

Basic Thermoluminescence Characteristics of Fabricated Germanium Doped Cylindrical Optical Fibres under Low Dose Particles Irradiations

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ABSTRACT

Germanium (Ge) doped optical fibres have been studied extensively as a substitute of commercially used TLD for passive dosimetry due to its affordable and robust nature. For space application, a passive dosimeter is required to be able to detect accumulated radiation from 0.5 mGy to 500 mGy. In this work, two types 6 mol% Ge doped cylindrical fibres with diameters of 483 μm (483CF) and 604 μm (604CF) were irradiated with low dose beta, low dose neutron and proton to study the fibres thermoluminescent (TL) response in terms of linearity. For all three types of radiation, compared to 483CF, 604CF shows higher TL responses due to the larger core volume of 604CF producing more TL response. The fibres show high linearity at for all types of radiations with correlation of determination (r^2) of higher than 0.97. From the minimum detectable dose (MDD) calculated, beta, neutron and proton irradiated fibres accordingly can detect at least until 0.029 mGy, 0.55 mGy and 33.81 mGy. From the MDD and linearity of the fibres at lower doses, it can be concluded that the fibres can be used for low dose particles radiation detection for space dosimetry.

Keywords: Low Dose Neutron, Low Dose Beta, Proton Irradiation, Optical Fibres, Thermoluminescence Dosimeter.

1. INTRODUCTION

Low earth orbit (LEO) at 300~500 km altitude from Earth provides many challenges when it comes to space radiation monitoring due to its complex mixed radiation field with the main sources of radiation being galactic cosmic rays, solar particles events, and, electrons and protons trapped in the Van Allen Belts [1]. Thermoluminescent dosimeters (TLD) such as TLD-600 and TLD-700 used by European Space Agency (ESA) and TLD:MSO by Japanese Aerospace Exploration Agency (JAXA) are usually used to detect low linear energy transfer (LET) radiations (LET <10 keV/ μm) [2, 3]. TLDs placed inside Pirs-1 module attached to the Russian Service Module of the International Space Station (ISS) measured 70.9 mGy absorbed dose from May to October 2007 and 127.7 mGy absorbed dose from May to October 2008 which shows an increase in absorbed dose with an equal amount of exposure time due to the elevation of ISS altitude and corresponding increase of the trapped proton flux during movement of the ISS through South Atlantic Anomaly (SAA) [4]. While the absorbed dose pose a considerable threat

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to astronauts on board of ISS, from the point of radiation detection material the absorbed doses were quite low which may veer from the material's linear detection range ergo, developing a new passive dosimeter for space radiation monitoring that requires consideration regarding the low dose region of a particular radiation. Furthermore, the absorbed doses mentioned are accumulated absorbed doses received in space. As space radiations are complicated mixed radiation field, the actual dose for beta, neutron and proton will be lower than afore mentioned doses.

2. METHODOLOGY

2.1 Materials

Table 1 Fibre properties

| Label | Ge dopant percentage [mol %] | Diameter [μm] | Core diameter [μm] | Core volume $\times 10^{-2}$ [mm^3] |
|-------|------------------------------|----------------------------|---------------------------------|--|
| 483CF | 6 | 483 | 78.1 | 2.88 |
| 604CF | 6 | 604 | 94.7 | 4.21 |

The fibre preform was fabricated with a technique called modified chemical vapour deposition (MVCD) and followed by fibre pulling process. Details on fabrication procedure can be found in other literatures as this work used fibres readily made in those literatures [5, 6]. There are two types of cylindrical optical fibres (SiO_2) used in this work. Both of them were doped with 6 mol % Ge and labelled as 483CF and 604CF corresponding to the fibres' diameter of 483 μm and 604 μm . The fibres were cut into 6.0 ± 1.0 mm length using a Thorlabs ruby cutter prior to annealing. The fibres were annealed using a Carbolite furnace with a ramping rate of $10^\circ\text{C}/\text{min}$ from 50°C to 420°C . The heating was not from room temperature due to the furnace's limitation. Thus the heating rate from room temperature to 50°C was uncontrollable. A dwell time of 1 h at 420°C was placed prior to slowly cooling of the fibres inside the furnace for 8 h down until 50°C to avoid any thermal stresses. The door of the furnace was opened to further cool down the fibres until room temperature. The purpose of annealing is to eliminate any pre-irradiation TL signal (such as tribological exposure) [7]. The handling of fibres was conducted using a Dymax 5 vacuum tweezer to lessen surface abrasion, reduce accumulation of dust and avoid deposition of oil from the skin of the handler onto the fibre [8]. Table 1 lists the properties of fibres used in this work briefly. The volume of the fibres' is used to normalise the TL response.

