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Article (Accepted Version)

Bodie, C S, Lioliou, G, Lefeuvre, G and Barnett, A M (2021) A single crystal chemical vapour deposition diamond soft X-ray spectrometer. Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors, and Associated Equipment, 989. a164950 1-7. ISSN 0168-9002

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A single crystal chemical vapour deposition diamond soft X-ray spectrometer

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Abstract

A 2 mm width by 2 mm breadth by 0.5 mm thick electronic grade single crystal diamond grown via chemical vapour deposition was configured as a metal-semiconductor-metal X-ray detector. The detector was electrically characterised before it was connected to a bespoke charge-sensitive preamplifier of low-noise. An ⁵⁵Fe radioisotope X-ray source was used to illuminate the detector with soft (5.9 keV and 6.49 keV) X-rays. The detector and preamplifier were temperature controlled and operated at 20 °C. The energy resolution (full width at half maximum) of the diamond spectrometer was 2.48 keV at 5.9 keV. Shaping time noise analysis conducted on the X-ray spectra found dielectric noise to be the largest electronic noise contribution for all but the shortest of shaping times, when series white noise dominated. To the best of the authors' knowledge, this is the first report of a diamond detector's spectroscopic photon counting response to soft (< 10 keV) X-rays.

1. Introduction

Radiation-hard wide bandgap semiconductor radiation detectors can offer advantages over Si detectors in photon counting X-ray spectrometers that need to operate in harsh radiation environments or environments of high temperature (> 20 °C). The cooling apparatus that is normally required to limit the thermally stimulated leakage currents of narrower bandgap detectors, and the shielding which can be necessary to protect detectors from intense radiation, can be reduced or discarded when high temperature tolerant and radiation-hard detectors are used [1]. Some harsh environments where wide bandgap semiconductor radiation detectors could be beneficial are space [2 - 4], gas turbine engines [5] [6], nuclear power plants [7, 8], and in the vicinity of deep-sea hydrothermal vents [9]. Wide bandgap semiconductor X-ray detectors that are capable of photon counting X-ray spectroscopy at elevated temperatures (up to 100 °C) include GaAs [10 - 13], Al_{0.8}Ga_{0.2}As [14 - 16], and SiC [17]. Diamond's properties of chemical inertness [18 - 20], intrinsic resistance to radiation damage [21], and wide bandgap ($E_g = 5.47$ eV [19, 22]) suggest that a diamond X-ray spectrometer could operate at elevated temperatures (≥ 20 °C) and without radiation shielding even in harsh environments. However, to date, there have been no reports in the literature of diamond radiation detectors being used in soft X-ray (< 10 keV) photon counting X-ray spectrometers.

Previously, diamond radiation detectors have responded to X-ray photons. Polycrystalline diamond detectors were shown to generate photocurrent when illuminated with 50 keV – 250 keV and 6 MeV X-rays [23], and a diamond detector's X-ray responsivity has been measured in the wavelength range 125 Å to 240 Å [24]. A linear relationship of photocurrent with X-ray radiation dose rate has been demonstrated with electronic grade polycrystalline chemical vapour deposition (CVD) diamond detectors [25]. Natural diamonds have also been shown to produce a photocurrent response when illuminated with 10 keV and 45 keV X-rays [26]. The photocurrent response and tissue equivalence of diamond detectors prompted the investigation of them for radiation dosimetry [27, 28]. The study that first achieved a spectroscopic response to fast neutrons with a single crystal CVD diamond also showed the detector to be capable of detecting 22.16 keV Ag K α photons emitted by a ¹⁰⁹Cd radioisotope X-ray/ γ -ray source [29]. In other work, the current mode response was obtained from a 35 μ m thick single crystal CVD diamond detector placed in the beamline of a synchrotron; the detector was used to study the X-ray beam intensity at two different X-ray energies (10.6 keV and 12.5 keV) [30]. X-ray beam profile monitoring with a polycrystalline CVD diamond detector has also been demonstrated at energies of 17.5 keV and 19.6 keV (Mo K α and K β) [31]. Somewhat tangentially, but importantly for the in situ

measurement of diamond films during the growth process, soft X-ray emission studies of HFCVD diamond films have been reported [32].

