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Investigation of the effect of gallium ion (Ga^+) irradiation on the fluorescence properties of synthetic microdiamonds

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The optoelectronic properties of diamond can be tailored via doping for use in a wide range of practical applications, including for intra-cavity laser optics. Understanding the interplay between the ion irradiation conditions and the resulting optical output of diamond is critical in order to develop more highly optimised diamond-based components. Here we present an experimental investigation of the effect of surface gallium ion (Ga+) irradiation as well as focused ion beam (FIB) milling on the fluorescence properties of synthetic microdiamonds fabricated via chemical vapour deposition (CVD). The results obtained from control, low-dose surface irradiated, and FIB-milled microdiamonds are compared. Both the surface irradiated and FIB-milled microdiamonds were observed to have a significant increase in fluorescence output compared to the pristine samples.

Chemical vapour deposition is an effective and widely employed approach to the controlled synthesis of single crystal diamond. By changing the CVD growth conditions the size, shape, and strain state of the sample can be tailored for a particular application [1]. In addition, by introducing different types of impurity atom into the chamber during fabrication, a wide variety of different ‘colour centres’ can be created [2]. Colour centres in diamond are optically active defects which often consist of a vacant atomic site or inclusion of an impurity atom resulting in a specific colour output or characteristic absorption profile.

For our experiments diamond microcrystals were grown on a silicon (Si) substrate using a microwave plasma-enhanced chemical vapour deposition (MPCVD) system. The details of seeding and growth of microdiamonds can be found in reference [1]. Nine crystals were selected to be used for this study based on the SEM results, each having a similar diameter (around 1 – 1.5 μm) and well-developed facets. Three microcrystals (ND#4, ND#7, and ND#8) chosen to act as a ‘control set’ and were left unirradiated. Three microcrystals (ND#1, ND#2, and ND#9) were selected for the ‘low-dose’ set and were surface irradiated with 30 kV Ga+ ions at a fluence of 1.6 × 10-9 C/cm2 (1 × 1010 ions/cm2) with the ion beam (0.115 μm diameter) incident normal to the substrate surface and focused on the uppermost facet of the microdiamond. A final three microdiamonds (ND#3, ND#5, and ND#6) were chosen to act as the ‘FIB-milled set’ and were milled using the Ga+ ion FIB by increasing the dwell time, until a pit approximately 250 nm deep was created in the topmost facet of the microdiamond.

The fluorescence properties of these microcrystals were studied using confocal fluorescence microscopy with a continuous wave laser of 532 nm wavelength at 15 μW power (power density of 24.4 kW/cm2), fitted with a single photon counting avalanche photodiode (APD) detector. A spectrometer (Princeton Instruments Acton 2500i, 0.2 nm resolution) operating over the wavelength range of 550 – 1000 nm, was also attached to this microscope, recording fluorescence spectra with an exposure time per dataset of 30 s. Each measurement was normalised to a separate dark frame exposure. Overall, the fluorescence intensity of the microdiamonds increased (up to 10 times) after ion irradiation using 30 keV Ga+ ions. However, prolonged FIB milling actually showed a small decrease (around 11 – 34 %) in the fluorescence intensity, compared to that of the low-dose crystals, suggesting the formation of a greater proportion of

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non-radiative defect centres in FIB-milled crystals. The confocal fluorescence spectroscopy results confirmed the presence of highly fluorescent $(Si\text{-}V)^{-}$ centres (ZPL at 740 nm), and the N-V Frenkel pairs (peak maximum at 632 nm) in all of the representative microdiamonds. These results also suggest the formation of self-interstitials (peak maxima at 580 nm, 616 nm and 671 nm), divacancy related defect centres (655 / 659 nm), and graphite related defects (643 nm).

