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Investigations of high quality diamond detectors for neutron fluency monitoring in a nuclear reactor

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Abstract

We report here on the characterisation of neutron detectors fabricated using a range of synthetic diamonds including polycrystalline as well as single crystal samples. After material characterisations including response to alpha particles (stability, detection efficiency etc), measurements were made using thermalised neutrons in a nuclear reactor at fluencies of 10^6–10^{10} neutrons/cm^2 s. The readout electronic was based on current preamplification: it enables to locate the electronic preamplifier remotely from the hostile environment, typically at distances that can exceed 20 up to 200 m. It requires high signal to noise ratios for the detection device. To enable the neutron conversion (diamond itself remains transparent to such low energy neutrons) a thin film converting material made of ^{235}Uranium element is used to emit charged particles that are detected by the diamond film. The device responses were studied in terms of sensitivity, response time, reliability, and conformity with respect to the fluency as measured using calibration fission chambers. The results exhibit remarkable linearity and stability. The fabricated devices display are capable of faithfully following the power cycles of a nuclear reactor. With respect to gas ionisation chambers, these devices of much smaller dimension can find a use for neutron fluency profiling with high position resolution.
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Keywords: CVD diamond; Detectors; Neutron; Single Crystal

1. Introduction

The fabrication of radiation detectors from chemical vapour deposited (CVD) diamond has now been demonstrated for several applications addressing photon and particle detectors in the frame of specific industrial applications [1–3]. Regarding the nuclear industry needs, diamond based detectors have been developed for the detection of neutrons [4–9] and this paper presents the principle and the characteristics of these detectors for the particular case of fission neutrons. The ability of diamond to withstand the high irradiation fluxes as encountered in nuclear reactors is one of the motivations. Further, since detection in nuclear reactors also implies extreme fluencies of gamma ray photons, the ability to use a detecting media that is naturally exhibiting a low sensitivity to photons is a plus. However, neutrons are light and uncharged particles, therefore exhibiting low interaction probability with nuclei and no interaction with electrons. Thus, neutrons are more difficult to probe using the conventional detection techniques. In a thermal reactor, neutron energies vary from 25 meV (thermal neutrons — *n*th) to a few MeV (fast neutrons). The present study focuses on the detection of thermalised neutrons, i.e. not providing sufficient kinetic energy for their detection via e.g. elastic scattering.

Of the few techniques available [10], the approach here relies on the use of a converting material, i.e. elements on which a nuclear reaction gives rise to the creation of charge particles that could be detected. Boron, and particularly ^{10}B, has been used successfully with a-Si:H [11], or with GaAs [12]. With the same order of neutron cross section and alpha particle energy, Lithium 6 could also be a candidate, but presents severe drawbacks for safety consideration in the lithium form, and in the LiF form the material does not exhibit sufficient radiation hardness. Other techniques can also be used and here we will focus on the use of the Uranium 235 isotope that gives rise to

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fission fragments (FF) from the following reaction, themselves interacting in diamond.

$$n\text{th} + {}^{235}\text{U} \rightarrow \text{FF}_1 + \text{FF}_2 + \upsilon n + E, \text{with } \upsilon = 2 \text{ or } 3$$

Typically, fission fragments are elements which atomic mass varies from $Z=70$ to 160, with typical equivalent charges of 25 e^-, energies of several tens of MeVs, and with a double peak statistical distribution centred around two mean atomic masses of 90 and 140 (See insert in Fig. 8) [13]. Uranium can be seen as a difficult material to handle. It has however to be compared with the large quantities of uranium regularly handled in a nuclear reactor. A few extra micrograms do not constitute an extreme difficulty. The range of fission fragments in diamond is of the order of 5 μm, and varies as a function of the nature of the fragment, e.g. from 7.3 μm for ^{97}Sr to 5 μm for ^{138}Xe. These values are much lower than that of alpha particles with typically 14 μm at 5.5 MeV.

