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# 1 Electron spectroscopy with a diamond detector

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### 8 Abstract

9 An electronic grade single crystal chemical vapour deposition diamond was investigated as a

10 prototype high temperature spectroscopic electron ( $\beta$  particle) detector for future space science

11 instruments. The diamond detector was coupled to a custom-built charge-sensitive preamplifier of

12 low noise. A <sup>63</sup>Ni radioisotope source (endpoint energy 66 keV) was used to provide a spectrum of  $\beta^{-1}$ 

13 particles incident on the detector. The operating temperature of the detector/preamplifier assembly

14 was controlled to allow its performance to be investigated between +100 °C and -20 °C, in 20 °C

15 steps. Monte Carlo modelling was used to: a) calculate the  $\beta^{-}$  particle spectrum incident on the

16 detector; b) calculate the fraction of  $\beta^{-}$  particle energy deposited into the detector; and c) predict the  $\beta^{-}$ 17 particle spectrum accumulated by the instrument. Comparison between the model and experimental

data suggested that there was a 4.5 µm thick recombination region at the front of the detector. The

spectrometer was demonstrated to be fully operable at temperatures, T, -20 °C  $\leq T \leq 80$  °C; the results

suggested that some form of polarisation phenomenon occurred in the detector at > 80 °C. This

21 article presents the first report of an energy calibrated ( $\leq 50$  keV) spectroscopic  $\beta$ - particle diamond

22 detector.

### 23

## 24 **1. Introduction**

25 Electron spectrometers are key instruments in space science. The distributions of electrons and other

26 charged particles following magnetic field lines are used to map the topology of magnetic fields and

27 measure plasma densities in space environments [1]. Electron energy spectra, spatial distributions,

and directions of travel allow absorption and acceleration processes to be understood in planetary

magnetospheres, and the understanding of electron populations at icy moons, asteroids, and comets, is

30 essential for exploring radiolytic processing and chemistry on the surface of those bodies [2, 3]. The

31 radiolytic chemistry of comets is most easily studied when they are around perihelion; a temperature

of 87 °C was measured at 1P/Halley at 0.8 AU [4]. The temperature at Mercury, where study of the electron radiation environment is also interesting, can be > 400 °C [5]. As such, the development of

electron radiation environment is also interesting, can be > 400 °C [5]. As such, the development of instrumentation which can operate in high temperature (>> 20 °C) environments is beneficial in order

to simplify thermal design of the spacecraft.

36

37 Electron spectrometers have been an integral part of numerous space missions to study the

38 interactions between the solar wind and magnetospheres and atmospheres. The Backward Facing

39 Electron Spectrometer (BESA) on board Mariner 10 [6] as part of the Scanning Electrostatic Analyser

40 and Electron Spectrometer (SESA) instrument, established that Mercury possessed a magnetic field,

41 and later the Energetic Particle Spectrometer (EPS) designed to measure composition, energy, and

42 angular distribution of electrons (> 20 keV) and ions (> 5 keV nucleon<sup>-1</sup>) was included in

43 MESSENGER's suite of instruments on the most recent mission to visit the planet [7]. The EPS

44 instrument [8], with minor modifications, flew on the New Horizons mission to Pluto and trans-

45 Neptunian object Arrokoth, as the Pluto Energetic Particle Spectrometer Science Investigation

46 (PEPSSI) [9]; the EPS/PEPSSI electron spectrometer featured an array of Si solid-state detectors. An

47 Electron Spectrometer (ELS) on the Cassini mission to the Saturn system was part of the Cassini

48 Plasma Spectrometer (CAPS); it was capable of detecting electrons with energies from 0.58 eV to

49 26.04 keV [1] with a field of view in excess of  $\pm$  60 degrees in elevation and azimuth [10]. The ELS

used a micro-channel plate (MCP) detector coupled with an electrostatic analyser to collect energy
 spectra [10].

52

53 Solid state detectors made from Si must be kept cool ( $\leq 20$  °C) to limit their thermally generated

- 54 leakage currents because of the material's relatively narrow bandgap ( $E_g = 1.12 \text{ eV}$ ) [11].
- 55 Furthermore, in harsh radiation environments, they must be shielded in order to avoid degraded

56 performance through radiation damage [12]. MCP detectors coupled with electrostatic analysers are

57 an alternative but they require high voltage and high vacuum to operate and are more complex

58 instruments [13].

59

60 As such, an electron spectrometer that could operate unshielded, uncooled, and with modest power

61 supply requirements would be very attractive for future space missions. Wide bandgap solid state

- 62 detectors may provide such benefits. Wide bandgap radiation detectors that have been proposed for
- 63 spectroscopic electron detection for space applications include GaAs [14, 15], SiC [16], AlGaAs [17,
- 18], and InGaP [19]. Each offers particular advantages and trade-offs in terms of radiation hardness,
- temperature tolerance, and electron detection efficiency. Single crystal chemical vapour deposition (CVD) diamond has previously been shown to be responsive to  $\beta^2$  particles at high temperatures [20]
- and spectroscopic to  $\beta^{-}$  particles within the estimated energy, *E*, range, 50 keV < *E* ≤ 820 keV [21].

Recently a single crystal CVD diamond detector has been reported to be spectroscopic to soft X-rays

- 69 (< 10 keV) at 20  $^{\circ}$ C [22].
- 70

Now, in this article, a diamond detector has been investigated as a potentially temperature, *T*, tolerant (-20 °C  $\leq$  *T*  $\leq$  100 °C) electron spectrometer using lower energy electrons from a <sup>63</sup>Ni radioisotope  $\beta^{-}$ particle source (endpoint energy 66 keV).

