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Performance of four CVD diamond radiation sensors at high temperature

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ABSTRACT

Ionising radiation detectors based on wide band-gap materials have the potential to operate at temperatures higher than 200 °C. Such detectors are important in applications such as monitoring near nuclear reactors and in deep oil and gas well borehole logging. We discuss the development of alpha particle detectors, based on CVD diamond, which operate with high charge collection efficiency and energy resolution at temperatures up to 225 °C. Four nominally identical commercial, electronic grade, CVD diamonds have been coated with a thin metal conductive layer in our laboratory and then attached to ceramic PCB. We present the I–V characteristics, the charge collection efficiency and the energy resolution for alpha particles from a mixed ^{239}Pu, ^{241}Am, ^{244}Cm source, for the four sensors operating at temperatures from 20 to 250 °C. Monte Carlo simulations of the energy spectra and charge collection efficiency and experimental measurements of these are presented. Energy resolutions between 1.6% and 4.0% at elevated temperatures with charge collection efficiency exceeding 96% were measured. The potential for thermal neutron detection is discussed.

1. Introduction

Radiation monitoring near nuclear reactors and in deep oil and gas borehole logging requires sensors capable of operating at temperatures > 200 °C. We are developing alpha particle detectors, based on CVD diamond, which operate with high charge collection efficiency (CCE) and energy resolution at these temperatures [1,2].

In recent years there has been significant research directed towards developing sensors, based primarily on diamond, which can act as neutron detectors at elevated temperatures. Some studies have demonstrated CVD diamond sensors operating continuously for periods exceeding 100 h with a variety of different metallisations and with bias potentials up to 300 V [3,4]. The latter developed a complete packaged sensor, specifically for bore-hole logging and demonstrated operation for more than 100 h at 200 °C and with a spectroscopic resolution of 3.3%. Recently a sensor has been shown to operate reliably at 300 °C with an energy resolution of 3.5% which was essentially independent of temperature [5]. Other wide band-gap materials have also been investigated, notably SiC which has been demonstrated to operate, in a self-biassed mode, up to 100 °C [6].

A notable feature of most papers is that either only one sensor is described and characterised or many are but each with a different metallisation or metal thickness or some other sensor-to-sensor variation. One of the aims of the work presented here was to make and characterise a number of nominally identical sensors based on CVD diamond from a single producer and subsequently cleaned and metallised by us identically.

2. Modelling the CCE and energy resolution

To determine the ultimate limits on detector performance in terms of resolution and CCE, a simple model of our experimental setup was developed. An approximation of the alpha source, diamond sensor and the PCB substrate was constructed in FLUKA [7]. Our triple isotope source, ^{239}Pu, ^{241}Am, ^{244}Cm with activity of 1 kBq per isotope, was modelled as an isotropic cylinder of 0.22 μm thickness and 3.5 mm radius. For the purposes of simulating energy spectra, the volume between the metalised contacts on either side of the diamond was treated as having 100% CCE, the rest of the volume being deemed to have 0% CCE. This assumption disregards edge effects but they were considered to be a minor contributor to the overall detector resolution when compared to the main sensor geometry effects. The resulting spectral peaks generated by the FLUKA model were fitted with Gaussian functions. The extracted CCE and energy resolution (FWHM) are shown in Table 1.

3. Experimental details

A set of four sensors of $2 \times 2 \times 0.5\ mm^3$, were fabricated using single crystal electronic grade CVD diamond (Element Six Ltd). We deposited metal coatings on the square surfaces of the diamond at a pressure of 10^{-3} mbar. The sensor capacitance was measured to be 0.34 pF at 1 MHz. The response of the sensors to alpha particles was evaluated at a pressure of < 10^{-4} mbar to minimise alpha particle energy loss.