2. Device fabrication

The current study is conducted on a range of synthetic diamonds including polycrystalline as well as on two single crystal CVD diamond samples obtained with the courtesy of *Element Six, Ltd, UK*. For the case of the polycrystalline devices, three samples were processed throughout the study, whereas only measurements of one of the three devices are presented here: no significant difference being probed on the others that would bring further insights to the discussion. The polycrystalline devices, referenced here as *PolyX*, were fabricated using a microwave PECVD system (5 kW ASTeX Inc., Lowell MA, ASTeX 5400), using a gas mixture of CH_4/H_2 of 1%, on silicon. After the film growth, all samples (including single crystal samples) were oxidized in boiling H_2SO_4 with KNO_3. The samples were cleaned in pure deionised water in an ultrasonic bath first for 10 min at 20 °C and secondly for 5 min at 90 °C. Gold layers were evaporated on both sides of the substrates for contact preparation, taking care of respecting a minimum of 500 μm gap spacing between the contact and the sample edge. The samples were assembled in an encapsulated cell enabling the Uranium converting foil to be mounted facing the device, via a 2 mm air gap, through which a PTFE pinhole could be used in order to prevent fission fragment interactions on the contacts borders, i.e. where non-uniform electric fields are likely to occur that may prevent even charge collection. Fig. 1 shows a schematic presentation of the device structure, together with a view of the final device before encapsulation (insert). The device is sealed in an ultrapure aluminium A7 cell (impurity level <0.3%, Mg content <2.83%), and with a 10 mm thick cap layer of the same composition. The radioactive decay of aluminium after neutron activation is short (few minutes). The box is sealed and the Uranium 235 layer is fixed between the Aluminium cap layer and the sample holder. This way, perfect confinement of the fission products is ensured, thus enabling measurements at high fluencies (10^{10} n cm^{-2} s^{-1}).

3. Experimental conditions

Measurements in core of a nuclear reactor imply very hostile environments for the device, as well as to the surrounding environments. As such, the connection used is ensured by a high immunity cable. Further, since the device is likely to be exposed to fluencies in the order of 10^8 to 10^{10} n cm^{-1} s^{-1} and at elevated temperatures, the use of a charge preamplifier is prohibited since it would require to be located as close as possible to the detector. The signal here requires to be transported for several tens of meters before being processed. This motivates the use of a current collection pre-amplifier: as opposed to a charge collection pre-amplifier, they rely on the transportation of the signal pulse in a 50 Ω cable with perfect impedance matching to reduce losses. The best compromised amongst the various configurations tempted came using a BAI (for *Bloc Amplificateur d'Impulsion*) type preamplifier with a gain of 1000. Such type of preamplifier as fabricated by Canberra Eurysis is commonly used in industrial environments in French nuclear reactors as well as experimental ones, and particularly with fission chamber detection devices. Its stability and reliability therefore have been well proven. One of the drawback however of this type of electronics is the extreme sensitivity to electronic noise. Since the preamplifier treats all signals, and not only those that have the shape of one interaction, as would a charge collection preamplifier, the result is a significant loss in signal to noise ratios. The technique therefore is only compatible with device responses that exhibit high signal to noise ratios. As such the use of U235 converting material is preferred since the fission fragments are well over more energetic (typ. 100 MeV) than the 1.5 MeV alpha particles that would be available in the case of a ^{10}B converter. It is to be

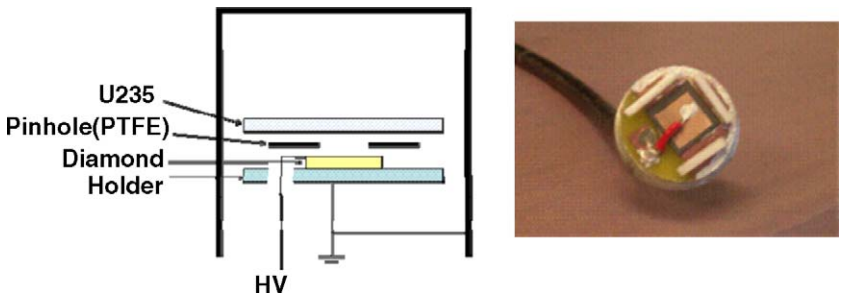

Fig. 1. Schematic representation of the device holder (left) and PolyX diamond device mounted in its holder before seal (right).

Table 1
Characteristics of both ^{235}U converting media chips used for the neutron detection experiments

^{235}U Chip	Mass (μg)	Area (cm^2)	Cross sectional mass ($\mu g/cm^2$)	Enrichment with ^{235}U	Number of fissions in the chip for 1 W reactor power ($\approx 10^6$ n cm^{-2} s^{-1})
#1 (PolyX)	30	1.33	22.6	90.08	35/s
#2 (SC)	53	0.5	105.0	97.00	151/s

noted that such a preamplification system hardly enables the accurate measurement of the energy of the interacting species in the device: since the signal travels over several tens of meters, its amplitude is subjected to broadening and flattening, and thus the information of the energy of the interacting particle weakened. Further, all the connection requires particular care, and all signal transport to be ensured by high immunity cables.