2.2 Irradiation Set-up for Low Dose Beta and Neutron Irradiation

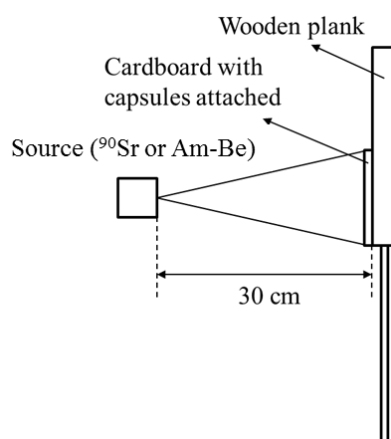


Figure 1. Irradiation setup for beta and neutron samples.

Delrin capsules filled with 20 pieces of annealed fibres were attached onto cardboard box using double sided tape. The cardboard was then attached onto a wooden plank using the same tape and the capsules were secured further with masking tape to avoid falling during exposure. The wooden plank was fixed vertically with the surface of the plank to source distance being 30 cm as shown in Figure 1. For both beta and neutron irradiations, the fibres were irradiated at Malaysian Nuclear Agency, Bangi Malaysia.

2.2.1 Beta Irradiation

The absorbed dose rate of Strontium-90 (^{90}Sr) beta source used in this work was $2.199 \mu\text{Gy/h}$ with an activity of 75 MBq. The fibres were irradiated up to 0.15, 0.41 and 0.56 mGy which require irradiation time of 68 h, 187 h and 255 h accordingly.

2.2.2 Neutron Irradiation

For neutron irradiation, the irradiation source was Americium-Beryllium (Am-Be) with an absorbed dose rate of $13.91 \mu\text{Gy/h}$. The neutron energy of the source is 4.5 MeV. The samples were irradiated up to 0.09, 0.97, 2.59 and 3.57 mGy with and irradiation time of 6.25 h, 68 h, 187 h and 255 h respective to the absorbed dose.

2.3 Proton Irradiation

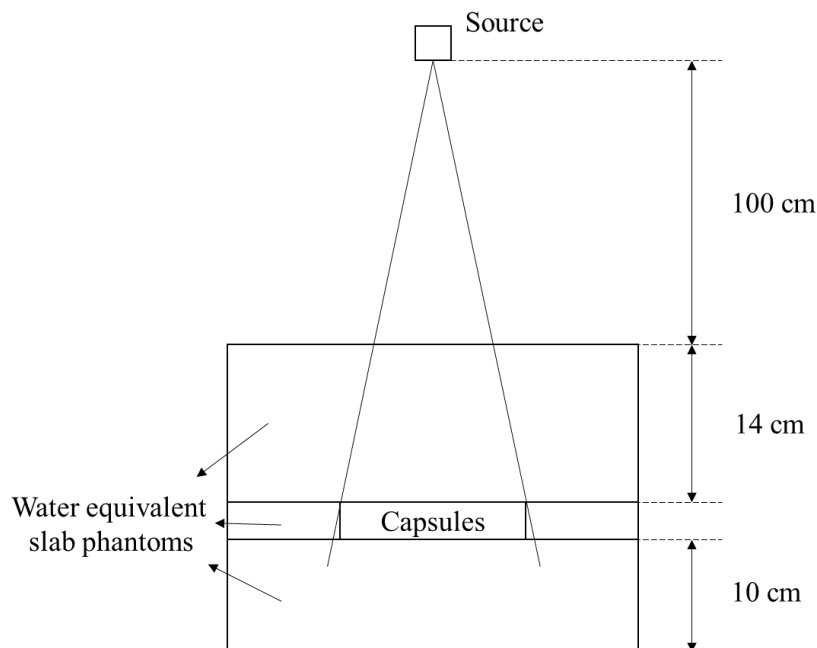


Figure 2. Irradiation setup for proton samples.