74

# 75 2. Electrical characterisation and <sup>63</sup>Ni β<sup>-</sup> spectroscopy 76

77 2.1. Diamond detector

78 An Element Six UK Ltd. electronic-grade single crystal CVD diamond (area 2.0 mm by 2.0 mm;

thickness 0.5 mm) was used [23]. A single square 1.4 mm  $\times$  1.4 mm contact (50 nm Ti, 200 nm Ag) was sputtered onto the centre of the front face and rear face of the detector. The detector was

mounted in a custom TO-39 style package with the detector's rear contact connected to the package

by silver-loaded epoxy [24] and the top contact connected to a pin of the package by wirebond.

83

84 2.2. Leakage current and capacitance of the packaged detector

85 The leakage current of the detector was measured as a function of electrical potential difference and 86 temperature. For this, the packaged detector was positioned inside a light shielded test harness. Once 87 the detector was positioned, the test harness was sealed and purged with dry  $N_2$  to displace 88 environmental moisture. The test harness holding the packaged detector was then placed in a TAS 89 Micro MT Climatic Cabinet. Once sealed inside, dry N<sub>2</sub> was continually fed into the climatic cabinet 90 to ensure the test environment remained dry (relative humidity < 5%). To commence measurement, 91 the climatic cabinet's temperature was raised from ambient ( $\approx 20$  °C) to 100 °C with a 30 minute pause in the temperature climb imposed every 20 °C. This allowed the test harness and the packaged 92 93 detector to reach thermal equilibrium inside the cabinet without undue thermal stress. At 100 °C, the 94 temperature inside the climatic cabinet was held constant for 1 hour before leakage current 95 measurements were commenced. The electrical potential difference was applied across the detector in 96 5 V increments up to 100 V in both polarities. This procedure was repeated in 20 °C decrements until 97 a temperature of -20 °C was reached; for the temperature descent, a 30 minute waiting time was 98 imposed at each temperature to allow the test harness and detector to reach thermal equilibrium. A 99 Keithley 6487 picoammeter/voltage source, controlled by National Instruments LabVIEW software,

100 was used to apply the potential difference and measure the leakage current. The results of the

- 101 measurements are presented in Figure 1.
- 102
- 103 The maximum packaged detector leakage currents recorded with +100 V of electrical potential
- applied across the detector were 63.4 pA  $\pm$  0.6 pA (T = 100 °C), 7.5 pA  $\pm$  0.4 pA (T = 80 °C), and 1.0
- 105 pA  $\pm$  0.4 pA (T = 60 °C). At T  $\leq$  40 °C, the leakage current remained below the noise floor of the
- 106 Keithley 6487 picoammeter ( $\pm$  0.4 pA). A portion of the larger leakage current measured at higher
- 107 temperatures was attributed to the thermal excitation of electrons into the diamond's conduction band 108 [25]. The detector's lookage summert density was 2.22 mA  $cm^2 + 0.02$  mA  $cm^2$  at 100 %C and 100 W
- 108 [25]. The detector's leakage current density was 3.23 nA cm<sup>-2</sup>  $\pm$  0.03 nA cm<sup>-2</sup> at 100 °C and 100 V 109 applied potential difference (field strength 2 kV cm<sup>-1</sup>); this assumed that the leakage current was
- 110 constrained to the volume under the detector's electrical contacts and that surface leakage current

- 111 pathways were negligible. Such a leakage current density is more than that reported by other
- 112 researchers using single crystal CVD diamond. As an example, two electronic grade single crystal
- 113 CVD diamond detectors, grown by Element Six UK Ltd [23], had leakage current densities of
- 114 110 pA cm<sup>-2</sup> and 220 pA cm<sup>-2</sup> at 100 °C and a field strength of 2 kV cm<sup>-1</sup> [26]. However, Kumar et al.
- 115 [27] reported a much larger leakage current density ( $\approx 40$  nA cm<sup>-2</sup> at an electric field strength of
- 116  $2 \text{ kV cm}^{-1}$ ) at 100 °C, for a single crystal CVD diamond detector grown by IIa Technologies Pte. Ltd., 117 Singapore. The leakage currents reported by Tchouaso et al. for their  $\beta^{-}$  particle diamond detector
- (also grown by Element Six UK Ltd) were approximately 6.4 pA cm<sup>-2</sup> and 12.7 pA cm<sup>-2</sup> at an electric
- filed strength of  $\pm 2 \text{ kV cm}^{-1}$  [21]. Those leakage current measurements were made at ambient
- 120 laboratory temperature. The asymmetry of leakage current between polarities (Figure 1) for the
- 121 presently reported detector have been reported for other CVD diamond detectors [28, 29]; asymmetric
- 122 leakage current and leakage current hysteresis in diamond detectors has been shown to depend upon
- 123 the surface treatment employed prior to metallisation and the type of metallisation used [30].
- 124
- 125 The leakage current of the packaged detector at T > 40 °C and at an applied electrical potential
- 126 difference of 100 V in both polarities reduced as a function of time, see Figure 2. The maximum
- 127 leakage current after 30 minutes with 100 V potential difference continuously applied across the
- 128 packaged detector was 39.1 pA  $\pm$  0.5 pA, 6.3 pA  $\pm$  0.4 pA, and 0.6 pA  $\pm$  0.4 pA at T = 100 °C, T = 80
- 129 °C and T = 60 °C, respectively.
- 130



131 132

Figure 1. a) The leakage current of the packaged detector as a function of electrical potential, in both polarities, up to 100 V, and at temperatures, T, 40 °C  $\leq$  T  $\leq$  100 °C. T = 100 °C (open squares),