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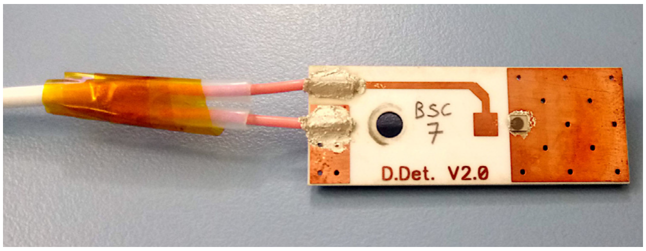

Fig. 1. PCB with co-axial cable and wire-bonded diamond sensor. The hole through the PCB is for mounting it to the copper heating block.

Table 1
CCE and energy resolution from simulation.

Alpha energy MeV	CCE %	Energy resolution (FWHM) %
5.16	99.0 ± 0.5	1.22 ± 0.005
5.49	99.7 ± 0.5	1.07 ± 0.006
5.81	99.2 ± 0.5	1.04 ± 0.005

Table 2
Sensor characteristics.

Sensor	Type	Surface roughness nm	Raman peak cm^{-1}	Raman peak width cm^{-1}
BSC5	Grid etched	5.38	1332.55	2.21
BSC6	Normal	5.78	1332.88	2.07
BSC7	Normal	5.24	1332.40	2.22
BSC8	Normal	8.06	1332.23	2.07

Table 3
I–V summary data. SS = Symmetrical Double Schottky and SA = Asymmetrical Double Schottky.

Sensor	Δ 0 V to 100 V pA	Δ 0 V to −100 V pA	Behaviour
BSC5	1.43E+05	−2.47E+05	SS
BSC6	22.5	−17.1	Ohmic
BSC7	42.4	−55.7	Ohmic
BSC8	749.1	−648.8	SA

The sensors were mounted on copper-coated, 1 mm thick alumina ceramic PCB (Rubalit 708S from CeramTec) with vias to connect the ground planes on both surfaces. Silver-filled epoxy suitable for temperatures up to 260 °C (Duralco 120, from Cotronics Corp.) was used both to fix the sensor to the board, and to connect a coaxial cable to the board. The electrical connection from the top of the sensor to the PCB was completed by a set of ultrasonically bonded 25 μm diameter aluminium bond wires from the edge of the top surface metallisation to a pad on the PCB, connected to the coaxial cable (see Fig. 1). The charge induced in the sensor was amplified by a Canberra 2004 pre-amplifier operating at 1 V/pC and a 2021 spectroscopy amplifier with a shaping time of 1 μs.

The surface roughness of the diamond was determined using a Zygo NewView 5000 and the Raman peak and its width were measured (see Table 2). The narrow peaks indicate a low concentration of substitutional nitrogen defects [8].

4. Results

The I–V characteristics tests of each sensor were measured using a Keithley 617 electrometer. Subtracted from the data, and attributed to a leakage current in our measurement system, was a current offset of approximately -130 pA at 0 V bias. The results are shown in Table 3.

Using our triple isotope source in vacuum, the energy resolution of the primary peaks was measured for both positive and negative bias voltages at temperatures up to 250 °C. Fig. 2 shows a typical dataset

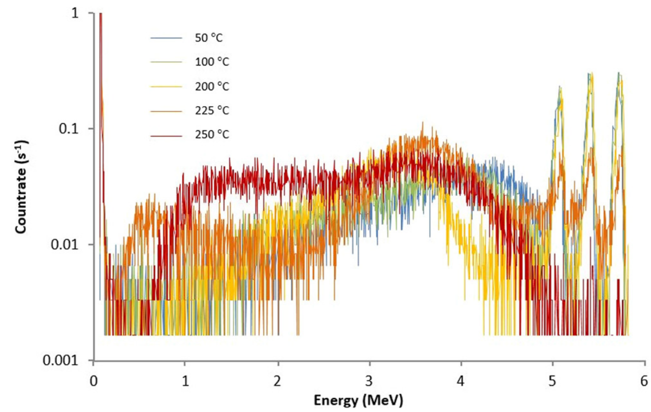

Fig. 2. Variation of response to triple alpha source as a function of temperature for sensor BSC6.