Ten pieces of annealed fibres were placed inside delrin capsules prior to irradiation using proton beam with the energy of 150 MeV at 0.5 up to 8.0 Gy. The capsules were placed horizontally in between water equivalent slab phantoms with a built up thickness of 14 cm (equivalent to the maximum dose) on top of the capsules and a built down thickness of 10 cm underneath the capsules to attenuate backscatter radiations as shown in Figure 2 [8]. The proton source was located perpendicular the capsules locations. The fibres were irradiated at Hyogo Ion Beam Medical Center, Japan with the source to surface (SSD) distance was 100cm.

2.4 Fibre Readout

Table 2 TTP of cylindrical optical fibre

| Preheat | | Acquire | | | Anneal | |
|------------------|----------|-------------------------|--------------------------|----------|------------------|----------|
| Temperature [°C] | Time [s] | Temperature rate [°C/s] | Maximum temperature [°C] | Time [s] | Temperature [°C] | Time [s] |
| 80 | 10 | 30 | 400 | 13 | 400 | 10 |

The fibres' TL response was read using a Harshaw TLD™ Model 3500 from Thermo Fisher Scientific located at Centre of Diagnostic Nuclear Imaging, Universiti Putra Malaysia. 0.5 bar nitrogen gas atmosphere was kept to stifle the occurrence of spurious light signals resulting from triboluminescence and diminish surface oxidation of the planchet used to place the fibres [8]. There is a possibility the fibres might produce extremely low TL response, hence, two pieces of fibres were read at the same time in a single reading for beta and neutron irradiated fibres. For proton irradiated fibres, a single piece of fibre will be used per reading. The time-temperature profile (TTP) used to read the cylindrical fibres is shown in Table 2 [8]. After being heated for 10 s at 80°C, the temperature will increase at a rate of 30°C/s until 400°C then the temperature will dwell at 400°C until 13 s. Further dwelling at 400°C for 10 s is to remove any residual signals in the fibres [7].

2.4.1 Linearity

The fibres' TL response were measured one day post irradiation for beta and neutron irradiated fibres and three days post irradiation for proton irradiated fibres to evaluate the fibres' linearity. Five readings were taken for each dose using two pieces of fibres per each reading for beta and neutron irradiated fibres. For proton irradiated fibres, a total of seven readings using a single piece of fibre per reading were conducted 3 days post irradiation. The difference in reading time post irradiation between proton and the irradiations is due to proton irradiation being conducted in Japan while the TLD reader used to read the fibres was in Malaysia. The TL response prior to normalisation provides an unfair comparison since the dimension and weight of the dosimeters was different. Thus, multiple previous studies normalised the TL response with the dosimeter's weight [5, 7, 9, 10]. The problem with weight based normalisation is the usage of actual fibre's weight and not the weight of the fibre's core. Here, it is of importance to use the core weight because the core is the one that genuinely produce the measured TL response due to defects caused by Ge doping. Thus, in this work core volume based normalisation is utilized. The core volume of the fibres can be calculated by measuring the core diameter from the images of fibre cross section produced by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX). The average of the readings is normalised with the core volume and fitted into a straight line to acquire the coefficient of determination (r^2).

2.4.2 Linearity Index

The linearity index $f(D)$ of irradiated fibres were evaluated using Equation 1 [11].

$$f(D) = \frac{\frac{S(D) - S_0}{D}}{\frac{S(D_1) - S_0}{D_1}} \quad (1)$$

Here, S_0 is the intercept of the TL intensity axis (y axis), $S(D)$ is the TL response corresponding to dose D , D_1 dose in the linear region, $S(D_1)$ TL response corresponding to dose D_1 . Linearity index is of importance to analyse the linearity of fibres at each dose instance. While higher r^2 value means higher linearity, linearity index imparts a better understanding of linearity at each dose tested. When $f(D) = 1$ the fibre is linear at a particular dose while $f(D) > 1$ and $f(D) < 1$ accordingly indicate supralinearity and sublinearity.

2.4.3 Minimum Detectable Dose (MDD)

The MDD of 483CF and 604CF were calculated using Equation 2 [9].

$$MDD = \frac{(BG_{mean} + 2\sigma)}{m} \quad (2)$$

Here, BG_{mean} is the average of five TL readings of the background fibres, σ is the standard deviation of the background fibres and m is slope obtained from linearity analysis of the fibres. MDD is used to know the lowest dose that can be detected by a particular TLD material. However, MDD value is not an indication that the TLD material will have linear response to a particular MDD value.