- 135 polarities, up to 100 V, and at temperatures, T, 40 °C  $\leq$  T  $\leq$  100 °C. T = 100 °C (open squares), 134 T = 80 °C (open diamonds), T = 60 °C (open triangles), and T = 40 °C (crosses). At T < 40 °C, the 135 detector's leakage current was below the noise floor of the picoammeter (± 0.4 pA). Error bars are
- omitted for clarity; at +100 V, the uncertainties in leakage currents were  $\pm 0.6$  pA and  $\pm 0.4$  pA at T =
- 137 100 °C and T = 60 °C, respectively.
- 138



Time of reading (minutes)
Figure 2. The leakage current of the packaged detector as a function of time at applied electrical
potential differences of (a) +100 V and (b) -100 V, at temperatures of 100 °C (open squares), 80 °C
(open diamonds), and 60 °C (open triangles).

- 144 The capacitance of the packaged detector and an identical device with its bondwires removed, were
- 145 measured as functions of applied potential difference, in 5 V steps up to 100 V in both polarities. The
- packaged detector and the device with bondwires removed were separately placed in a test harness,
- which was purged with dry  $N_2$  and subjected to the same environmental regime as had been used for the leakage current measurements. A HP 4275A Multi Frequency LCR meter, with test signal set to
- 148 the leakage current measurements. A HP 42/3A multi Frequency LCR meter, with test signal set 149 50 mV rms magnitude and 1 MHz frequency, was used to record the capacitance of the packaged
- 150 detector and the device with bondwires removed. The packaged detector's capacitance was
- 151 independent (within the LCR meter's uncertainties) of applied potential difference and temperature; it
- 152 was 870 fF  $\pm$  20 fF and 840 fF  $\pm$  20 fF at 100 °C and -20 °C, respectively. The capacitance of the
- 153 device with bondwires removed was 540 fF  $\pm$  20 fF and 520 fF  $\pm$  20 fF at the same temperatures. The
- 154 capacitance of the bare die detector was therefore invariant and 320 fF  $\pm$  30 fF at all reported
- 155 temperatures and electrical potential differences.
- 156

# 157 2.3. <sup>63</sup>Ni $\beta$ <sup>-</sup> particle spectra

The packaged detector was connected to a custom-built low-noise charge-sensitive preamplifier with a Vishay 2N4416A Si input Junction Field Effect Transistor (JFET) [31] at its input. The preamplifier

- 160 is unconventional in that it did not have a feedback resistor installed in parallel with a feedback
- 161 capacitor to discharge charge pulses collected from  $\beta$  particle interactions. Instead, the feedback
- 162 capacitor is discharged through the gate-to-source channel of the Si JFET. The design of the
- 163 preamplifier was similar to that reported by Bertuccio et al. [32]. The detector and preamplifier were
- 164 co-located in a light shielded test harness. Charge pulses (in the form of voltage pulses) from the
- 165 preamplifier's output were shaped and amplified by an Ortec 572A shaping amplifier. An Ortec
- 166 EASY 8k multi-channel analyser (MCA) subsequently sorted the voltage pulses from the shaping
- 167 amplifier into appropriately distributed bins. The electrical potential difference across the detector
- 168 was applied by a Keithley 6487 voltage source. The  $\beta^2$  particle spectrum was provided by a <sup>63</sup>Ni
- 169 radioisotope  $\beta$ <sup>-</sup> particle source (activity 179 MBq; endpoint energy 66 keV). The <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle
- 170 radioisotope source was a 7 mm by 7 mm by 3  $\mu$ m thick <sup>63</sup>Ni layer with a 1  $\mu$ m Ni overlayer,
- 171 electroplated onto a Ni foil substrate. It was held in a cylindrical stainless-steel holder with a 6 mm
- 172 diameter open aperture. The <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle source was positioned  $\approx$  10.5 mm above the detector, 173 inside the light shielded test harness.
- 174

The  $\beta$ - particle spectra were collected with the test harness located in a TAS Micro MT climatic 175 176 cabinet for temperature control. The shaping amplifier, MCA, and voltage source were located 177 outside of the climatic cabinet and operated at ambient laboratory temperature. The test harness was 178 purged with dry  $N_2$  before it was placed in the climatic cabinet, and thereafter the climatic cabinet was 179 continually fed with dry  $N_2$  to ensure the test environment remained dry. The temperature inside the 180 climatic cabinet was raised from laboratory temperature ( $\approx 20$  °C) in 20 °C steps to 100 °C. At each 181 20 °C temperature step, a minimum 30 minutes waiting time was applied to allow the contents of the 182 test harness to reach thermal equilibrium before the climatic cabinet temperature was raised further. 183 <sup>63</sup>Ni β<sup>-</sup> particle spectra were accumulated at 100 °C and then in 20 °C decrements, down to -20 °C. 184 Each  $\beta$ - particle spectrum was accumulated with a live time limit of 1800 s and with the MCA

operated in 4096 channels mode. The detector was operated with an applied potential difference of 50
 V and the shaping amplifier was set to a shaping time of 2 µs. The potential difference and shaping