Here, a photon counting spectroscopic X-ray detector was fabricated from CVD-grown diamond purchased from Element 6 Ltd. [33]. After electrical characterisation, the detector was paired with a bespoke charge-sensitive preamplifier of low-noise and onwards to a standard readout electronics chain. The detector was illuminated with X-rays emitted from an ^{55}Fe radioisotope source which gave Mn K α (5.9 keV) and Mn K β (6.49 keV) soft X-ray photons. With this experiment, X-ray spectra at energies < 10 keV have been accumulated with a diamond detector and are reported for the first time.

2. Diamond detector

An electronic grade CVD single crystal diamond (2.0 mm width by 2.0 mm breadth by 0.5 mm thickness) grown by Element 6 Ltd. [33] was cleaned in an acid bath to remove organic and non-organic surface contaminants. Square contacts (1.4 mm width by 1.4 mm breadth) were formed by sputtering both a Ti layer (50 nm thick) and an Ag layer (200 nm thick) on to the front and rear of the detector, in turn. After deposition of each contact, the detector was annealed at 400 °C. The detector was mounted in a TO-39 package using silver epoxy adhesive [34] and wirebonded. A schematic diagram of the diamond detector is shown in Fig. 1.

Fig. 1. Schematic of the diamond X-ray detector showing its connections to the biasing power supply and the preamplifier (not to scale).

3. Detector characterisation

3.1 Leakage/dark current measurements

The packaged detector's leakage current was investigated as a function of applied electrical potential difference. The detector was placed in an electromagnetically screened test harness in a TAS Micro MT Climatic Cabinet (kept at 20 °C). A Keithley 6487 picoammeter/voltage source was used to apply electrical potential and measure the leakage current. The detector was operated in a dry N₂ environment to mitigate against known effects of humidity on TO packages [14]. Electrical potential difference was applied across the detector in 5 V steps up to 100 V in both polarities with the leakage current recorded after a 5 second wait at each step. This process was automated with LabVIEW software. At applied potential differences up to 100 V, the leakage current of the packaged detector was less than the noise floor of the picoammeter (i.e. < 0.4 pA). This implied a leakage current density of < 20 pA cm⁻² at an electric field strength of 2 kV cm⁻¹, assuming that the field was solely between the area of the two contacts, or < 10 pA cm⁻² if the whole 2.0 mm by 2.0 mm area of the diamond was active. Such leakage current densities are lower than reported by other researchers using CVD diamond radiation detectors. For example, a single crystal CVD diamond detector grown by Ila Technologies Pte. Ltd., Singapore had a leakage current density of 400 pA cm⁻² at the same electric field strength. and at 25 °C [35]. Tsubota et al. reported two 0.5 mm thick single crystal CVD diamond detectors with leakage densities

of 140 pA cm⁻² and 30 pA cm⁻² at an electric field strength of 2 kV cm⁻¹ and at a temperature of 20 °C [36]. Although the upper limit of the leakage current densities reported here for the present detector are lower than has been previously reported with diamond, they may be still be far greater than those reported for some other types of wide bandgap semiconductor radiation detectors (only an upper limit to the leakage current density could be determined and that upper limit was greater than the leakage current densities measured for some detectors made of other materials). For example, a leakage current density of 1 pA cm⁻² has been achieved with 4H-SiC detectors at a temperature of 27 °C and with an electric field strength of 104 kV cm⁻¹ [37]. Low leakage currents are necessary to limit the parallel white noise component of X-ray spectrometers, particularly when longer amplifier shaping times are selected [38].

