EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Addendum to IS668 to the ISOLDE and Neutron Time-of-Flight Committee

Quantum colour centers in diamond studied by emission channeling with short-lived isotopes (EC-SLI) and radiotracer photoluminescence

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Abstract

Within IS668, electron emission channeling (EC) experiments at ISOLDE have successfully established the existence of implanted impurities Sn, Ge, Ga, Mg and Xe in diamond in the split vacancy configuration. We propose to continue the structural characterization of quantum colour centers in diamond by means of EC, which shall be supplemented by conventional and radiotracer photoluminescence (PL) measurements, with the aim of correlating the lattice location results with the optical properties (linewidth, intensity, lifetimes) of known luminescence lines, but also for establishing the chemical nature of PL lines of unknown or doubtful origin. The aim is to extend the studies performed so far with Sn, Ge, Ga, and Mg towards low temperature implantations, and in selected cases to diamonds pre-doped with acceptors or donors. In addition, we would like to address elements (Pb, Si, He, Ne, Ar, Kr) for which it was not possible to schedule beam time during the two years that IS668 has been running.

Requested shifts: 20 shifts (split into ~8 runs over 2 years)
Experimental Area: GHM and off-line lab in building 508-R-008

Motivation

Modern confocal microscope techniques allow the optical excitation, detection and manipulation of single, isolated point defects in solids. Such single photon emitters, that consist of a foreign atom or a native defect, as manifested, e.g., by the so-called "colour centers" in diamond, are currently at the forefront of efforts to establish crucial building blocks for a number of quantum technologies [1-3], in particular quantum information processing (including quantum cryptography and quantum repeaters for use in quantum networks) and quantum metrology (e.g. nano-sensors for magnetism or temperature). Since the submission of our original proposal [4] in 2020, research on colour centers in diamond for quantum applications has further intensified. Besides continued interest in N*V* centers (which are not part of this proposal, no N beams are available at ISOLDE), particular focus in the literature has been on group-IV *vacancy* centers (as reviewed in Refs. [2, 5-7]), but also other impurities such as Mg [8-11], with approximate number of publications from Web of Science since Jan 2020 being 82 on Si, 37 on Ge, 20 on Sn, 6 on Pb, 4 on Mg. In the case of Si, Ge, and Sn, the microscopic structure of the underlying centers has meanwhile been quite well established; it consists of the group-IV foreign atom centered inside a double vacancy with a <111> symmetry axis, in a position that is equivalent to a bond-centered (BC) site in an unperturbed crystal. This so-called "split-vacancy configuration" is theoretically predicted [13] to be frequently found for foreign atoms in diamond, owing to the fact that atoms that are larger than carbon require more space around them in order to be stable in the tight diamond lattice.

In the case of Sn, direct evidence for implanted Sn in the split-vacancy configuration was provided by emission channeling experiments [14]. Thus, while the nature of the main Sn zero phonon line (ZPL) at 620 nm is due to split-vacancy, recent results point to the fact that a second ZPL observed around 646 nm may be ascribed to other configurations of Sn in the lattice [15-17], in particular since it seems to have a different symmetry than <111> [15].

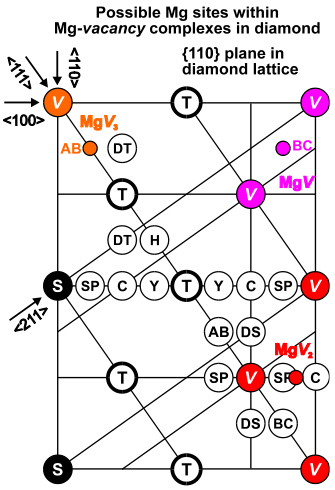

Fig. 1. Positions of the substitutional and major interstitial sites in diamond. Also shown (in magenta, red, and orange, respectively) are the split-vacancy Mg*V* defect with Mg on BC sites and possible structures of MgV_2 and MgV_3 complexes. Note the MgV_3 complex contains 3 more vacancies out of the {110} plane. From [Corte 22].

On the contrary, for Pb the microscopic structures of the centers that are responsible for various ZPLs in the region 520-575 nm, remain unclear [18-21]. Theory has predicted the ZPL for split-vacancy Pb*V* to be around 516 nm [5, 19] but it has been emphasized that other configurations of Pb may also play prominent roles [18, 20-21], including complexes with a larger number of vacancies.