- 187 time were selected in light of preliminary measurements made with the detector and system: when the 188 detector had been previously used as a prototype detector for X-ray spectroscopy, optimum energy
- resolution was achieved (as measured by the full width at half maximum of a Mn K $\alpha$  (5.9 keV)
- 190 photopeak from an <sup>55</sup>Fe radioisotope source) with similar settings [22]. The optimum shaping time
- occurs when the quadratic sum of series and parallel white noise reach a minimum value [33]. An
   exposition of the different electronic noise contributions of a non-avalanche X-ray spectrometer can
- be found in refs. [34, 35, and 36]. At each temperature, a spectrum was collected without an electrical
- 194 potential difference applied across the detector to check for the presence of any residual electric field
- 195 sweeping charge in the detector. A characteristic shoulder attached to the so called zero energy noise
- 196 peak without electrical potential applied externally would have been indicative of such polarisation.
- 197 When the detector was confirmed to be unpolarised, the potential difference was applied (50 V) and  $\beta^{-1}$
- 198 particle spectra were collected.  $\beta^2$  particle spectra were repeated (at constant temperature, applied

- 199 potential difference, and shaping time) to check that each spectrum was stable and repeatable. The
- 200 turn-around time (the time to save a spectrum and start to acquire another) was < 2 minutes. To
- 201 investigate any possible instability in performance, the turn-around procedure was repeated (i.e.
- 202 spectra were collected repeatedly) until a second spectrum was obtained which was identical to that
- which had been acquired immediately previously. Thereafter, the applied potential difference was 203
- 204 removed and the detector was checked for polarisation effects. If polarisation was detected (by the
- 205 presence of a shoulder attached to the zero energy noise peak), the check was repeated until the 206 apparent polarisation had dissipated.
- 207
- 208 A series of repeated spectra accumulated at 100 °C are presented in Figure 3. In Figure 3 most of the
- 209 counts of the zero-energy noise peak have been removed from the accumulated spectra; a low energy
- 210 threshold was set on the MCA after establishing the noise peak's position, and for clarity, the counts
- 211 accumulated below this threshold prior to its implementation are not presented.



212

213 **Figure 3.** The <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle spectra repeatedly collected, 11 times, at 100 °C. Shown are the  $\beta$ <sup>-</sup>

- particle spectra accumulated: initially (black line); at 32 minutes (red line), at 63 minutes (orange 214
- 215 line), at 94 minutes (yellow line), at 126 minutes (green line), at 157 minutes (cyan line), at 189
- 216 minutes (blue line), at 224 minutes (indigo line), at 278 minutes (violet line), at 338 minutes (magenta
- line), and at 382 minutes (grey line). All spectra were collected with a live time of 1800 s. (The 217
- 218 reader is referred to the web version of this article for the colour form of this figure.)
- 219

220 The  $\beta$ -particle spectra accumulated at 100 °C varied in morphology as a function of time. Two

- 221 identical  $\beta$  particle spectra at 100 °C were first accumulated 382 minutes after the potential difference
- 222 was first applied to the detector. In this time, the endpoint channel number had reduced from channel 223
- 1004 to channel 887, and the total number of counts had reduced and from 104,400 to 54,100. It was
- 224 hypothesised that polarising effects in the detector were responsible for the changing  $^{63}$ Ni  $\beta$ <sup>-</sup> spectrum.
- 225 After the applied potential difference was removed from the detector, it was found that a residual 226 electric field continued to sweep charge in the detector.
- 227

228 The reduction in MCA endpoint channel number and the reduction of counts collected when the 229 spectrum was repeated, without changing the spectrometer's settings, was indicative of a

- 230 progressively reducing electrical potential across the active region of the detector, as a function of
- 231 time. This apparent polarisation of the detector is thought to have been caused by traps in the
- 232 material's bandgap [37] that created space charge regions acting in opposition to the applied field
- 233 across the detector; such a phenomenon has been reported previously in both polycrystalline [38] and
- 234 single crystal [39] diamond detectors. Polarisation phenomena have previously been shown to
- 235 increase as a function of temperature, up to 55 °C, in CdTe radiation detectors [40]. Studies with SiC
- 236 radiation detectors radiation damaged by 6.5 MeV protons and 1.0 MeV neutrons have suggested that
- 237 high temperatures, up to 580 °C, can decrease the time taken to depopulate charge from deep traps
- 238 and thus counter this polarisation effect [41].
- 239
- 240 The de-trapping lifetime,  $\tau_d$ , can be expressed as [42, 43],
- 241

(1)

242 
$$\tau_d = \frac{1}{N_c \sigma v_{th} exp\left(\frac{-E_t}{kT}\right)},$$

243

244 where  $N_c$  is the density of states,  $\sigma$  is the capture cross section of the trap,  $v_{th}$  is the drift velocity in the 245 applied potential difference,  $E_t$  is the activation energy of the trap, k is the Boltzmann constant, and T 246 is the temperature of the detector in Kelvin. Equation 1 implied that at higher temperatures the de-247 trapping lifetime increased and consequently trapping lifetime decreased. However, a shorter 248 trapping lifetime implied that there would have been more free traps and that the rate of charge 249 trapping would have been increased causing carrier mobility to decrease and consequently decreased 250 charge collection efficiency. Intrinsic charge carrier concentration is also proportional to  $T^{3/2}$  [44]. At 251 100 °C, the greater intrinsic concentration of charge may have filled traps at a rate greater than the 252 rate of any trap depopulation, this would have caused the polarisation to build up and disrupt the 253 electrical field across the detector. This would have created a potential barrier for charge carriers 254 transiting the affected regions of the detector. Deep traps also act as recombination centres 255 eliminating charge carriers created in the detector; they are less affected by thermal activation so 256 persist for long periods and are often not easily depopulated. Deep traps in diamond are emptied at 230 °C  $\leq T \leq 280$  °C [45, 46] and were thus thought to less likely to be responsible for the unstable 257 258 performance exhibited by the presently reported detector at 100 °C. However, shallow traps are 259 emptied at lower temperature ( $T \lesssim 80$  °C) [46]; which would account for a continuously evolving trapping population polarizing the detector at 100 °C. Further characterisation of the spectrometer at 260 100 °C showed that the polarisation effect could be reversed by removing the applied potential 261 262 difference from the detector for a short period ( $\approx 1$  hour). After the detector was depolarised in this 263 fashion, when the 50 V potential difference was reapplied, the detector would repolarise as before. 264 Further detailed study of the apparent polarisation phenomenon is warranted. A detailed study of the 265 roles of the contact metallisation and diamond surface preparation in the observed behaviour may 266 reveal that a key role is played by the detector fabrication process. 267

