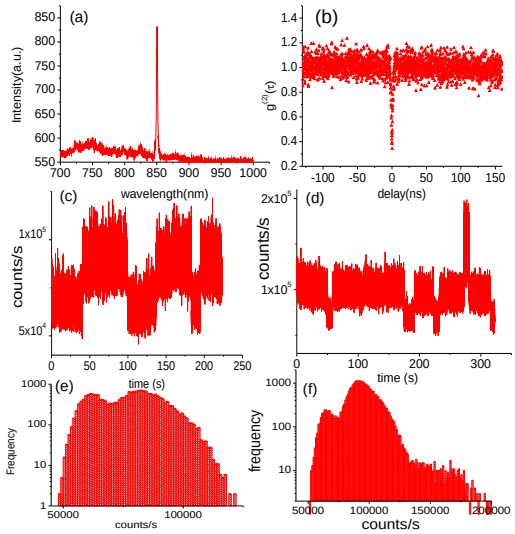

Figure 4. (a) Example of a ZPL at 850 nm, (b) Single photon second order correlation function of the emission line at 850 nm (c) and (d) Examples of blinking fluorescence time traces observed for emitters at 850 nm and their histogram (e) and (f) indicating complex blinking statistics in the photon emission.

A large number of ZPLs have been identified using ensemble room and low temperature spectroscopy as well as EPR with conclusive structures nickel centers, other associated to unspecified nickel complexes. Their behavior on annealing has been studied and some of these lines have also been observed in natural diamonds. Analysis of their behavior on annealing led to their division into three main groups, namely: (I) the ZPLs present in as-grown HPHT diamond. These correspond to isolated nickel atoms in different sites in the diamond lattice (Ni_s or Ni_i). Such complexes disappear after annealing at 1950 K; (II) ZPLs which appear after annealing at approximately 1950 K, but their intensity is lower or they disappear completely after annealing at 2200 K; (III) ZPLs whose intensity increases after annealing at 1950 K and does not decrease after further annealing at 2200 K. A combined optical and EPR study allowed the ZPLs of group I to be conclusively attributed to individual impurities, whereas the ZPLs within group III were associated with nickel-nitrogen complexes containing a single nickel ion in the divacancy position surrounded by a few ($n \geq 2$) nitrogen atoms. The systems within group II were related to a relaxed single nickel defect (nickel ion in divacancy position) and simple nickel-nitrogen complexes, containing a single nitrogen atom. This group has also been referred to as the transient defects group, which consists of defects likely to transform to complex structures at higher temperature.

Our sample corresponds to a different diamond material from the above summarized results from ref. 19 and no annealing has been performed apart from the 700°C growth temperature. Despite this several emission lines common to the above-mentioned work have been observed. It is not possible to perform high-temperature annealing of this sample due to the presence of the silica substrate, but it seems probable that the observed ZPLs are the same as those characterized in the single-crystal HPHT samples previously investigated, but with a distribution in the exact wavelength due to variations in the local environment (e.g. strain). Preliminary work, where a diamond film is grown on diamond substrate in the same conditions as the one here studied, showed emission lines in as-grown material as the one here presented. Upon annealing at high temperature (2000°C), some emission lines disappear and the trend is towards the production/transformation of the more thermodynamically stable complexes such as NE8. Further work is required however to fully confirm this behavior.

An unusual defect at 711 nm has also been observed as single-photon emitter (Fig. 5(a)). This ZPL corresponds to the one observed in an implanted high purity CVD diamond single crystal with Ni and N followed by thermal annealing.[26] According to the studies reported in ref. 26 the defect responsible for the 711 nm peak anneals out between 1100 and 1400°C, which suggests it may be a transient nickel defect (group II). One defect that has been previously identified is NE4 (ZPL 720.7 and 722.7 nm, group II) is also present in our sample, and