Although the main Ge-related ZPL is generally accepted to also originate from split-vacancy Ge*V*, the reliable and reproducible fabrication of Ge*V* defects has remained a key challenge and is the main obstacle for practical development of Ge*V*-based spin-photon interfaces. In that respect, the formation yield of optically active Ge*V* following ion implantation was reported to be as low as 0.4-0.7% only [22-23], considerably lower than in the case of Si (~2.5% for SiV^- [23-24]) or Sn (2.5% [9, 23]).

While ion implantation remains the most widely used technique to introduce the foreign atoms in diamond [2, 7-9, 11-12, 14-25], the challenge that is faced, is to controllably incorporate the impurities in such a way that a large number of them assume the required structural configuration with a minimum of other

defects in the neighbourhood, thus showing the desired and well-defined optical properties. In that regard, a new aspect that has recently been addressed, is that co-doping with acceptors or donors can critically modify the luminescence efficiency of implanted foreign atoms in diamond [9, 15, 23].

We propose to continue the structural characterization of quantum colour centers in diamond by means of electron emission channeling (EC) [25] at ISOLDE. The EC experiments shall be supplemented by conventional and radiotracer PL measurements, with the aim of correlating the lattice location results with the optical properties (linewidth, intensity, lifetimes) of known luminescence lines, but also for establishing the chemical nature of PL lines of unknown or doubtful origin. The proposed experiments will provide data for three PhD theses (two at KU Leuven, one at UAveiro).

Summary of results

Since the start of IS668, we have performed beam times for Mg, Xe, Ga/Ge, and Sn, using the isotopes ^{27}Mg, ^{135}Xe, ^{72}Ga, ^{75}Ga, ^{76}Ga, ^{75}Ge, ^{119m}Sn, and ^{121}Sn. Results from the Mg run have meanwhile been published [16] and represent first direct evidence (Fig. 2) of Mg on bond-center (BC) sites, a position that is compatible with the theoretically predicted MgV defect [10] in the split-vacancy configuration. Samples implanted at ISOLDE (Fig. 3) as well as at KU Leuven [12] exhibited the characteristic ZPL of MgV at 557 nm. While the narrow

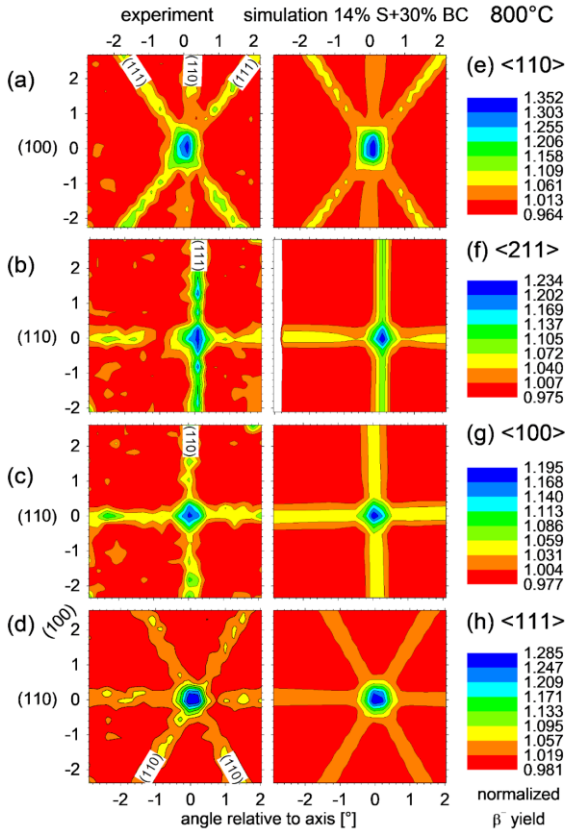

Fig. 2. (a)-(d): Experimental β^- emission channeling patterns from ^{27}Mg in diamond around the <110>, <211>, <100>, and <111> directions during 800°C implantation. The plots **(e)-(h)** are simulated theoretical patterns considering 14% on substitutional and 30% on bond-center sites. From Ref. [Corte 22].