268 The  $\beta$  particle spectra collected at  $T \le 80$  °C are presented in Figure 4. The spectra are shown in their raw form with the MCA scale uncalibrated; i.e. the channel width (in energy and charge terms) 269 270 of the spectra will have changed with temperature by virtue of variation in the preamplifier's 271 conversion factor with temperature. At each temperature, the spectra were repeatable and the 272 changing  $\beta$  particle spectra morphology, which was indicative of polarisation, was absent; however, a 273 residual electric field remained in the detector when the applied potential difference was removed. This residual electric field dissipated in less than 30 minutes. The change in position of the apparent 274 275 endpoint on the MCA's scale was primarily due to the change in performance of the preamplifier as a 276 function of temperature. However, some contribution may have been from variation of the average 277 electron-hole pair creation energy, which is known to vary as a function of temperature in other 278 materials [47, 48, 49]. The number of counts in each spectrum decreased as a function of 279 temperature. At 80 °C, the total number of counts collected in each spectrum was  $\approx$  78300 with an 280 endpoint channel of 1080; at -20 °C the total number of counts collected were  $\approx 50300$  with the 281 endpoint channel of 840. Some of the change in the position of the endpoint channel number will have been due to change in the conversion factor of the preamplifier with temperatures. However, 282 283 given that the live time limit and shaping amplifier shaping time were unchanged between each 284 spectrum; and given that the apparent endpoint of the lower temperature spectra reduced towards 285 lower channel numbers on the MCA scale, a corresponding increase in the number of counts per 286 channel would have been expected, as each MCA channel would have had to encompass a larger 287 range of  $\beta$ -particle energies. However, the opposite was observed; the number of counts per channel 288 remained either relatively constant or slightly reduced when the endpoint channel reduced with 289 temperature (Figure 4). The reason for this is not fully understood at present; it may have been 290 caused by the higher temperatures ionising trapping centres which removed space charge. In so 291 doing, the effective volume of material available for electron-hole pair creation leading to detection of 292 the incident radiation through the induction of charge on the contacts of the detector by virtue of the 293 movement of the charge carriers within the detection medium may have been larger at the higher 294 temperatures. Alternatively, or in addition, higher temperatures may have enabled collection of a 295 greater portion of the charge created in the diamond outside of the volume defined by the contact

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296 metallisation. Based on the change in number of counts, the effective active volume of the diamond

would have had to be 35 % greater at 80 °C than it was at -20 °C. Diffusion of charge from low field

regions at higher temperatures may have contributed at least in part to a greater effective active volume under those conditions but the quantitative extent to which it did is presently unknown; more

300 evidence would be required draw definitive conclusions as to the extent of this contribution.

500 Evidence would be required draw definitive conclusions as to the extent of this contribution.

301 Furthermore, additional investigation of the phenomena reported here is essential if a complete

302 understanding of the detector is to be achieved



303

**Figure 4.** <sup>63</sup>Ni  $\beta^{-}$  particle spectra accumulated with the diamond detector from -20 °C  $\leq T \leq 80$  °C. 305 T = 80 °C (black line), T = 60 °C (red line), T = 40 °C (orange line), T = 20 °C (yellow line), T = 0 °C 306 (green line), and T = -20 °C (cyan line). The spectrum live time limit was 1800 s in each case. It

should be noted that the channel width in units of energy (or charge) for each spectrum is not
identical. (The reader is referred to the web version of this article for the colour form of this figure.)

310 2.4. <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle spectrum modelling

The CASINO (monte CArlo SImulation of electron trajectory in sOlids) computer program [50, 51] 311 312 was used to simulate the paths and the energy losses of the  $\beta^2$  particles emitted from the <sup>63</sup>Ni  $\beta^2$ 313 particle source as they traversed the 1  $\mu$ m Ni overlayer and the 10.5 mm dry N<sub>2</sub> atmosphere between the <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle source and the top of the detector. Each simulated  $\beta$ <sup>-</sup> particle was allocated an 314 315 energy between 1 keV and the <sup>63</sup>Ni endpoint energy of 66 keV, in 1 keV increments. The simulated spectrum of  $\beta^{-}$  particles emitted from the <sup>63</sup>Ni  $\beta^{-}$  particle source was dependent upon the emission 316 probability of each  $\beta$  particle's energy when emitted by <sup>63</sup>Ni and included the effects of self-317 absorption within the <sup>63</sup>Ni radioisotope source [52]. A total of 18,549,380  $\beta$  particles across the 1 318 319 keV to 66 keV  $\beta^{-}$  particle energy range were simulated. The total number of  $\beta^{-}$  particles simulated 320 was selected in order to provide good counting statistics across the whole of the energy range rather 321 than to reflect the activity of any specific  ${}^{63}Ni \beta^{-}$  particle source. The density of the 1  $\mu$ m Ni overlayer was modelled as 8.908 g cm<sup>-3</sup> and the density of the  $N_2$  atmosphere was modelled as 0.0012 g cm<sup>-3</sup>. 322 323 The physical model selected for the modelling was 'Mott'; the developers of CASINO have reported 324 the associated backscattering coefficients to be in better agreement with experimental results at low 325 electron energies (< 10 keV). The Mott model has also been found to be more accurate than the Rutherford cross section for low Z materials [51]. Of the three Mott models available to CASINO 326 327 'Mott by Interpolation' was selected as this model was computationally faster and known to be more 328 accurate than the other selectable Mott models [50]. Other pertinent CASINO settings were: 329 Ionisation Potential – Joy and Luo [53]; Random Number Generator – the Press et al. [54]; Directing 330 Cosine – Drouin et al [51]; and Effective Section Ionisation – Casnati et al. [55]. The computed 331 trajectories of the simulated  $\beta$  particles were used to calculate their residual energy after interacting 332 with the 1  $\mu$ m Ni overlayer and the dry N<sub>2</sub> atmosphere above the detector's face. The simulated  $\beta$ -333 particles at the face of the detector were analysed and the distribution of those energies produced the 334 spectrum of  $\beta^{-}$  particles incident upon the face of the detector. Both  $\beta^{-}$  particle spectra (i.e. emitted 335 from source and incident on detector) are presented in **Figure 5**. The endpoint  $\beta$ -particle energy 336 emitted from the <sup>63</sup>Ni layer of the  $\beta$ - particle source was 66 keV, but after traversing the 1  $\mu$ m Ni 337 overlayer and the dry N2 layer the endpoint energy of the spectrum reduced to 63 keV. It should also