3.2 Capacitance measurements

The capacitance of the packaged detector was measured with a HP 4275A Multi Frequency LCR meter as a function of applied potential difference. The packaged detector was placed in an electromagnetically screened test harness and after a ‘zero open check’ the LCR test signal was set to 1 MHz frequency and 50 mV rms magnitude. As per the leakage current measurements the test harness was located in a TAS Micro MT climatic cabinet at 20 °C with a dry N₂ environment. The capacitance of the detector was measured in 5 V steps up to a maximum applied potential difference of 100 V in each polarity. The capacitance was found to be independent of the applied potential difference; it was 870 fF ± 20 fF across the potential difference range measured. The capacitance contribution of the detector’s packaging was also estimated (by measuring the capacitance of a similarly packaged diamond device with its wirebond removed) and found to be independent of applied potential difference; the capacitance of the detector’s packaging was 520 fF ± 20 fF in parallel with the detector, thus implying a detector capacitance of 350 fF ± 30 fF. Given the relative permittivity of diamond (5.7) [20, 39], a detector thickness of 500 µm, contacts of 1.4 mm by 1.4 mm, (1.96 mm²), and assuming that the capacitance characteristics of the detector could be approximated to those of a parallel plate capacitor, a capacitance of 200 fF was expected. The apparent capacitance of 350 fF ± 30 fF, implies a detector contact area of 3.47 mm² ± 0.30 mm², equivalent to an apparent square contact of 1.9 mm by 1.9 mm assuming a detector thickness of 500 µm, or alternatively assuming a contact area of 1.4 mm by 1.4 mm, a detector thickness of 280 µm ± 40 µm would be implied. It is possible that the greater than expected apparent capacitance was a result of edge effects around the detector’s contacts. Capacitance sources associated with detector packaging and that were not included in the 520 fF ± 20 fF estimated, included the wirebond and the silver-loaded epoxy used in die attach; these sources may have also contributed to the measured capacitance.

4. Radiation detection characterisation

4.1 X-ray Spectroscopy

The quantum detection efficiency of the detector to X-rays was required to fit Gaussians to the accumulated X-ray spectra. The quantum efficiency of the detector was calculated using the Beer Lambert Bouguer Law for light attenuation and absorption,

$$QE = \prod_{i=1}^{i=n} \exp(\mu_i \rho_i t_i) (1 - \exp(-\mu_{en} \rho t)), \quad (1)$$

where the first exponential term on the right side of Eqn. 1 is the attenuation of photons in the i^{th} dead layer in front of the detector’s active region. μ_i is i^{th} layer’s mass attenuation coefficient, ρ_i is that i^{th} layer’s density, and t_i is that i^{th} layer’s thickness. The second exponential term in Eqn. 1 is the absorption of photons in the active region. Here, μ_{en} is the mass absorption coefficient of the active region (diamond), ρ is the density of the active region, and t is the thickness of the active region [40]. The dead layers were considered to be solely the Ag and Ti of the front contact, in the present case.

The XCOM database, produced by NIST [41] was accessed to obtain mass attenuation and absorption coefficients for X-ray photons in the relevant materials. The quantum detection efficiency of the detector is presented as a function of X-ray energy in Fig. 2. The discontinuities between 3.35 keV and 3.81 keV and at 4.97 keV are caused by the Ag L and Ti K absorption edges of the detector's front contact.

Fig. 2. The diamond detector's calculated detection efficiency at soft X-ray energies, E , ($1 \text{ keV} \leq E \leq 10 \text{ keV}$)

The detector was paired with a bespoke charge-sensitive preamplifier of low noise. A similar design of preamplifier was first reported by Bertuccio et al. [42]. The feedback resistor in the preamplifier was removed and the leakage current of the detector was discharged through the input Junction Field Effect Transistor (JFET) which was slightly forward biased. The JFET used in this preamplifier was a Vishay 2N4416A Si JFET [43]. The detector and preamplifier were housed in an electromagnetically screened test harness. Output signals from the preamplifier were shaped and amplified by an Ortec 572A shaping amplifier before being fed to an Ortec EASY 8k multi-channel analyser (MCA). The detector's potential difference was applied by a Keithley 6487 voltage source.