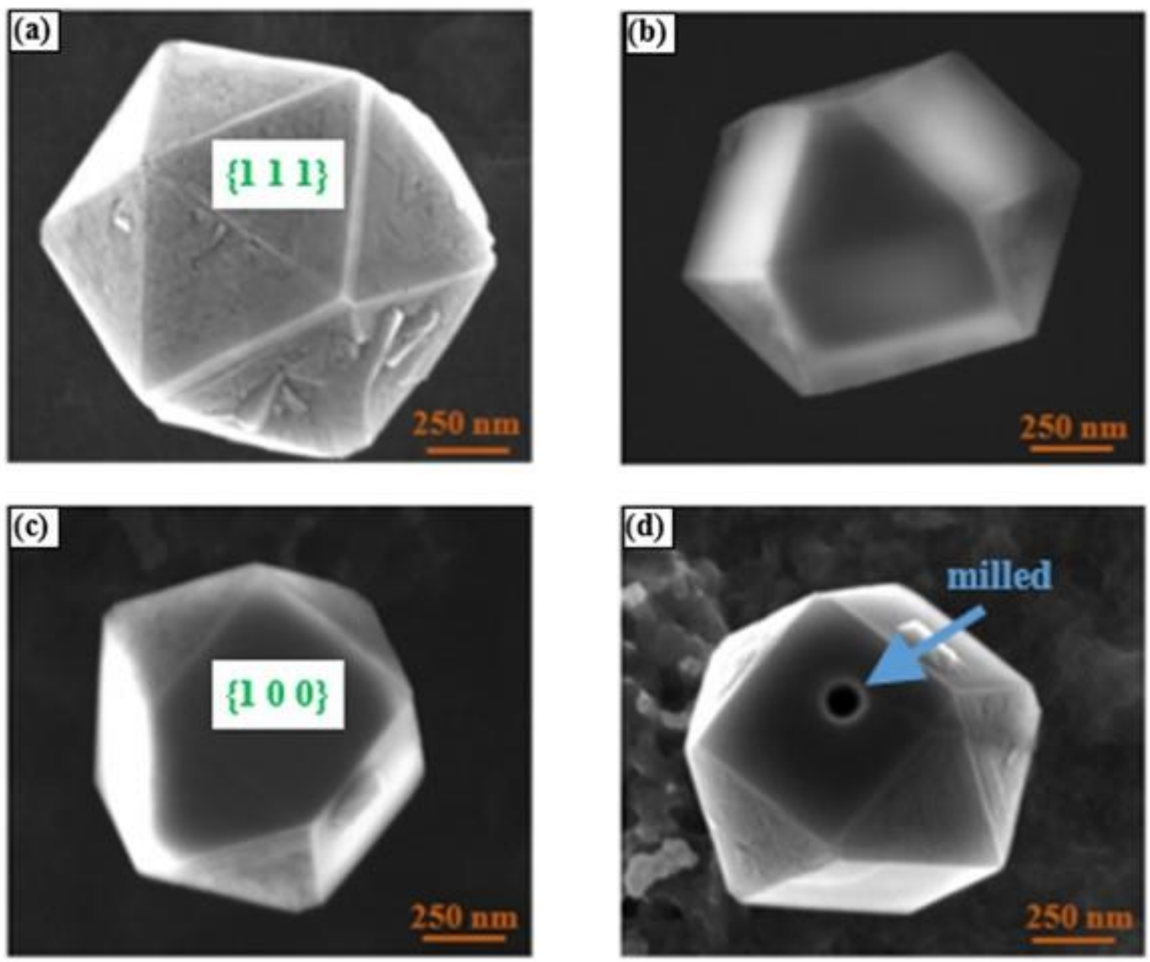

Figure 1. Figure 1 SEM images of the representative (a) control (ND#8), (b) low-dose (ND#2), and (c) – (d) milled sample (ND#3) before and after FIB milling, respectively.

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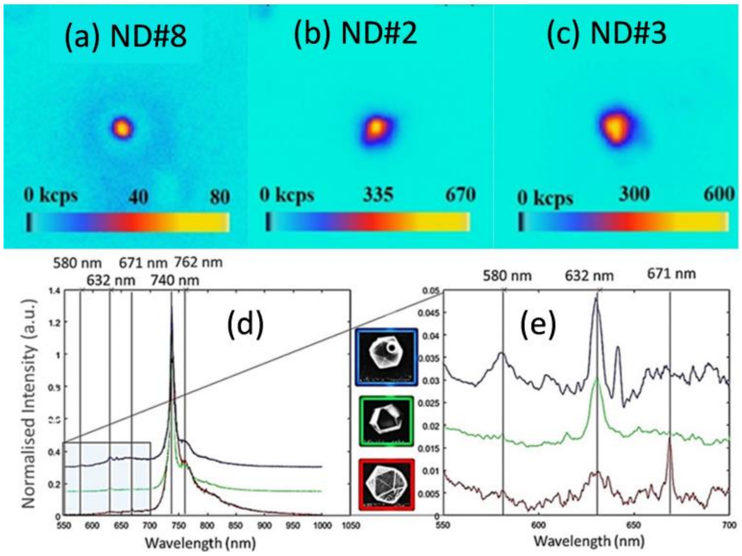

Figure 2. Figure 2 Confocal fluorescence microscopy of (a) control (ND#8), (b) low-dose (ND#2), and (c) FIB-milled (ND#3) microdiamonds. (d) Confocal fluorescence spectra of control (ND#8, red), low-dose (ND#2, green) and FIB-milled (ND#3, blue) microdiamonds. (e) an enlarged section of the fluorescence spectra in the range of 550 – 700 nm wavelength.

References

1. Stacey, A., et al., *Controlled synthesis of high quality micro/nano-diamonds by microwave plasma chemical vapor deposition.* Diamond and Related Materials, 2009. **18**(1): p. 51-55.
2. Collins, A.T., *The characterisation of point defects in diamond by luminescence spectroscopy.* Diamond and related materials, 1992. **1**(5-6): p. 457-469.

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