be noted that the temperature range reported here did not change the density of the Ni overlayer or the dry  $N_2$  atmosphere sufficiently to alter the results of the CASINO modelling.



340

**Figure 5.** Simulated  $\beta^2$  particle spectrum emitted from the <sup>63</sup>Ni layer of the  $\beta^2$  particle source



having been attenuated by the 1  $\mu$ m protective Ni overlayer and 10.5 mm of dry N<sub>2</sub> atmosphere (open squares).

345

The CASINO program was also used to calculate the quantum detection efficiency of the detector (i.e.

the fraction of the energy deposited in the detector by  $\beta^2$  particles incident upon the face of the detector, as a function of energy up to 66 keV). To do this, 4000  $\beta^2$  particles at each energy from 1

keV to 66 keV in 1 keV steps were simulated as incident upon a) the top metalized contact, and b) the

350 non-metalized portion of the detector's top face, i.e.  $8000 \beta^2$  particles were simulated at each energy.

351 The number of  $\beta^{-}$  particles simulated was chosen in order to give good counting statistics rather than

352 to reflect any specific individual case of illumination of the detector with  $\beta^{-}$  particles. At each energy,

both simulations were combined in the ratio of the areas of the detector's face covered (49%) and not

354 covered (51 %) by the contact. As shown in **Figure 6**, the fraction of  $\beta^2$  particle energy deposited in

the detector was 0.49 at 1 keV and this rose to 0.97 at 66 keV. However, it should be noted that this

356 was calculated based on the assumption that the entire thickness (i.e.  $500 \,\mu\text{m}$ ) of the diamond usefully

contributed to the quantum detection efficiency; this resulted in an overestimate of the detector's
 quantum detection efficiency since, as will be shown, the detector actually had a 4.5 µm deadlayer in

the diamond at its top. Furthermore, it should be noted that the values stated and presented as part of

360 the figure include backscattering losses.



361



 $(500 \ \mu m)$  of the diamond contributed usefully to the quantum detection efficiency.

364

In order to predict the  ${}^{63}$ Ni  $\beta^{-}$  particle spectrum that would be detected by the spectrometer, the

366 CASINO simulations in Figure 5 and Figure 6 were combined. The resultant spectrum excluded any

367 pulse pile up and detector edge effects, as well as the noise processes (Fano, electronic, and any

368 incomplete charge collection noise) present in the spectrometer. The results for spectra at selected

369 temperatures (80 °C, 20 °C, and -20 °C) are presented in Figure 7; similar results were obtained for 370 all temperatures but three temperatures were selected for clarity of presentation. For comparison of 371 the simulated spectrum and the experimentally detected spectra, the predicted spectrum and the 372 experimentally detected spectra at each temperature were normalised. For this, the predicted 373 spectrum was normalised to the mean number of counts in the generally constant region between 10 374 keV and 20 keV. The experimentally detected spectra were normalised to the mean number of counts 375 in the generally constant region between 15 keV and 30 keV; this avoided counts from the zero noise 376 energy peak adding to the averaged count. Experimental spectra were calibrated using the endpoint

- 377 energy from the simulated spectrum, the position of the zero energy noise peak, and by taking account
- 378 of the relative probability of detection as indicated by the number of counts that were detected.



**Figure 7.** <sup>63</sup>Ni  $\beta^{-}$  particle spectra experimentally accumulated (black line) at (a) T = 80 °C, (b) T = 20 °C, and (c) T = -20 °C, as compared with that predicted by the simulation (open squares). To enable morphological comparison, the spectra are presented in terms of relative counts and the energy axis has been calibrated based on the position of the zero energy noise peak, the endpoint energy predicted by the simulation, and by taking account of the relative probability of detection as indicated by the number of counts that were detected.

388 As can be seen from **Figure 7**, the simulated and experimentally measured spectra had substantially 389 different morphologies. The particular morphological difference shown is evidence of an inactive 390 "dead" layer within the diamond at its front. Electron-hole pairs generated in a deadlayer are not able 391 to move in the normal way so as to induce on the contacts of the detector the charge that they would 392 have induced had they been created in an active region of the detector. Consequently, those charge 393 carriers do not contribute to the accumulated spectrum. Such deadlayers (where generated charge is 394 lost) have been reported at the detector/contact interface of other semiconductor radiation detectors 395 (e.g. ref. [56]). Further modelling in CASINO was used to compute the apparent thickness of the 396 deadlayer in the present diamond detector.