Soft (5.9 keV and 6.49 keV) X-rays were produced by an ^{55}Fe radioisotope X-ray source. The ^{55}Fe source, of 106 MBq activity, was placed inside the test harness $\approx 6 \text{ mm}$ above the detector. A 0.14 mm Al absorber (attenuating 99 % of the 5.9 keV photons) was positioned between the source and the detector to limit the photon flux incident on the detector. All spectra were accumulated with the test harness located inside a TAS Micro MT climatic cabinet at 20°C . Spectra were accumulated at four applied potential differences (25 V, 50 V, 75 V, and 100 V) and the six shaping times available to the shaping amplifier. The shaping times, τ , used to accumulate the spectra were 0.5 μs , 1 μs , 2 μs , 3 μs , 6 μs , and 10 μs . The live time limit of each spectrum was pre-set to 90 s. In each case, the photopeak detected as a result of illumination with the ^{55}Fe source, was the combined characteristic ^{55}Fe X-ray emission (Mn $K\alpha$ (5.9 keV) and Mn $K\beta$ (6.49 keV)) [44]. Therefore, Gaussian fitting was required to disentangle the Mn $K\alpha$ and $K\beta$ emission lines. The fitted Gaussians took account of the relative X-ray emission ratios, and the detector's relative detection efficiency at the emitted X-ray energies. The spectrometer's charge scale was energy calibrated using the centroid of the fitted Mn $K\alpha$ X-ray photopeak and the position of the spectrometer's zero energy noise peak. The relationship between energy and charge was then assumed to be linear. In the accumulation of each spectrum, a low energy threshold was set to limit MCA dead time once the position of the zero-energy noise peak had been established. Once the low energy threshold was set, further counts from the zero-energy noise peak were excluded from the spectrum. The ^{55}Fe X-ray spectrum accumulated with the spectrometer operated at 50 V applied potential difference and with a shaping time of 3 μs selected is presented in Fig 3a. The fitted Gaussians that represent the detected Mn $K\alpha$ and $K\beta$ X-ray components are shown as dotted lines. The dependence of the full width at half maximum ($FWHM$) upon shaping time is shown in Fig. 3b. When 50 V and 75 V potential difference were applied across the detector, the energy

resolutions ($FWHM$ at 5.9 keV) achieved were identical (2.48 keV); at 25 V and 100 V applied potential difference the $FWHM$ at 5.9 keV were 2.56 keV and 2.54 keV, respectively.

Fig. 3. a) The ^{55}Fe X-ray spectrum (solid line) accumulated with 50 V potential difference applied across the detector and the shaping time set to 3 μs . The fitted contributions of the Mn $K\alpha$ and $K\beta$ photons to the spectrum are shown as dotted lines; the dashed line shows the combination thereof. b) The energy resolution ($FWHM$) achieved at 5.9 keV with the detector at a 50 V potential difference as a function of shaping time.

^{55}Fe X-ray spectra collected with the detector operated at four different applied potential differences (25 V, 50 V, 75 V, and 100 V) but with the same shaping time (3 μs) for the shaping amplifier are presented in Fig. 4. To permit the reader to distinguish clearly each of the four ^{55}Fe X-ray spectra, in addition to displaying each spectrum superimposed on the others in Fig. 4a, each spectrum is also presented as a separate plot (25 V in Fig. 4b; 50 V in Fig. 4c; 75 V in Fig. 4d; 100 V in Fig. 4e). The photon count rate within the fitted Mn $K\alpha$ (5.9 keV) peak (not shown for clarity in Fig. 4), but similar to that in Fig. 3a, did not vary significantly with potential difference (or shaping time) thus suggesting that the detection efficiency of the detector was constant across the conditions investigated. The mean count rate in the Mn $K\alpha$ photopeak over the four potential differences and the six shaping times investigated was $1110 \text{ s}^{-1} \pm 30 \text{ s}^{-1}$ (rms error), where the rms error, R , was given by,

$$R = \sqrt{\frac{\sum (x_i - \bar{x})^2}{n}} \quad (2)$$

where x_i is the value of the datum (in this case the count rate) of the i^{th} accumulated spectrum, $\bar{x}$ is the mean of the data (in this case the mean count rate in the fitted Mn $K\alpha$ photopeak of all 24 accumulated spectra), and n is the number of data considered (24 in this case – four applied potential differences each with six shaping times). Background spectra of comparable duration to the accumulated X-ray spectra were accumulated before and after illumination of the detector with the ^{55}Fe radioisotope X-ray source. No background counts, beyond those of the zero energy noise peak, were detected. The detected X-ray flux (both Mn $K\alpha$ and $K\beta$) corresponded to a photocurrent of $0.131 \text{ pA} \pm 0.004 \text{ pA}$ (rms error).

Fig. 4. (a) The ^{55}Fe radioisotope X-ray spectra collected with the diamond detector operated at 25 V (black line), 50 V (blue line), 75 V (red line), and 100 V (green line) applied potential differences. Each spectrum is replicated in a separate plot using the same colour (b) 25 V, (c) 50 V, (d) 75 V, and (e) 100 V. The shaping time was 3 μs in each case. (The colour form of this figure can be found in the web version of the article.)