The experiments were performed in the ULYSSE facility in CEA in Saclay. This reactor consists of a reduced size experimental reactor based on highly enriched Uranium. The core is made of Aluminium and moderation is ensured by water circulating within the uranium elements. All the experiments here were performed within the *thermal column* where the neutrons are thermalised through a 1.4 m long graphite block. Calibration using gas chambers gives a neutron fluency in this column that corresponds to 10^6 n cm^2 s^{-1} for a 1 W reactor power. This fluency varies linearly with the reactor power and will enable calibration of the experiments. From the mass and exposed area of the uranium converting foil, it hence becomes feasible to calculate the amount of fissions that occur within the U layer per second. Two ^{235}U chips were used for the current experiments. The chip referenced as #1 was used for the experiments on PolyX samples, whether #2 was used for monocrystalline samples (this choice was purely made for experimental convenience in order to be able to reduce deadtimes between experiments while the chips cool down). Their mass and characteristics are given in Table 1. From the calculation of the probability of interaction of the neutrons within the chips, it is possible to evaluate the rate of fission reactions in the ^{235}U converting layers in order to enable further calibration of the measurements and give a figure for the detector efficiency.

4. Detection performances of the polycrystalline devices

For the case of the polycrystalline diamond sample, priming is required and X-ray irradiation using a minimum dose of 10 Gy from a 50 kV X-ray tube was used. After this pre-irradiation the signal response under X-rays remains stable with time. Fig. 2 presents the charge detection spectra as obtained under the interaction of 5.5 MeV alpha particles emitted by a ^{241}Am source. The mean distribution of the collected charge is spread over a broad range of energies: this is due to the non-uniform detection performance caused by the polycrystalline nature of the sample [14]. Since alpha particles interact randomly in the device, the mean energy distribution is spread over a broad range of energies. Rough calibration of the spectrum in Fig. 2

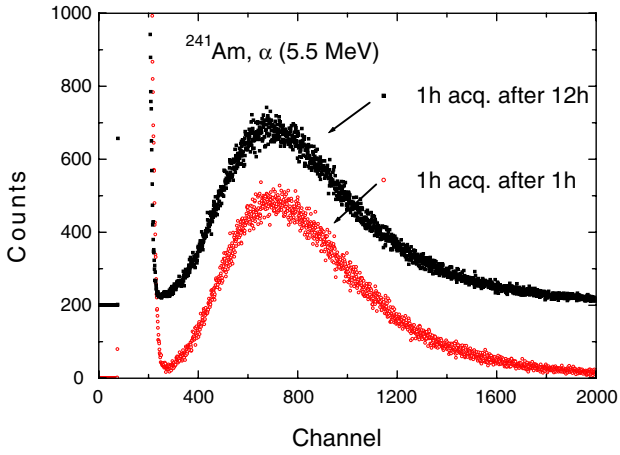

Fig. 2. Pulse height spectra obtained with the PolyX samples under ^{241}Am alpha particles. Two spectra acquired during 1 h are plotted: the top one being recorded 12 h after continuous alpha irradiation (top spectra is shifted vertically by 200 counts for clarity).

leads to a mean energy around 1200 KeV, thus corresponding to an average collection efficiency of 21%. This value remains rather low. However, one very important feature is the stability of the spectrum with time, as also probed in Fig. 2, where the same detection spectrum is acquired for the same duration (1 h) but after 12 h of irradiation (and shifted vertically by 200 counts for clarity). The device shows perfect stability with time. When this sample is mounted in the device holder of Fig. 1, and exposed to thermal neutrons, the interaction of fission fragments in the device leads to the signal displayed in Fig. 3: each interaction induces a typical current pulse which values lies between 3 and 5 V (amplification is 1000 from the BAI current preamplifier). This relative high signal to noise ratio renders the devices well compatible with measurements in the reactor.