397

398 The simulations which led to the production of **Figure 6** were rerun to include the presence of a

399 deadlayer within the diamond immediately proximate to the front contact. The thickness of deadlayer

- 400 was varied from non-existent to  $4.5 \,\mu m$  thick, in  $0.5 \,\mu m$  increments. This produced new quantum
- 401 detection efficiency predictions which included the effects of a deadlayer. Each simulation was then

402 combined with the simulations of Figure 5 to produce a number of predicted spectra for different403 deadlayer thicknesses.

404

A match between the predicted and experimentally detected spectra was accomplished when a 4.5 μm
deadlayer at the top of the detector was included. The simulated spectrum (with 4.5 μm deadlayer)
and the experimentally detected spectra was normalised to the mean number of counts in the generally
constant region between 17 keV and 25 keV. The experimental spectra were then energy calibrated
using the endpoint energy from the simulated spectrum and the position of the zero energy noise peak.
In positioning the endpoint of the simulated spectrum, the relative probability of detection, indicated

- 411 by the number of detected counts was also considered.
- 412

413 It was recognised that inclusion of a single deadlayer thickness across all temperatures was likely to

414 be an oversimplification (the thickness of recombination regions are known to be temperature 415 dependent in ultraviolet detectors [57]) but the approach employed here was considered adequate for

416 present purposes. In future, synchrotron measurements could be employed to measure the profile and

417 thickness of the dead region by measuring the X-ray quantum efficiency as a function of X-ray energy

418 [58]. The calculated quantum detection efficiency for the detector, including the 4.5 µm deadlaver, is

419 shown in **Figure 8**; for comparison, the quantum detection efficiencies which would have resulted had

420 the deadlayer been 1.5  $\mu$ m and 3.0  $\mu$ m are also included. With a 4.5  $\mu$ m deadlayer, the quantum

421 detection efficiency (fraction of  $\beta^2$  particle energy usefully deposited in the active region of the

422 detector) was 0.02 and 0.79 at 30 keV and 66 keV, respectively, cf. 0.87 and 0.97 without a dead

423 layer. The spectra predicted to be detected when the 4.5  $\mu$ m deadlayer was included in the

424 simulations are shown in **Figure 9** together with the experimentally detected  ${}^{63}$ Ni  $\beta$ - particle spectra;

425 for clarity of presentation, only the spectra at temperatures of 80 °C, 20 °C, and -20 °C are shown. As

426 was the case for **Figure 7**, the spectra are presented in terms of relative counts, based on the position

427 of the zero energy noise peak, the endpoint energy predicted by the simulation, and by taking account

428 of the relative probability of detection as indicated by the number of counts that were detected.



429

430 **Figure 8**. Quantum detection efficiency for the detector as a function of  $\beta^{-}$  particle energy when no 431 deadlayer (open squares), and deadlayers of 1.5 µm (open diamonds), 3 µm (open triangles), and 4.5 432 µm (open circles) are included.

433

434 The apparent endpoint energy of the detected spectra ( $\approx 46 \text{ keV}$ ) was lower than the endpoint energy 435 of the spectrum illuminating the detector (63 keV) because the detector's top contact and the 4.5  $\mu$ m 436 deadlayer attenuated the energy of the  $\beta$  particles before they could reach the active region of the 437 detector. Electronic grade single crystal CVD diamond wafers are scaif polished (a mechanical

438 process where diamond particles are used to abrasively polish the wafer's surface) [59].

439 Imperfections caused by the polishing process can reach 10 µm below the polished surface [60]. It is

440 hypothesised that this may have altered the electrical characteristics of the diamond resulting in the 441  $4.5 \,\mu\text{m}$  thick deadlayer at the top of the detector.

442





**Figure 9.** Comparison of <sup>63</sup>Ni  $\beta$ <sup>-</sup> particle spectra predicted when a 4.5  $\mu$ m deadlayer was included 445 446 (open squares) and experimentally detected (black line) at temperatures of (a) 80 °C, (b) 20 °C, and 447 (c) -20 °C, as compared with that predicted by the simulation (open squares). To enable 448 morphological comparison, the spectra are presented in terms of relative counts and the energy axis 449 has been calibrated based on the position of the zero energy noise peak, the endpoint energy predicted

450 by the simulation, and by taking account of the relative probability of detection as indicated by the

- 451 number of counts that were detected.
- 452

453 Given that the front and rear of the detector were substantially similar, the presence of a similar

454 deadlayer immediately proximate to the bottom contact was hypothesised but could not be concluded

455 from the present data. The detector was sufficiently thick that the presence (or absence) of a

456 comparable deadlayer at the bottom would not have changed the quantum detection efficiency within 457 the energy range applicable for the present work; hence, regardless of the presence or absence of a 4.5

458 µm deadlayer at the bottom of the detector, the detected spectrum would be the same. Further

459 CASINO simulations which included a deadlayer proximate to the bottom contact were performed

460 and showed that the spectra predicted to be detected were identical regardless of the inclusion or

461 exclusion of such a 4.5 µm thick bottom deadlayer.