3. RESULTS AND DISCUSSION

3.1 Beta Irradiation

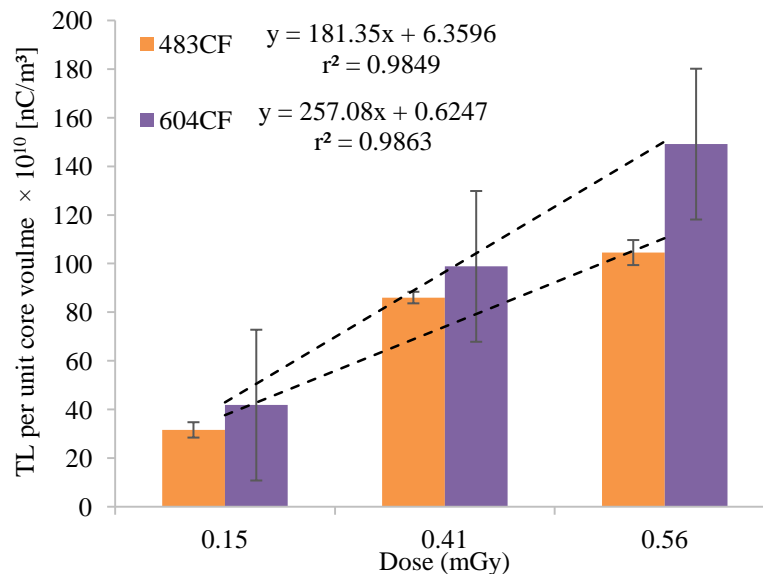


Figure 3. TL response per unit core volume vs dose of beta irradiated fibres.

Since each readings used two pieces of fibres, to obtain the TL responses normalised per unit core volume, the actual readings were divided by two multiplied by fibre's core volume. Figure 3 shows the expected single reading normalised per unit core volume. Both of 483CF and 604CF are linear at the tested doses with high r^2 value of 0.9849 and 0.9863, respectively (Figure 3). Meanwhile, 604CF exhibit larger TL responses with maximum value of 149×10^{10} nC/m³. It is due to 604CF's core possessing a wider cross sectional area than that of 483CF's core which means larger area with defects that produce TL thus the higher response. Here, defect is the area in the fibres that is doped with germanium which is the core of the fibres.

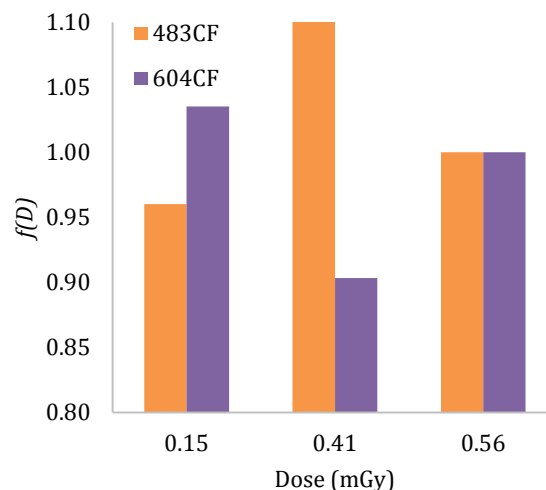


Figure 4. Linearity index of beta irradiated fibres.

Table 3 *MDD* of beta irradiated fibres

| Fibre type | Slope, $m \times 10^{10}$ [nC/mGy m^3] | Bg Average \times 10^{10} [nC/ m^3] | Bg Deviation \times 10^{10} [nC/ m^3] | MDD [mGy] |
|------------|--|---|---|--------------|
| 483CF | 181.35 | 2.52 | 2.68 | 0.029 |
| 604CF | 257.08 | 5.61 | 0.98 | 0.026 |

Figure 4 is plotted for further investigation in fibre's linearity at low beta dose. 483CF shows sublinearity at 0.15 mGy and supralinearity at 0.41 mGy with $f(D)$ value of 0.96~1.11. Contradictorily, 604CF was supralinear at 0.15 mGy and sublinear instead at 0.41 mGy with $f(D)$ value of 0.90~1.04. The *MDD* of both fibres are shown in Table 3. For low dose beta irradiation, both fibres show extremely low *MDD* with 604CF at being lower compared to 483CF. Both low *MDD* and linearity at the tested dose of both fibres signify that Ge doped optical fibres are susceptible to be used at low dose beta radiation dosimetry with further lower dose beta radiation detection due to *MDD* being even lower than the linearity range that is tested. Compared to photon irradiation where 604CF can detect down until 27 mGy it can be said that the fibre is extremely sensitive to beta irradiation as it can detect as low as 0.026 mGy [5].