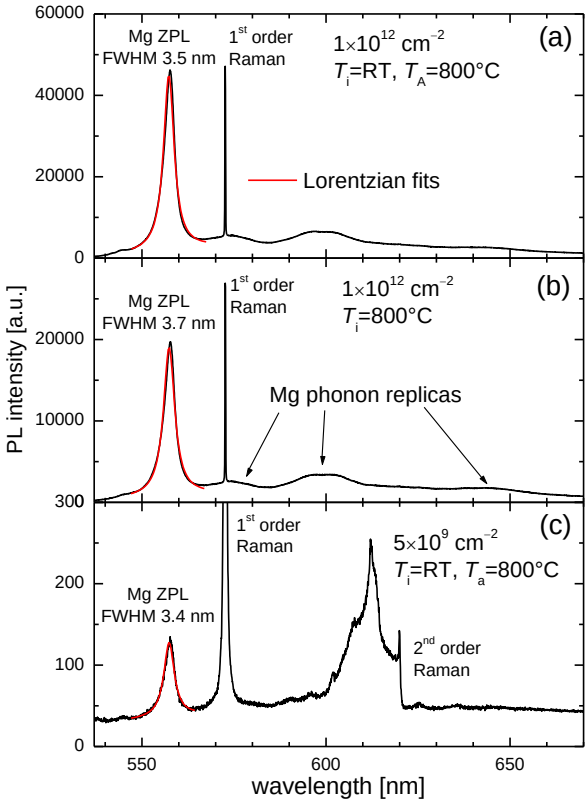

Fig. 3. RT PL spectra (532 nm excitation) of "electronic grade" diamond implanted at ISOLDE with 30 keV ^{24}Mg, measured at University of Turin: **(a)** 1×10^{12} cm^{-2} RT implantation, followed by 20 min 800°C annealing; **(b)** 1×10^{12} cm^{-2} implanted at 800°C; **(c)** 5×10^{9} cm^{-2} RT followed by 20 min 800°C annealing.

FWHM of the 1st order Raman peak at 572.6 nm is given by the instrument resolution, the near Lorentzian shapes and practically identical FWHM of the Mg ZPLs at 557 nm obtained under different implantation conditions indicate that they are dominated by the luminescence lifetime, hence that defect-induced broadening plays a minor role even at comparably low annealing temperature of 800°C. Previously, the formation efficiency of optically active Mg*V* centers in undoped diamond implanted at RT and annealed to 800°C had been estimated as 2-3% only [9] from a comparison of fluorescence intensity and implanted fluence. Our data show that 30-40% of implanted Mg atoms are found on BC sites (Fig. 1), hence that the structural efficiency of formation of the split-vacancy configuration is certainly much higher than a few percent only, thus suggesting that a large part of the Mg*V* centers may be optically inactive. The fact that significantly higher optical activation has been reported for P-doped diamond, reaching values as high as 48% following 1200°C annealing [9], indicates that co-doping with the *n*-type dopant P may transform an inactive form of Mg*V* into optically active Mg*V* centers. In our fit analysis of the EC data, relatively large fractions of Mg emitter atoms (43% at RT, 56% at 800°C) were assigned to flat contributions to the emission patterns, the so-called "random sites". The assignment of random sites cannot be the consequence of significant radiation damage or amorphization of the sample, since for the light mass of ^{27}Mg at the applied fluences, the effect of damage should be negligible in diamond, especially for the implantation temperature of 800°C. The most likely interpretation is that the random fraction represents additional ^{27}Mg sites of relatively low crystal symmetry, which only produce weak anisotropies in the angular-dependent electron emission yields. Two defect configurations, which could be responsible for such Mg lattice sites, are Mg inside a triple vacancy or inside a quadruple vacancy, which corresponds to the so-called MgV_2 or MgV_3 complexes (possible structures for these complexes are shown in Fig. 1).

In case of Xe, the fraction on BC sites was ~30% immediately following RT implantation, and increased to ~66% following 900°C annealing. Our findings hence show that Xe*V* can be very effectively formed by means of ion implantation and annealing at moderate temperatures, also exceeding the optical activation of 28% reported in the literature [25].

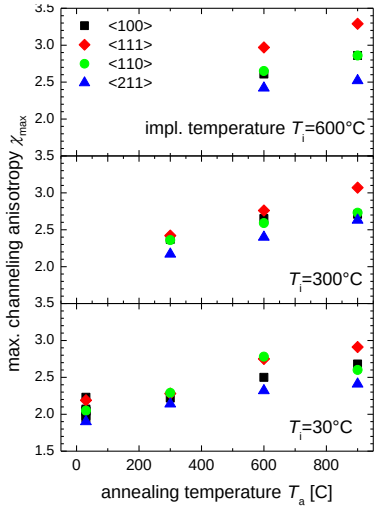

Fig. 4. Maximum channeling anisotropy of <100>, <111>. <211> and <110> directions of diamond as a function of implantation and annealing temperatures T_i and T_a of ^{75}Ge.