4.2 Noise analysis

There are three independent noise contributions in a direct detection photon counting X-ray spectrometer: they are Fano noise (related to ionisation processes in the detector [45]); electronic noise, A , [46], and incomplete charge collection noise, R_{ICC} , (a consequence of carrier recombination and trapping [47]). Therefore, the energy resolution (expressed in units of equivalent noise charge (ENC) e^- rms) is,

$$\Delta E (e^- \text{ rms}) = \sqrt{\frac{FE}{\omega} + A^2 + R_{ICC}^2}, \quad (3)$$

where ω is the electron-hole pair creation energy of diamond, E is the energy of an incident X-ray, F is the Fano factor of diamond. Electronic noise can be further broken down into four components, i.e. parallel white noise, series white noise (including induced gate current noise), dielectric noise, and $1/f$ series noise. White noise (series, ENC_{sw} , and parallel, ENC_{pw}) are related to amplifier shaping times, τ ,

$$ENC_{sw} = \frac{B}{q_0} \sqrt{\frac{A_1}{2} 4kT \frac{\gamma}{g_m} C_T^2 \frac{1}{\tau}}, \quad (4a)$$

$$ENC_{pw} = \frac{1}{q_0} \sqrt{\frac{A_3}{2} 2q_0(I_D + I_{JFET})\tau}, \quad (4b)$$

where, for Eqn. 4a, B is a correction for induced gate current noise (0.8), A_1 is 1.85, γ is a dimensionless product of noise resistance and transconductance, g_m , of the preamplifier's input JFET, q_0 is the electron unit charge, and C_T is the total capacitance at the input of the preamplifier's JFET. For Eqn. 4b, A_3 is 1.85, and I_D and I_{JFET} are the leakage currents of the detector and the Si JFET, respectively. In summary, parallel white noise is proportional to the shaping time, whilst series white noise has an inverse relationship with amplifier shaping time. Parallel white noise arises from the leakage currents of the detector and input JFET [38, 46, 48]; series white noise arises from the detector's capacitance, the preamplifier's input transistor capacitance, the feedback capacitance, the test capacitance (if present), and any other stray capacitances present at the input to the preamplifier.

Dielectric and $1/f$ series noise are shaping time independent. The ENC of $1/f$ and dielectric noise are given by Eqns. 5a and 5b, respectively

$$ENC_{1/f} = \frac{1}{q_0} \sqrt{A_2 \pi A_f C_T^2}, \quad (5a)$$

$$ENC_{die} = \frac{1}{q_0} \sqrt{A_2 2kTdC_{die}}, \quad (5b)$$

where A_2 is 1.18, A_f is a characteristic constant of the JFET, C_{die} is a representative capacitance of the lossy dielectrics close to or in contact with the input JFET, and d is the respective dielectric dissipation factor.

The Fano noise of diamond at 5.9 keV was estimated to be 6 e⁻ rms (183 eV *FWHM*). In this calculation it was assumed that the electron-hole pair creation energy of diamond illuminated with X-ray radiation was 12.82 eV ± 0.13 eV [49] and its Fano factor was 0.08 [50]. The electronic noise contributions were extracted using multidimensional least squares fitting from measurements of the experimental photopeak *FWHM* at 5.9 keV (Mn K α) as a function of accumulated spectra shaping times. The difference between an experimentally measured and a multidimensional least squares fitted energy resolution was < 2 e⁻ rms (60 eV *FWHM*) for all 24 data points (four applied potential differences, six shaping times). The multidimensional least squares fittings derived the total capacitance at the input of the JFET; this capacitance was then used to calculate the 1/*f* series noise. The combined dielectric and incomplete charge collection noise component was derived by subtracting (in quadrature) series white noise (including induced gate current noise), parallel white noise, Fano noise, and 1/*f* series noise, from the measured energy resolution. The extracted noise components are presented in Fig. 5.

Fig. 5. The noise components of the spectrometer expressed as a function of shaping time at 5.9 keV with 50 V potential difference applied across the detector. The noise contributions are total noise (open diamonds), quadratic sum of dielectric and incomplete charge collection noise (+ symbols), parallel white noise (open squares), series white noise (× symbols), Fano noise (open circles), and 1/*f* series noise (stars). The lines are included to guide the eye only; fitted *FWHM* (dashed line); fitted parallel and series white noises (dotted lines).