When mounted and probed under neutrons in the Ulysse reactor, the device displays perfect stability with time as shown in Fig. 4. Also probed in insert is the linearity of the

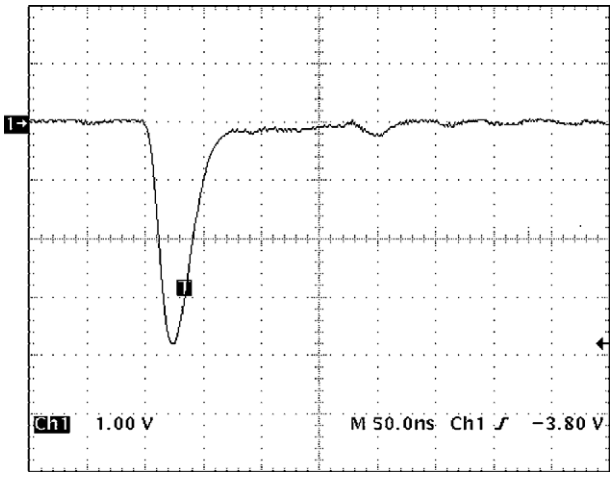

Fig. 3. Temporal evolution of the device output when one fission fragment interacts in the polyX sample. The signal is probed after the current preamplifier: the detection surface is 1.21 cm^2, the Uranium 235 layer used here is of 22.6 μg cm^{-2}, bias is +60 V, and measurement is probed after a length of 17 m of cable.

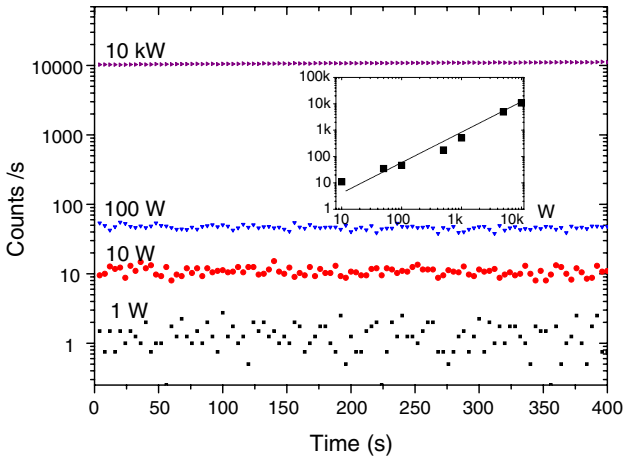

Fig. 4. Stability of the PolyX response under neutron irradiation and appreciation of its linearity (insert).

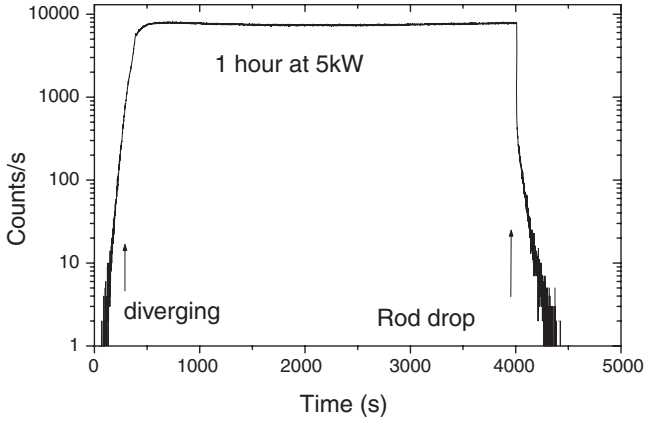

Fig. 6. Probing the neutron fluency during a 1 h reactor cycle.

device response with the reactor power, thus the neutron fluency. It shows however that the device linearity is more difficult to appreciate. In fact, the current collection spectra as obtained during the measurement are shown on Fig. 5. The noise is primarily appreciated from one initial measurement before neutrons are irradiating the device (<1 W reactor power). However, the values given in Fig. 4 rely on the appreciation of the discrimination threshold and thus of the noise distribution from the pedestal in Fig. 5, from which a significant error may occur: clearly, a better precision would be obtained if the detection signals would be more intense and come further out of the electronic noise. Nevertheless, the device can be used to monitor the profile of the reactor activity, as probed in Fig. 6, displaying the neutron fluency rise during divergence of the reactor, the plateau during which the power is maintained constant shows little variations (<10%) caused by the reactor control that was essentially done manually for the tests. Correlation with reference detectors confirmed that the observed behaviour has been measured by the diamond device. Finally, the rod drop leads to fast decay of the neutron fluency as displayed by the detector. For this experiment the total integrated neutron fluency that had reached the U235 layer was of $1.8 \cdot 10^{13}$ n, and thus calculation of the U235 chip giving a ratio of $3 \cdot 10^{-5}$, it comes that the polyX diamond did not show any performance alteration after over $5 \cdot 10^{8}$ fission fragments interaction.

