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Prototyping and performance study of a single crystal diamond detector for operation at high temperatures

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ABSTRACT

Prototype single crystal diamond detectors with different types of metallization and post metallization treatment were fabricated for the applications requiring fast neutron measurements in the Indian Test Blanket Module (TBM) at the International Thermonuclear Experimental Reactor (ITER) Experiment. The detectors were characterized by leakage current measurements to ascertain that the leakage currents are low and breakdown voltages are higher than the voltage required for full charge collection. The detector response to charged particles was evaluated using a $^{238+239}$ Pu dual energy alpha source. The detectors showed an energy resolution of about 2% at 5.5 MeV. In order to study their suitability for the operation at higher temperatures, leakage current variation and alpha response were studied up to 300 °C. At 300 °C, peaks corresponding to 5.156 MeV and 5.499 MeV alphas could be separated and there was no significant degradation of energy resolution. Finally, the detector response to fast neutrons was evaluated using a Deuterium-Tritium (D-T) neutron generator. The observed spectrum showed peaks corresponding to various channels of n-C interactions with a clear isolated peak corresponding to ~8.5 MeV alphas. The detectors also showed high sensitivity of 3.4×10^{-2} cps/n/(cm^2 s)–4.5×10^{-2} cps/n/(cm^2 s) and excellent linearity of response in terms of count rate at different neutron flux in the observed range of 3.2×10^{5} n/(cm^2 s) to 2.0×10^{6} n/(cm^2 s).

1. Introduction

Diamond as a material for radiation detection has several outstanding properties such as high bandgap, high carrier mobilities, high breakdown voltage and high resistance to a corrosive environment. Recent advancements in chemical vapour deposition (CVD) technology for the growth of artificial diamond substrates have resulted in commercial availability of single and polycrystalline diamond substrates to study their suitability for various detector applications. Several studies investigating their performance in applications such as fast timing measurements in particle and nuclear physics experiments [1], beam monitoring [2,3], dosimetry [4–6] and space applications [7,8] have been reported. Diamond detectors have specially attracted interest for on line fast neutron monitoring in future fusion facilities such as ITER due to their superior properties over conventional neutron detectors such as gas counters, scintillators, silicon detectors, activation foils, etc. These detectors can directly measure fast neutrons through n-C interactions without any converter materials. Various experimental reports have shown that polycrystalline [6,9,10] as well as single crystal diamond (SCD) detectors [11–17] are promising for fast neutron monitoring in fusion facilities. There are grain boundaries in the bulk of polycrystalline diamond which act as trapping centres for the generated charge carriers. The pulse height spectrum of neutrons obtained with a polycrystalline diamond detector shows a continuum without peaks due to charge trapping at these defects. As a result, these detectors have poor energy resolution and hence are not suitable for neutron spectrometry applications. However, these detectors have been observed to exhibit very good linearity of response in terms of count rate. Hence, these detectors can be used in counting mode for the measurement of neutron rate. SCD detectors can give neutron energy information. The neutron pulse height spectrum obtained using these detectors show various peaks corresponding to different channels of n-C interactions. The 14 MeV fast neutron interaction with a diamond detector gives a well isolated peak in the spectrum corresponding to ~8.5 MeV alphas released through ^{12}C(n, α_0)^{9}Be reaction channel.

The diamond detector development presented in this paper is being carried out for monitoring neutrons at different locations in the Indian TBM Experiment at the upcoming ITER facility [18,19]. The neutron flux and temperatures inside the TBM at various locations are expected to be 10^{10} n/(cm^2 s) to 10^{13} n/(cm^2 s) and 300–500 °C respectively. The neutron flux and temperatures outside the TBM at various

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locations such as shield block, pipe forest, etc., are expected to be 10^{10} n/(cm^2 s) to 10^4 n/(cm^2 s) and ~50–100 °C respectively. We have earlier reported performance results of polycrystalline diamond detector prototypes developed for this application [20]. The count rate observed with 14 MeV neutrons showed very good linearity with neutron rate. However, the observed pulse height spectrum with neutrons showed a continuum without any peaks. Hence neutron energy information could not be obtained with these detectors. Since it is also necessary to measure the energy of fast neutrons generated by Deuterium-Tritium (D-T) fusion, development of detectors based on single crystal diamond substrates has been taken up. The fast neutron response of the fabricated detectors was studied using a D-T fast neutron source. In order to estimate the maximum temperature suitable for stable operation at higher temperatures, the leakage current variation and alpha particle response were studied up to 300 °C which was the limit imposed by the experimental setup and the detector package. An overview of detector fabrication and performance results of the first batch of prototype detectors are presented in this paper.