The series white noise dominated the noise contributions at 0.5 μs shaping time; here, series white noise contributed 78 e⁻ rms ± 26 e⁻ rms (2.4 keV ± 0.8 keV *FWHM*) and 78 e⁻ rms ± 23 e⁻ rms (2.4 keV ± 0.7 keV *FWHM*) at 50 V and 100 V applied potential difference, respectively. At 10 μs shaping time, series white noise reduced to 17 e⁻ rms ± 6 e⁻ rms (0.5 keV ± 0.2 keV *FWHM*) and 17 e⁻ rms ± 5 e⁻ rms (0.5 keV ± 0.2 keV *FWHM*) at 50 V and 100 V, respectively. At 0.5 μs shaping time, parallel white noise contributed 12 e⁻ rms ± 6 e⁻ rms (0.4 keV ± 0.2 keV *FWHM*) and 13 e⁻ rms ± 4 e⁻ rms (0.4 keV ± 0.2 keV *FWHM*) of noise at 50 V and 100 V applied potential difference, respectively. At 10 μs shaping time, parallel white noise had risen to 55 e⁻ rms ± 26 e⁻ rms (1.7 keV ± 0.8 keV *FWHM*) and 57 e⁻ rms ± 20 e⁻ rms (1.7 keV ± 0.6 keV *FWHM*) at 50 V and 100 V, respectively.

The total leakage current at the input of the JFET was $27.5 \text{ pA} \pm 4 \text{ pA}$ (uncertainty propagated from the nonlinear least squares fitting) at 25 V applied potential difference and $28.5 \text{ pA} \pm 3 \text{ pA}$ at 100 V applied potential difference. Assuming that all leakage current contributions at the input of the JFET were independent of each other, the system leakage current was equal to $2(I_D + I_{DG})$, where I_D was the leakage current of the packaged detector and I_{DG} was the drain-to-gate leakage current in the Si JFET [46, 51]. The leakage current measured from the packaged detector at 20 °C and $\leq 100 \text{ V}$ applied potential difference was below the noise floor of the Keithley 6487 picoammeter (i.e. $< 0.4 \text{ pA}$) (section 3.1) which implied $I_{DG} \approx 14 \text{ pA}$ in the forward biased Si JFET when the spectrometer was operating.

The total capacitance acting at the input of the preamplifier was inferred from the multidimensional least squares fitting. The capacitance was constant at $5.6 \text{ pF} \pm 0.1 \text{ pF}$ (standard error) regardless of the potential difference applied across the detector. Here, the standard error, Q , is the standard error of the mean capacitance at the input of the JFET; Q is given by,

$$Q = \sqrt{\frac{\sum(x_i - \bar{x})^2 / (n-1)}{n}} \quad (6)$$

where x_i , $\bar{x}$, and n have been previously defined, but in this case, x_i is the capacitance at an applied potential difference and $\bar{x}$ is the mean capacitance of n (four) spectrometer operating voltages. Having measured the capacitance of the packaged detector directly ($870 \text{ fF} \pm 20 \text{ fF}$), it is clear that the series white noise was dominated by capacitances other than that of the detector. The dielectric noise of the detector, the Si JFET, and the detector's packaging can be estimated by considering the dielectric dissipation factors for these individual components and their associated capacitances. Assuming the dielectric dissipation factor of diamond was 6×10^{-4} [20] and the detector's capacitance was $350 \text{ fF} \pm 30 \text{ fF}$ (section 3.2) the detector itself contributed $9 \text{ e}^- \text{ rms}$ (267 eV FWHM) of dielectric noise. Some of this dielectric noise was likely also attributable to the silver epoxy adhesive [34] used to attach the detector die to the TO-39 package (section 3.2). The capacitance associated with the TO-39 package with the detector mounted on it, but with the wirebond removed, was $520 \text{ fF} \pm 20 \text{ fF}$ (section 3.2) with the majority of this capacitance attributed to the borosilicate glass ceramic insulated leg on the TO-39 package. Using the borosilicate glass dielectric dissipation factor stated in the manufacturer's catalogue (0.0127 [52]), the TO-39 package was calculated to contribute $50 \text{ e}^- \text{ rms}$ (1.5 keV FWHM) of dielectric noise. The preamplifier's Si input JFET (nominal capacitance of 2 pF [38, 43, 46], dissipation factor of 8×10^{-4}) contributed $24 \text{ e}^- \text{ rms}$ (737 eV FWHM) of dielectric noise.