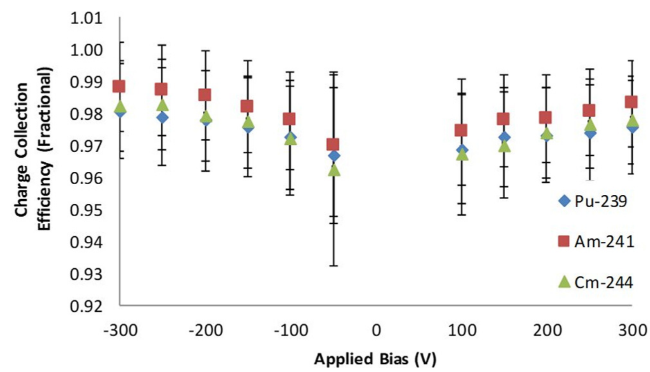

Fig. 3. CCE as a function of positive and negative bias for sensor BSC8.

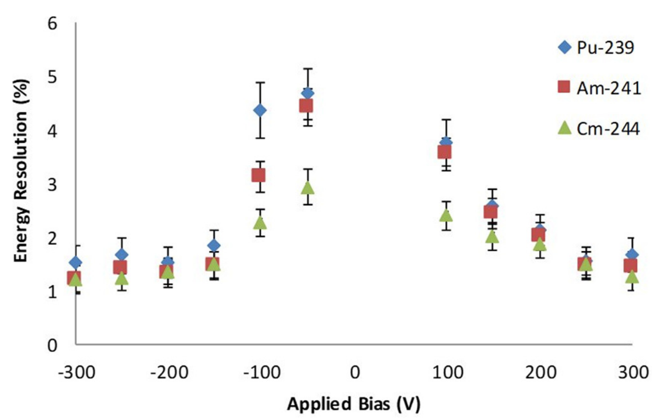

Fig. 4. Alpha particle energy resolution as a function of positive and negative bias for sensor BSC8.

for the sensors with moderate leakage currents. The large, suppressed peak close to 0 MeV is the electronic noise in the absence of the source.

We determined the variation of CCE and energy resolution with bias. In Figs. 3 and 4 we show the response of one sensor as a function of bias voltage and alpha particle energy respectively. The error bars shown are the one sigma boundaries.

Table 4 summarises the highest temperature at which each sensor operated spectroscopically. The CCE and energy resolution at the peak temperature are those quoted in the table. Sensor BSC5, which was plasma etched with a surface grid pattern, had such a high leakage current even at 100 V bias (see Table 3) that no alpha peaks could be resolved even at room temperature. The failure of sensor BSC5 due

Table 4
CCE and alpha particle energy resolution. In this table the highest temperature at which no alpha particle energy-dependent effects on CCE or energy resolution was observed is stated for each sensor.

Detector	Bias V	Mode	Peak temperature °C	CCE %	Energy resolution (FWHM) %
BSC6	300	hole	225	99 ± 5	1.6 ± 0.5
BSC6	−300	electron	225	97 ± 3	2.1 ± 0.5
BSC7	200	hole	200	98 ± 2	4.0 ± 0.7
BSC7	−200	electron	225	96 ± 2	2.0 ± 0.5
BSC8	300	hole	225	98 ± 3	3.3 ± 0.7
BSC8	−300	electron	225	97 ± 3	2.6 ± 0.5

to high leakage is attributed to misalignment in the shadow masking, which permits direct conduction down the comparatively low resistance sides of the diamond crystal. Due to limitations in the mounting of BSC5 for metalisation it is possible for these areas to retain a degree of hydrogen termination [9] which may contribute to this effect.