2. Experimental

SCD detectors were fabricated using commercially available (IIa Technologies Pte Ltd, Singapore) chemically vapour deposited single crystal diamond substrate of size 5 mm×5 mm 400 µm. Prior to metallization, the substrates were given standard pretreatment such as chemical cleaning in hot sulphochromic acid (saturated CrO_3 in H_2SO_4) for creating an oxygen terminated surface to reduce the surface leakage currents. Additionally, the substrates were subjected to oxygen plasma treatment after a chemical cleaning step. Both sides of substrates were metallized using an e-beam evaporation process for a sequential deposition of Cr and Au (20 nm/200 nm), or Al metal (250 nm) at a vacuum of ~10^{-7} mbar. The metallization was carried out through a metal mask to prevent metal deposition up to the edges of substrates. The metallization covered area of 4 mm×4 mm on both sides resulting in a detector with a square geometry of area of 16 mm^2. Since the metallization was carried out using a shadow mask, no guard rings were provided. One of the Cr/Au metallized substrates was annealed in an argon atmosphere at 300 °C to study the performance at temperatures higher than the room temperature. The leakage currents were measured using a wafer prober prior to packaging to verify that the metallized substrates have acceptable leakage current/breakdown voltage characteristics. These measurements were performed by mounting the metallized substrates on the chuck of the prober and by making a pressure contact to the front surface of the detector using a probe head. The electrical connections to the detector were provided through the probe head and through the contact to the chuck. Subsequently, the metallized substrates were mounted on a transistor outline (TO) header (material Kovar (Fe/Ni/Co alloy), diameter of about 15 mm) using a conductive epoxy. Since the operation at higher temperatures was to be investigated, a conductive epoxy suitable for operation up to 300 °C was selected for mounting the substrates. A TO metal can (about 7 mm height and 0.3 mm thickness) was attached to the top of the header using an adhesive epoxy. The can was provided with a circular opening in the centre so that the alpha particles were not blocked during alpha response measurements. The front contact was provided by gold wire bonding. The leakage current and alpha response measurements were carried out using a custom made setup with a vacuum chamber for mounting the detector (Fig. 1). An arrangement for placing the alpha source in the vacuum chamber at an adjustable distance from the detector was also provided for the alpha response measurements. The electrical connections from the detector were provided through feed throughs to external BNC connections for making leakage current or alpha response measurements. The chamber housed a copper chuck incorporating an electrical heater for increasing the temperature up to 350 °C. The detector was mounted on this chuck for the measurements at higher temperatures. The temperature of the chuck was controlled using a temperature controller.

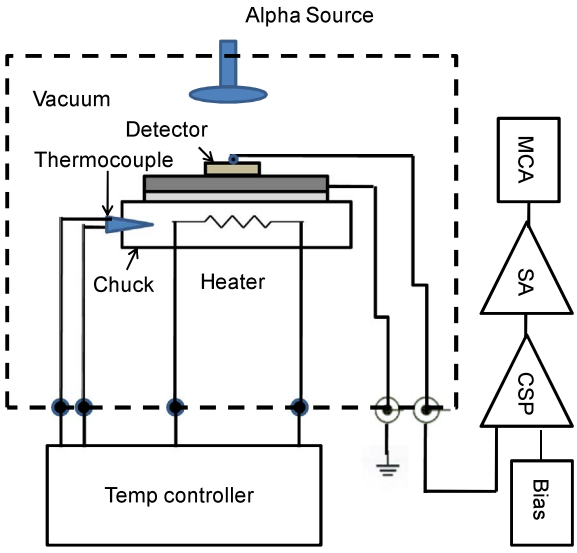

Fig. 1. Schematic diagram of the experimental setup used for I-V and alpha response measurements at higher temperatures in vacuum. The detector is mounted on an electrically heated chuck. The chuck temperature is controlled by a temperature controller. A thermocouple measures the chuck temperature. An electronics chain comprising a charge sensitive preamplifier (CSP), a spectroscopy amplifier (SA), a detector bias supply and a multichannel analyzer (MCA) is used for alpha response measurements. For I-V measurements, the detector is connected to a picoammeter with a voltage source.

Subsequent to fabrication of the detectors, leakage current vs voltage (I-V) characteristics were measured using a programmable picoammeter with a voltage source at different bias voltages up to ± 400 V. For the measurements at higher temperatures, the detector was placed over a heated chuck and the temperature was varied from room temperature up to 300 °C in steps of 100 °C using a temperature controller (Fig. 1). The temperature controller also displayed the temperature of the chuck as measured by a K-type thermocouple. As the thermocouple was located inside the chuck and not close to the surface, the temperature attained at the top of the chuck where the detector was placed during measurements was lower by a few degrees than the temperature which was measured by the temperature controller. The temperature at the top of chuck was calibrated for different temperature settings of the temperature controller to correct for this deviation.

Subsequent to I-V measurement, performance of the SCD detector to charged particles was evaluated using a dual energy alpha source of $^{238+239}$Pu. The alpha spectrum was obtained at room temperature as well at 300 °C. During these measurements, the alpha source was mounted at a distance of about 5 cm from the detector. It was verified that at this location the temperature was below 50 °C. This precaution was taken to prevent the degradation of the alpha source that might take place due to heating. The entire heating system inside the chamber was maintained at a vacuum of 10^{-2} mbar using a rotary pump. The alpha spectrum was obtained using standard electronics comprising a detector bias supply, a charge sensitive preamplifier (CSP), a shaping amplifier (SA) and a 4k channel multichannel analyzer (MCA). The output signal of the charge sensitive preamplifier (gain −44 mV/MeV (silicon), noise < 2 keV (silicon)) was shaped and amplified using a shaping amplifier with a shaping time of 2 µs and a gain of 50. Output signal of the amplifier was monitored on a cathode ray oscilloscope (CRO) and was fed into a MCA to obtain the alpha spectrum. As discussed in Section 3, the detector bias voltage for alpha measurement was optimized based on the voltage required for full charge collection.