Other sources of capacitance in a charge-sensitive preamplifier for photon counting spectroscopy are the feedback, test, and stray capacitances [46]; although the preamplifier used in this report did not have a test capacitor installed. Feedback and stray capacitances are not easily calculated separately because the individual dielectric dissipation factors and dielectric constants of all those sources of capacitance (most notably the stray capacitance) within the preamplifier are unknown. However, the multidimensional least squares fitting implied that the quadratic sum of dielectric and incomplete charge collection noise was $73 \text{ e}^- \text{ rms} \pm 1 \text{ e}^- \text{ rms}$ (rms error) ($2.21 \text{ keV FWHM} \pm 0.04 \text{ FWHM}$) at all of the applied potential differences. The calculated known dielectric noise contributions were subtracted in quadrature from the quadratic sum of total incomplete charge collection noise and dielectric noise. This yielded a remaining noise of $46 \text{ e}^- \text{ rms} \pm 2 \text{ e}^- \text{ rms}$ (rms error) ($1.40 \text{ keV FWHM} \pm 0.07 \text{ FWHM}$) of dielectric noise and incomplete charge collection noise (if any) in the spectrometer.

Incomplete charge collection noise would be a consequence of the detector's charge collection efficiency (CCE) being less than unity. For detectors where $\text{CCE} < 1$, incomplete charge collection noise was expected to reduce when the potential difference applied across the detector was increased. For the detector presently reported, the quadratic sum of dielectric and incomplete charge collection

noise ($73 \text{ e}^- \text{ rms} \pm 1 \text{ e}^- \text{ rms}$ (rms error)) did not change as the detector's applied potential difference was increased; this implied that incomplete charge collection noise was not a significant contributor to the noise measured. This is congruent with the observation that the count rate did not change when the potential difference applied across the detector was increased.

The total capacitance at the input of the JFET was $5.6 \text{ pF} \pm 0.1 \text{ pF}$ (standard error). After subtracting the capacitance attributed to the packaged detector and the Si JFET, the remaining stray and feedback capacitance at the input of the preamplifier was $2.7 \text{ pF} \pm 0.1 \text{ pF}$ (standard error). As previously discussed, the noise attributed to this combination of capacitances is not readily directly calculable given the lack of detailed knowledge of the dissipation factors of the individual dielectric components of which it is comprised, although previous (unreported) tests on earlier models of the preamplifier found feedback capacitance to be $\sim 10 \text{ fF}$. Nevertheless, the apparent noise contribution of these dielectrics ($46 \text{ e}^- \text{ rms} \pm 2 \text{ e}^- \text{ rms}$ (rms error)) and the unattributed, combined stray and feedback capacitance ($2.7 \text{ pF} \pm 0.1 \text{ pF}$ (standard error)) can be used to calculate an equivalent dielectric dissipation factor associated with the remaining capacitance. The effective dissipation factor for the entirety of the remaining capacitance was $2.1 \times 10^{-3} \pm 0.2 \times 10^{-3}$. This effective dissipation factor was compared with the dielectric dissipation factors of materials contained in the preamplifier to qualitatively assess the veracity of the effective dissipation factor.

The preamplifier's printed circuit board (PCB) was FR4, which had a nominal dissipation factor of 1×10^{-2} [53]; Si components had a dissipation factor of 2×10^{-4} [54]; and other dielectric components – considered to be low loss – had dissipation factors $\sim 10^{-5}$ [54]. The calculated effective dissipation factor in the previous paragraph ($2.1 \times 10^{-3} \pm 0.2 \times 10^{-3}$) is of reasonable order given the nominal dissipation factors of the preamplifier's materials and components. Given this, the earlier conclusion that incomplete charge collection noise was a minimal component of dielectric and incomplete charge collection noise was considered reasonable in this context. However, the simplicity and limitations of this analysis are acknowledged.