From the device geometry it is possible to calculate the amount of fission fragments that hit the diamond. Comparing this value with the absolute number of fissions that occur in the chip, it comes typically that one should expect at a reactor power of 10 kW to get $35 \cdot 10^{4}$ fissions per second in the chip, and from this leads to approximately 84 kcounts/s. The values probed on Fig. 4 rather give figures of the order of 10 kcounts/s measured. Clearly, even though the device seems to follow faithfully the reactor fluctuations, has fast response times, and also does not show alteration nor saturation, it appears that over 85% of the interactions are lost since they are too weak and hardly come out of the electronics noise. This value is the combination of the relatively low charge collection efficiency of the device (measured at 21% under alpha particles) and the

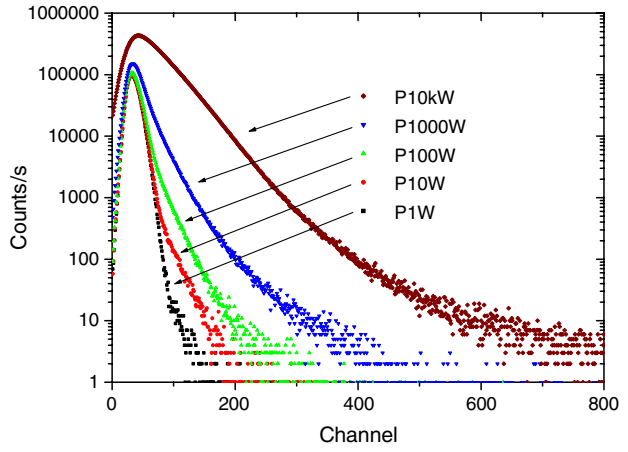

Fig. 5. Pulse height spectra as measured when the diamond is used in the reactor and irradiated with thermal neutrons at the given reactor powers.

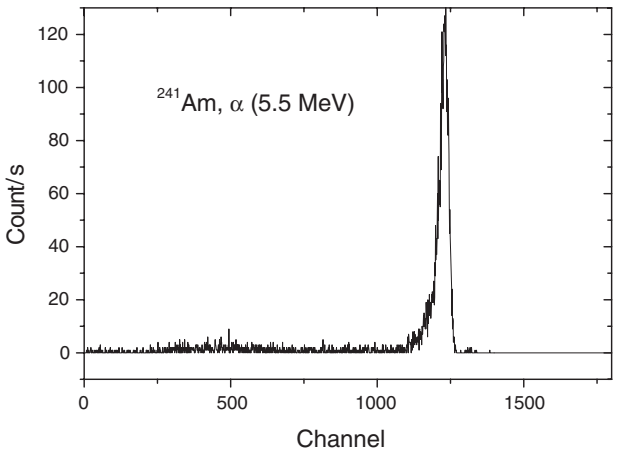

Fig. 7. Pulse height spectrum as measured on a single crystal diamond sample (provided by e6, Ltd, UK), under ^{241}Am alpha particles, as obtained using a charge preamplifier (gain is differing from preceding experiments as here the pic corresponds to the 5.5 MeV alpha energy).