462

### 463 3. Discussion, conclusions, and further work

464 An electronic grade single crystal CVD diamond detector was investigated for its performance as part

of an electron ( $\beta$  particle) spectrometer. The detector was connected to a custom-built low-noise 465 466 charge-sensitive preamplifier which was itself connected to a standard electronics chain. The detector

was illuminated by a <sup>63</sup>Ni radioisotope  $\beta$ - particle source. The spectrometer was investigated across 467

the temperature range -20 °C  $\leq$  T  $\leq$  100 °C. At 100 °C, the detector experienced significant 468

469 polarisation. Performance was stable at each of the lower investigated temperatures (i.e. -20 °C  $\leq T \leq$ 

470 80 °C) and polarisation did not impede the collection of spectra across this temperature range.

471

472 The detector's performance was modelled using CASINO. By comparison between the spectra

473 predicted by the model and those experimentally accumulated, it was determined that the detector had

474 a 4.5  $\mu$ m thick deadlayer in the material in direct proximity with the front contact. It was

- 475 hypothesized that imperfections were introduced into this region of material during a polishing
- 476 process used by the supplier of the diamond from which the detector was fabricated.

### 477

478 These results show that spectroscopic detection of relatively soft (energy  $\leq 63$  keV) electrons ( $\beta$ 479 particles) is possible at elevated temperatures ( $\leq 80$  °C) using a single crystal CVD diamond detector. 480 The presently reported detector by virtue of being thick (500  $\mu$ m) had excellent quantum detection 481 efficiency compared with most other wide bandgap semiconductor detectors proposed for space-borne 482 electron spectrometers. Had the deadlayer been absent, with the presently reported contact 483 configuration, the fraction of  $\beta^{-}$  particle energy deposited in the detector would have been 0.49 at 1 484 keV which would have risen 0.97 at 66 keV. Even so, with the deadlayer being present, detection 485 efficiency was 0.79 at 66 keV; at 100 keV and 300 keV the detection efficiency of the detector would 486 rise to 0.95 and 0.98, respectively. Other wide bandgap detector materials have been reported as 487 proof of concept or prototype  $\beta^2$  particle detectors that are temperature tolerant and radiation hard. A 488 prototype GaAs  $p^+$ -i- $n^+$  mesa diode detector (10 µm active i layer) [15] reported a detection efficiency 489 of 0.73 at 70 keV. In that case, the detection efficiency of this detector per unit thickness was 490 bolstered by the greater stopping power of GaAs cf. diamond. However, the efficiency of the GaAs 491 detector declined to 0.49 at 100 keV (100 keV  $\beta$  particles were less likely to fully deposit their energy 492 in the active i layer). Other reported emergent semiconductor detectors considered suitable for deep 493 space electron spectrometry are InGaP [19] and AlGaAs [18]. The active i layers for both reported 494 detectors were thin; 5 µm for InGaP and 3 µm for AlGaAs, consequently and despite the superior 495 stopping power of the materials used in the detectors, the  $\beta$ -particle detection efficiencies were lower 496 than for the presently reported diamond detector. The efficiency of the InGaP detector was 0.45 at 62 497 keV and the AlGaAs detector's peak efficiency (0.53) occurred at 38 keV. Thicker i layers would be 498 required for both of these detectors to improve their detection efficiencies.

499

500 Despite the specific limitations of the detector reported here, the results show that diamond is a

501 promising candidate material for future solid state electron spectrometers which will be required for

502 various terrestrial and space applications. Whilst polarisation limited operation of the detector at

100 °C, the wide bandgap of diamond (5.47 eV [61]) suggests that operation at even higher
 temperatures is likely to be possible if the present polarisation problem can be eliminated. This

505 potential for operation at extremely high temperatures and the expected radiation hardness of diamond

506 detectors are key motivating factors for its study by researchers developing next generation

507 instrumentation for space science. One key challenge to address is understanding the nature and

508 mechanisms of the apparent polarisation phenomenon: detectors made from different single crystal

- 509 CVD diamonds should be characterised and it may be advantageous to measure the change in
- 510 performance of the present detector in detail at temperatures 80 °C  $< T \le 100$  °C in order to identify 511 the transitionary behaviours between stable and polarised performance. Another potentially valuable
- avenue of exploration would be in seeking to understand how surface preparation changes the
- 513 properties of the diamond detector close to its surface; a reduction in thickness (or preferably,
- elimination) of the deadlayer would improve the quantum detection efficiency at soft energies ( $\leq 25$

515 keV). It is also informative to compare the diamond detector's response to  $\beta$  particles with that for

516 photons of similar energies. The detection efficiency of the diamond detector for  $\beta$ -particles is high

517 because of its thickness. Despite the detector being thick, the detector's detection efficiency for 518 photons would be low, by virtue of carbon's low atomic number. For example, at 66 keV the

518 photons would be low, by virtue of carbon's low atomic number. For example, at 66 keV the 519 detector's  $\beta^{-}$  particle detection efficiency was 0.79; however, its quantum detection efficiency for 66

keV photons was computed to be  $0.71 \times 10^{-5}$ . This suggests that this type of detector could find

521 particular utility in detecting electrons or  $\beta^2$  particles in mixed electron/photon radiation environments

- 522 as the need to discriminate or subtract the photon background would be reduced cf. for higher atomic
- 523 number detectors.
- 524

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- 533

# 534 Data Statement

- 535 Data underlying this work are subject to commercial confidentiality. The authors regret that they 536 cannot grant public requests for further access to any data produced during the study, however the key 537 findings are included within the article.
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Journal

- A proof-of-principle high temperature tolerant electron spectrometer is reported
- The spectrometer utilised a prototype chemical vapour deposition diamond detector
- Electrons from a <sup>63</sup>Ni radioisotope  $\beta$ <sup>-</sup> particle source detected spectroscopically
- The instrument operated successfully at temperatures  $\leq 80 \text{ }^{\circ}\text{C}$

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# **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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