It is informative to compare the performance of the presently reported diamond X-ray spectrometer with similar studies undertaken using SiC X-ray detectors; SiC is another wide bandgap and relatively low atomic number material used in solid state detectors which are high temperature tolerant and radiation-hard. The best reported spectroscopic X-ray energy resolution obtained with a SiC detector stands at 196 eV FWHM at 5.9 keV [37]; that SiC detector was wirebonded to a custom built, state of the art, Complementary Metal Oxide Semiconductor preamplifier with an electronic noise contribution of $6.1 \text{ e}^- \text{ rms}$ (113 eV). However, 10 years earlier, the first reported spectroscopic response of a SiC detector was much more modest (2.7 keV FWHM obtained on a pulser line when illuminated with an ^{241}Am radioisotope) [55]. The new result presented here (energy resolution of 2.48 keV FWHM at 5.9 keV , with a diamond detector) emphasises the importance of a charge-sensitive preamplifier with ultra-low noise at the start of the electronics chain, given that the electron hole pair creation energy in diamond is large ($12.82 \text{ eV} \pm 0.13 \text{ eV}$ [49]) compared with competing detector materials, e.g. SiC (7.28 eV) [56]. Given the calculated dielectric noise contribution of the TO-39 package, the importance of low noise packaging is also significant.

The noise associated with the preamplifier's dielectric components used herein could be reduced by changing the FR4 PCB to glass, ceramic, or polytetrafluoroethylene. Another simple return in terms of dielectric noise reduction would be gained by mounting the detector and FET close together on a common package of low capacitance made from a low loss material. The use of different bonding epoxies to attach the detector to the TO-39 package could also reduce dielectric noise. Likewise, bump bonding the detector to the preamplifier (cf. die attach using silver epoxy and electrical connections made via wirebond) would also be beneficial for dielectric noise reduction.

5. Conclusions and further work

A 2 mm by 2 mm by 0.5 mm electronic grade single crystal CVD diamond was configured as a metal-semiconductor-metal detector and coupled with a bespoke preamplifier of low noise for X-ray photon counting. The detector was illuminated by an ^{55}Fe radioisotope X-ray source and X-ray spectra were accumulated. At 50 V and 75 V applied potential difference and 3 μs shaping time, an energy resolution ($FWHM$) of 2.48 keV at 5.9 keV was achieved. This was the first report of a diamond detector's spectroscopic photon counting response to soft (< 10 keV) X-rays.

Electrical characterisation showed that the leakage current of the packaged detector was negligible (< 0.4 pA) at applied potential difference of up to 100 V in each polarity (electric field strength of 2 kV cm^{-1}), and that the packaged detector's capacitance ($870\text{ fF} \pm 20\text{ fF}$) was invariant across the same range of applied potential difference. Shaping time noise analysis was conducted; the results suggested that when compared with other noise sources present in the spectrometer, any incomplete charge collection noise in the detector was negligible. The noise analysis also demonstrated that the majority of the noise (and thus degradation of the energy resolution beyond the Fano limit) was due to the detector's packaging, its connection to the preamplifier, and the preamplifier itself, rather than the bare die detector.

The results reported here are agenda setting in that they show for the first time that CVD diamond is a viable detector material for photon counting spectroscopic X-ray detection at photon energies < 10 keV. Although the presently reported energy resolution is modest, this was shown to be more a consequence of the detector's packaging and readout electronics rather than the detector itself. Future investigations are planned to characterise the detector's performance when it is operated at elevated temperatures ($>> 20^\circ\text{C}$) and when illuminated with X-rays and γ -rays of higher energy.

Acknowledgements and Authors' data statement

This work was in part supported by the Science and Technology Facilities Council, United Kingdom, through Grant No. ST/P002595/1 (A.M.B. PI). A.M.B. acknowledges funding from the Leverhulme Trust, United Kingdom, in the form of a 2016 Philip Leverhulme Prize. C.S.B. acknowledges funding received in the form of a Ph.D. studentship from the Science and Technologies Facilities Council Grant No. ST/P002595/1 (A.M.B. PI). The authors thank N.R. Gemmell for computer programming assistance with MATLAB. The authors thank M.D.C. Whitaker for computer programming assistance with LabVIEW scripts used to automate some measurements and for technical computing assistance with data processing. The data underlying this work are subject to commercial confidentiality. The authors regret that they cannot grant public requests for further access to any data produced during the study, however the key findings are included within the article.

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