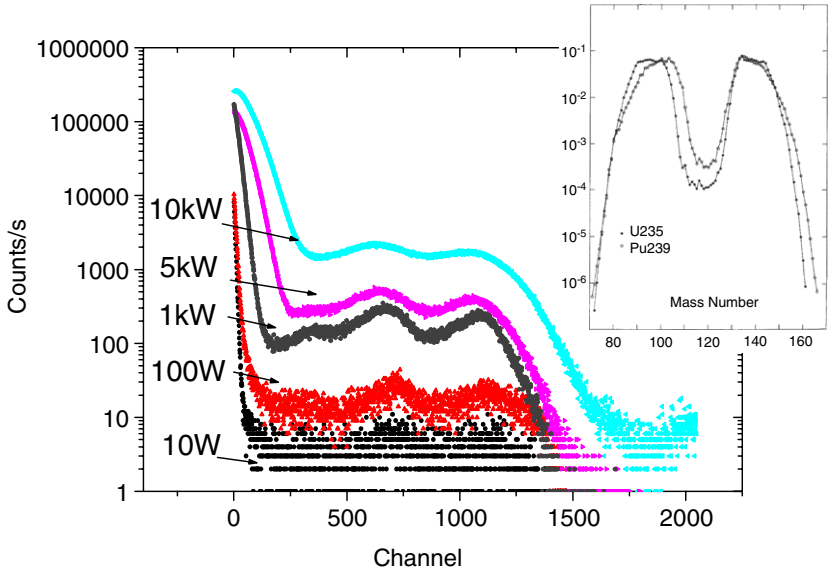

Fig. 8. Pulse height spectra as measured on one single crystal diamond sample and as a function of the reactor power. The two-lobe structure of the spectra can be associated with the two-lobe structure of the fission fragment structure as generated in the ^{235}U converting media.

difficulty to accurately determine a threshold in the neutron pulse height spectra.

5. Detection performances of the monocrystalline devices

Two single crystal synthetic diamond layers grown using the CVD technique by *Element Six, Ltd UK* were used as a reference in the same conditions as the preceding experiments. Both devices were mounted as described earlier and the alpha detection spectra here exhibit good characteristics as displayed in Fig. 7. Here however since the samples were much smaller, care was taken in order to avoid particle interaction in the contact edge via the use of a pinhole well aligned with the electrical contact. No priming nor pumping is required for this material, and the influence of the bias is weak: from 0.2 to 1 V/μm: the pulse heights hardly shift further towards the higher energies above the value obtained at 0.2 V/μm, chosen for the experiments. The calibration of the detection chain gives 100% collection efficiency and the mean resolution of the device is of the order of 4%, a satisfying value since the acquisition electronics setup is here not optimised for spectroscopy experiments.

When this set-up is used and assembled for thermal neutron detection, we can see that there was an extreme interest in having a better signal to noise discrimination and a good energy resolution, as here the detection spectra (Fig. 8) are displaying the two bump spectral structure as expected from the energy distribution of the fission fragments as displayed in the insert of Fig. 8. This unique feature is here visible only because the devices are collecting 100% of the deposited energy and that there is no broadening of the collected charge distribution as

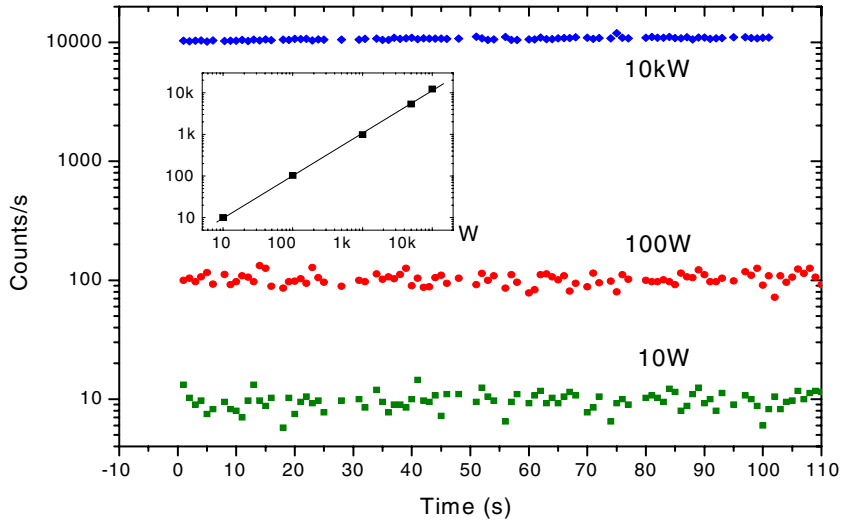

Fig. 9. Stability of the single crystal response under neutron irradiation and appreciation of its linearity (insert).

observed in PolyX diamond. The signal was also perfectly stable with time. The devices when used in the nuclear reactor gave perfect stability as displayed in Fig. 9, as well as a better linearity with the neutron fluency (insert).