In electron sensitive mode each of the three unetched sensors was found to function well, with good performance up to a temperature of 225 °C but losing useful spectroscopic performance past this, see for example Fig. 2. The energy resolution, in hole mode, for sensor BSC7 shows a dependence on the alpha particle energy at a temperature of 225 °C varying from 2.1% for alphas with 5.16 MeV energy to 7.0% for alphas with 5.81 MeV energy. These limitations on performance, with observable impact around 180 °C, correspond with findings in the wider literature on thermally stimulated current and thermoluminescence [10,11]. Trapping–detrapping effects are thought to be the main limiting factor on the operation of diamond sensors at elevated temperature and these have been investigated elsewhere by means of transient current [12,13], thermally stimulated current [14] and thermoluminescence techniques. Identified in these works are deep traps with activation energies that indicate their contribution becomes significant in the temperature range studied here. A series of deep trapping levels with activation energies of 0.97, 1.14, 1.29 and 1.4 eV have been identified [15]. This corresponds to a temperature range of 127 – 327 °C with a particularly strong trapping activation energy at an equivalent temperature of 227 °C. This corresponding well with the observed degradation in performance observed here. Due to the short range of alpha particles, contributions to the trapping caused by the metal-diamond interface will have an exacerbated effect on the space charge compared to those present in the bulk.

5. Enhancing neutron detection efficiency

Micropatterning of the CVD diamond surface by plasma etching is expected to enhance the thermal neutron detection efficiency. We have tested the patterning technique experimentally using a ≈ 35 μm pitch grid (see Fig. 5).

Using FLUKA [7] we simulated the response to neutrons for a diamond with pits replicating those shown in Fig. 5 filled with ^{10}B. In the simulation we varied the width of the diamond ridge that surrounds each of the etched pits and with a width of approximately 4 μm we predict a neutron detection efficiency exceeding 25% as shown in Fig. 6.

6. Summary

Radiation sensors intended for high temperature operation have been constructed and tested under alpha irradiation at temperatures up to 250 °C. Of those not immediately rendered non-functional during the fabrication process (BSC5) all were found to deliver workable detection performance at up to 225 °C with a consistent degradation when heated past this, attributed to a sharp increase in thermally induced trapping–detrapping past this point. The sensors were stable throughout the applied heat cycles.

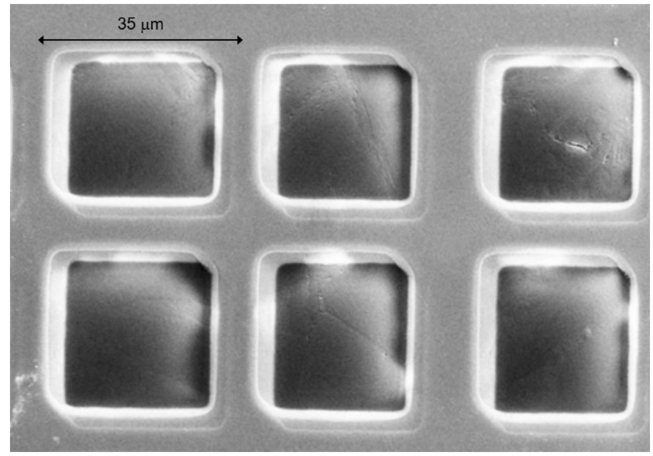

Fig. 5. SEM photograph of part of a grid, plasma etched into the surface of sensor BSC5.

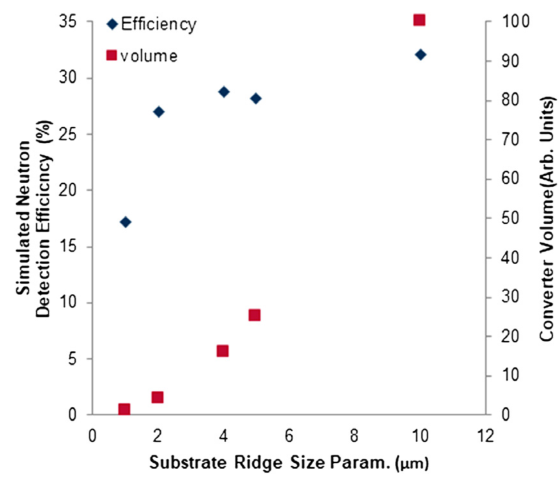

Fig. 6. MC simulation predicting the enhancement of neutron detection.

A surface profile on a diamond sensor which we generated using plasma etching, has the potential to improve the neutron detection efficiency above that of a planar sensor.

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