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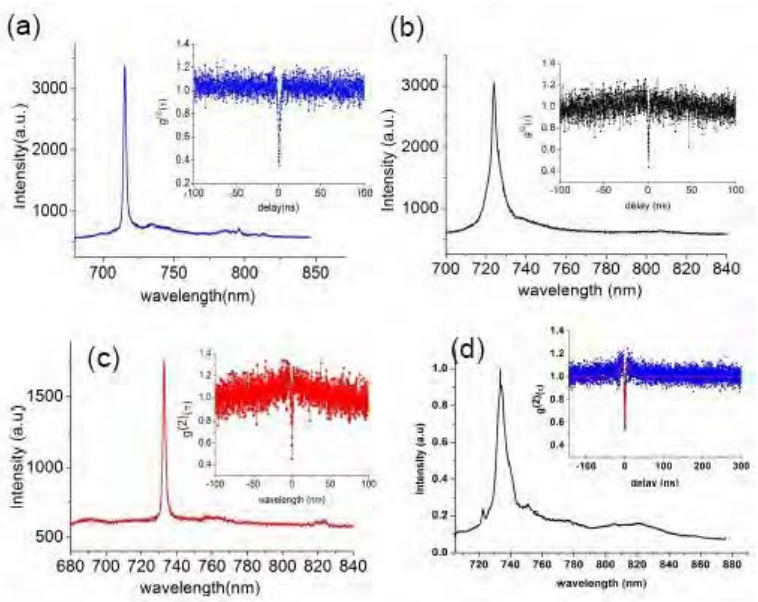

Figure 5. Single-photon second order correlation function from single photon emission lines at 715 nm (a), 720 nm (NE4)(b,c), and SiV (d).

single-photon emission is reported for the first time in Fig. 5(b). The NE4 defect also appears with a narrower spectral emission.[28] The broad emission in the region of 734 nm with observed single-photon emission properties can be tentatively attributed to SiV centers,[12] which are likely to be present in the sample due to the etching of the silica substrate(Fig. 5(d)). Growth of diamond on silica substrates effectively enables doping of the growing diamond with Si. The extent to which this occurs in a diamond film is a function of the film thickness; the thicker the film becomes, the less Si (from the substrate) incorporates. However emission such as a narrow bandwidth line at 733 nm (Fig. 5(c)) are much more common in the sample than a more likely emission related to SiV. To summarize in this sample we observed typical emission corresponding to NV and SiV, which are common in CVD diamond. In addition we observed a known nickel defect as NE4 (ZPL 720.7 and 722.7 nm, group II), as a 711 nm defects found in Nickel implanted diamond. ZPLs in the region 765 to 775 nm have been observed; there is no mention of such features in previous studies using samples synthesized by HPHT. These centers may be associated to Si-Ni complexes showing linewidths between 1.4 to 2.7 nm and also around 10 nm.

4. CONCLUSION

In summary, we have demonstrated the growth of diamond films by CVD which contain single optically-active NE8, NE4 and other nickel-related centers previously identified in HPHT diamond, a Ni-Si complex previously observed in implanted bulk diamond, SiV color centers and a single emission in the IR.

This work, even at this early stage, demonstrates that CVD diamond growth has the potential for controllable production of a wide range of nickel and nitrogen related defects which are found as single centers, a situation which is hard to achieve by HPHT synthesis. Full exploitation of this is a challenging task, a systematic study, planned for the future, where the doping level in the diamond is varied is required. With higher concentrations grown into single crystal CVD diamond EPR would permit the identity of the defects present to be unambiguously determined. The synthesis of further samples suitable for HPHT annealing is also planned, which should provide clues as to the assignment of the features observed in PL by comparing their thermal stability.

Another parameter not studied in detail so far is the effect of the N content in the feedstock, which is important for the formation of nickel-nitrogen complexes. By using different substrate than silica, it should be possible to minimize other contaminants in the reactor (such as silicon) and possibly to increase the yield of a specific dopant, reducing the concurrency of various defects that could induce quenching. Other parameters, such as substrate temperature, can be correlated to defect incorporation and formation yield.

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