The analysis of the Ga/Ge and Sn experiments is ongoing. While detailed fits still need to be performed, the particularly intense β^- channeling effects (~300% anisotropy compared to ~30% for ^{27}Mg) observed following the decay of ^{75}Ge for all crystallographic directions (Fig. 4) suggest very high substitutional and rather low BC fractions, the latter probably being a few % only. These results indicate that Ge*V* complexes show a much lower structural yield of formation than the other group-IV impurities, with substitutional Ge dominating. In particular, taking the maximum channeling anisotropy as an indicator for substitutional occupation, it suggests that high-temperature implantation and annealing favour the formation of substitutional Ge over Ge*V*. While this would confirm the considerable problems of its optical activation as Ge*V*, it remains an intriguing question why Ge, which is heavier than Si but lighter than Sn, shows this particular behaviour, for which there exists so far no theoretical explanation; in particular the binding energy for Ge*V* is predicted to be in between those of Si*V* and Sn*V* [13].

During the Sn beam time, we were surprised by the high contribution of isomeric states in the beam. Optimizing the laser ionization process for enhancing the ionization of the desired isotope ^{121}Sn ($t_{1/2}$=27.1 h) over unwanted ^{121m}Sn (55 y), allowed to improve the ratio of ^{121}Sn/(^{121}S+^{121m}Sn) from 17% to 28%, though not as much as was hoped for. On the other hand, the high yield of isomeric states allowed high fluence implantations with the conversion electron (CE) emitting isomeric state ^{119m}Sn (293 d). These ^{119m}Sn experiments are still ongoing and while they will give information on lattice location of implanted Sn in the fluence regime 1-5×10^{13} cm^{-2}, the low energies (36 and 62 keV) of the emitted CEs provide better angular resolution of the channeling effects, and hence may allow for even more precise assessment of the lattice location of Sn. The results of ^{121}Sn in artificial, chemical-vapour deposition (CVD) diamond seem to confirm the previously obtained ones for natural diamond [14]. Due to the fact that no more suppliers of natural diamond suitable for electronic applications could be found, contrary to the original intention we decided to focus entirely on single crystals grown by the CVD technique.

Proposed experiments

During the two years of running IS668, we have made good progress in investigating formation conditions of colour centers in diamond for RT implantation followed by thermal annealing, as well as in many cases by high temperature implantation. No experiments have been performed for implantations below RT, which was foreseen at a later stage in IS668. We propose to attempt this now, in particular for the cases of Mg, Sn and Ge.

Another new aspect that we would like to address, is lattice location of colour center impurities following implantation into diamonds that are previously doped with donors or acceptors. Donor or acceptor doping should change the charge state distribution of impurity-vacancy complexes, but it has also been hypothesized that it directly influences their formation yield by supressing or enhancing vacancy mobility [9, 15]. While *p*-type diamonds are commercially available, donor-doped diamonds are only produced within research environments. We are currently exploring ways to procure such samples, an alternative is to ion implant B acceptors or P donors at KU Leuven.

So far no experiments were performed for ^{209}Pb, which we consider hence to have priority for the allocation of beam time. In this case, careful assessments of contaminants of the beam will need to be performed, in particular to ensure that contaminations of ^{209}Fr are at a tolerable level. We aim at a series of lattice location experiments that start by implanting ^{209}Pb at RT, followed by an annealing sequence up to 900°C, where EC lattice location is performed in the as-implanted state and following each annealing step. This will be followed by implantations at successively higher temperatures, in analogy to what was performed for Ge shown in Fig. 4. Depending on the intensity of the ^{209}Pb beam available, these measurements may be performed for various implantation fluences. This should on the one hand establish that Pb*V* centers in split-vacancy configuration are formed, on the other hand whether their formation is altered by higher temperatures. This is particularly relevant for the high mass of Pb, which produces significantly more vacancies during implantation than elements of lower mass.