Here again from the known device geometry it comes possible to calculate the amount of fission fragments that hit the diamond. Comparing this value with the absolute number of fissions that occur in the chip, it comes typically that one should expect at a reactor power of 10 kW to get $151 \cdot 10^4$ fissions per second in the chip, and this theoretically leads from the interaction probability in the ^{235}U layer and the solid angle between the chip and the diamond to approximately 7.8 kcounts/s. The values probed on Fig. 9 here give figures close to 10 kcounts/s measured. Of course the preceding value is affected by a relatively high error bar that could easily reach 25% from the geometrical losses evaluations. Here, and as opposed to the values obtained on the polyX samples, this shows that the device faithfully respects the absolute value of the count rates of the fission neutrons incident on the detector. We can say that from the improved diamond signal to noise sensitivity such single crystal diamond thermal neutron detectors can be used for absolute evaluation of the neutron fluency in-core of the reactor.

From the complete set of experiments performed here, we have also measured the total integrated neutron fluencies at which the devices had been exposed. From the two single crystal devices, namely SC1 and SC2, respectively, received in total $2.38 \cdot 10^{13}$ and $1.45 \cdot 10^{13}$ n cm^{-2}. It is assumed that the radiation degradation from the neutron interaction is negligible at such low fluencies with respect to that from the fission fragments interacting in the diamond surface. In fact, only after typically 5 10^{15} thermal neutrons/cm^2 integrated fluencies, hardly a low detection properties alteration has been observed [15–21]. From the ^{235}U chip interaction probability and the geometrical losses within the device, one can evaluate that it leads to typically $1.8 \cdot 10^7$ and $1.1 \cdot 10^7$ fission fragments that interacted in SC1 and SC2, respectively. Even though both values are extremely similar, we have observed a strong reduction in the device sensitivity after irradiation on SC1. For instance, the alpha detection efficiency had drop by a factor of 8 from the 100% value observed before irradiation. Indeed, one difference was that SC1 had been used solely without PTFE the pin-hole during tests, therefore that a much greater amount of fission fragments have also interacted in the contact edges. Surely fission fragments that are extremely ionising from their elevated mass as well as charge should be strongly damaging the surface layer. STRIM calculation evaluate the range of fission fragments to 5 μm in diamond, and absolute vacancy numbers for one single fission fragment reach almost 10^4. This value being very theoretical, it is possible that part of the vacancies created are not stable and restore especially at room temperature. However, it seems a critical limit for such a type of absolute neutron detectors, and unfortunately the availability of such devices prevents from a reliable statistical study. It is expected that further progress in this field will be made in the near future from the progressive vulgarisation of single crystal synthetic samples.

6. Conclusions

We have demonstrated that synthetic diamonds can be used for neutron fluency monitoring in a nuclear reactor. In order to be compatible with EMC and the hostile radiative environments, the devices must provide significantly high signal to noise ratios to be compatible with current collection preamplifier that could be placed at a distance (>10 m) of the detector. Therefore, signal to noise limits have here imposed ^{235}Uranium as converting media. This can be seen as a difficulty but in the context of a nuclear reactor use, precautions on how to handle Uranium compounds are well in hands. Somewhere it is even easier and less at risk than other converting materials like lithium. The fission fragments interacting in the diamond provide the ability to accurately measure the neutron fluency in the nuclear reactor. The tests performed on extreme quality single crystal diamonds (provided by Element Six, Ltd, UK) showed that almost all fission fragment interactions are probed, therefore enabling the fabrication of a absolute detector. On the other hand, in polycrystalline diamond, charge loss occurs that prevents the absolute measurement of the fission fragment interaction. However, the polyX devices remain stable, show relatively good linearity behaviours, and enable the fast monitoring of power fluctuations in the reactor. Since calibration would nevertheless be necessary for all devices, it seems that polycrystalline materials to some extent are interesting for such hostile environments application from their current availability with respect to single crystal diamonds.

Consideration on the damage caused by fission fragments in diamond have been discussed, but clearly the present data do not suffice for statistical considerations (2 single crystal samples were tested). Progress on this issue is expected shortly with the progressive increased availability of single crystal diamond materials.

We believe that this work demonstrates the possible use of diamond for neutron detection in nuclear reactors from fission fragment sensing. The advantages of diamond here are the much smaller size with respect to fission chambers which may find applications for example for neutron profile localisation much more accurately. Also, the portability can be of high interests in confined environment like those encountered in e.g. propulsion nuclear reactors.

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