As described above, the very high anisotropy of the ^{75}Ge EC patterns indicates that the efficiency of formation of Ge*V* is about an order of magnitude less than in the cases of Sn*V*, Ga*V*, Xe*V*, or Mg*V*. The reason for this peculiarity of Ge is quite unclear and of interest both from fundamental aspects of our theoretical understanding of split-vacancy configurations and for applications. It should hence be further investigated with high priority, also whether it can be improved, in particular by means of low temperature implantation. The research on

Ge*V* will be incorporated in a recently funded project titled "Towards the ultimate quantum light matter interface" (2022-2025, 2.75 M€), within a consortium of the QSP group with several Flemish partners. An efficient photon-matter interface is a key building block in both quantum networks and quantum computing applications, QSP is developing such a quantum node based on the Ge*V* center in diamond, which, thanks to its symmetry properties (split-vacancy), is less sensitive to the environment compared to the widely investigated N*V* center, and through its larger ground state splitting (170 GHz) allows for operation at higher temperatures (max. operating temperature between 0.4 and 8 K) than the better established Si*V* center (50 GHz, corresponding to 0.1-2 K).

For the noble gas isotopes ^{6}He, ^{23}Ne, ^{41}Ar, and ^{87}Kr, for which no beam time could be scheduled so far, we propose short exploratory studies in conjunction with other users that make extended use of targets that can provide these elements. In the case of ^{6}He, we require a cold plasma source. While this was available during a first beam time in May 2022 and was used for Xe beams, it failed before the EC experiments with ^{6}He could be performed. A hot plasma source tried in Oct 2022 proved unsuitable due to overwhelming contamination with $^{12}C^{2+}$. Elements that will not be pursued further are Ca, Sr, Xe and Ni.

So far it has not been possible to successfully use the PL lab at ISOLDE. While we have bought a 532 nm Cobolt DPL-06 green laser, the setup was found to lack sensitivity due to constraints in the imaging lenses, and exhibit poor reproducibility resulting from the absence of a high-precision mechanical sample moving stage. These pieces of equipment have been acquired, and it is anticipated that it can be used in 2023.

Summary of requested shifts:

isotope	half-life	yield (atoms/μC)	target - ion source	Shifts (8h)
^{209}Pb	**3.25 h**	no yield in data base	UC_x-Nb - RILIS Pb or LIST Pb	4
^{75}Ga→**^{75}Ge**	126 s→**82.8 min**	3×10^{7}	UC_x-W - RILIS Ga	3
^{121}Sn	**27.06 h**	1×10^{8}	UC_x-W - RILIS Sn	4
^{27}Mg	**9.5 min**	1×10^{7}	Ti-W - RILIS Mg	2.5
^{28}Mg	**21 h**	6×10^{6}	Ti-W or UC_x-W - RILIS Mg	0.5
^{6}He	**807ms**	7×10^{7}	UC_x or BeO **cold** plasma	3.0
^{23}Ne	**37.2 s**	1.6×10^{6}	UC_x plasma	1.0
^{41}Ar	**109 min**	1.6×10^{6}	UC_x or TiO_2 plasma	0.5
^{87}Kr	**76.3 min**	2×10^{8}	UC_x or PbBi plasma	0.5
^{31}Al→**^{31}Si**	644 ms→**157 min**	2.5×10^{5}	UC_x-W - RILIS Al	1
			Total shifts:	**20**

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Appendix

DESCRIPTION OF THE PROPOSED EXPERIMENT

The experimental setups comprise: the EC-SLI on-line setup at GHM and 3 off-line EC setups dedicated to emission channeling experiments, the SSP collection chamber at GLM, and the ISOLDE SSP Photoluminescence Spectrometer

Part of the experiment	Design and manufacturing
(1) Emission Channeling setups	☒ To be used without any modification EC-SLI setup at b.170-GHM [1] and off-line EC setups at b.508-R-008. ☐ To be modified
(2) SSP Photoluminescence Spectrometer	☒ To be used without any modification The SSP photoluminescence spectrometer is permanently assembled in b.508-R-001 (edms documentation in preparation) ☐ To be modified
(3) SSP collection chamber(s) at GLM	☒ To be used without any modification Standard SSP collection chamber at GLM[2] ☐ To be modified
[insert lines if needed]	

[1] https://edms.cern.ch/document/1960302/1 (EC-SLI setup)
[2] https://edms.cern.ch/document/1693386/1 (SSP collection chamber)