3.2 Neutron Irradiation

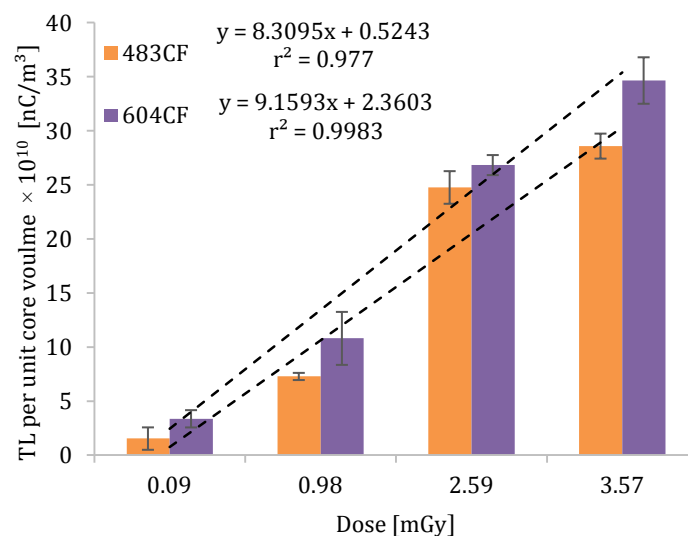


Figure 5. TL response per unit core volume vs dose of neutron irradiated fibres.

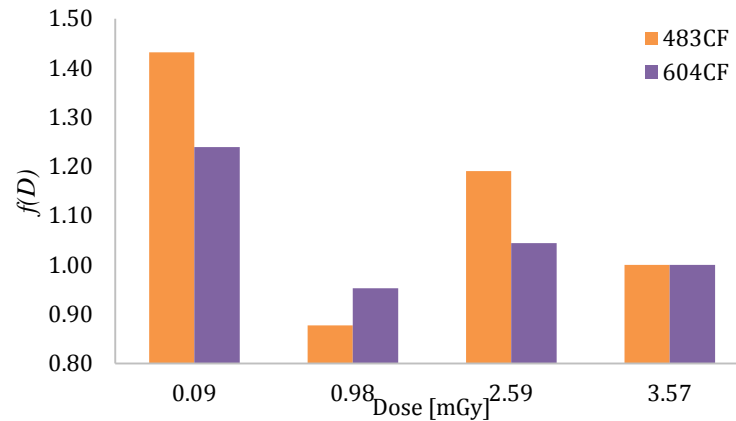


Figure 6. Linearity index of neutron irradiated fibres.

Table 4 *MDD* of neutron irradiated fibres

| Fibre type | Slope, $m \times 10^{10}$ [nC/mGy m^3] | Bg Average $\times 10^{10}$ [nC/ m^3] | Bg Deviation \times 10^{10} [nC/ m^3] | MDD [mGy] |
|------------|--|---|---|-----------|
| 483CF | 8.31 | 0.62 | 2.57 | 0.38 |
| 604CF | 9.16 | 2.61 | 1.95 | 0.55 |

For neutron irradiation, again compared to 483CF, 604CF shows higher TL response as shown in Figure 5. Both fibres show good r^2 value with 483CF's r^2 value being 0.977 and 604CF's r^2 being exceptionally high at 0.9983 inside the tested dose range of 0.09 mGy ~ 3.57 mGy. As with beta irradiation, 604CF higher TL response was due to larger defect size. The TL maximum TL response at 3.57 mGy was 34.66×10^{10} nC/ m^3 for 604CF and 28.59×10^{10} nC/ m^3 for 483CF. As in Figure 6, the $f(D)$ values for both fibres show large margin of supralinearity at the lowest dose of 0.09 mGy at 1.43 and 1.24 for 483CF and 604CF accordingly. From Table 4, the *MDD* of both fibres are 0.38 mGy and 0.55 mGy respective to 483CF and 604CF. 0.09 mGy is a dose that is lower than the *MDD* where the fibres are supposedly is not able to detect neutron anymore, thus the non-linearity. Additionally, since $f(D)$ used the highest dose of 3.57 mGy as a point of normalisation, the further the dose is from the point of normalisation, even slight changes will produce higher/lower $f(D)$ value that is obvious. Ignoring the $f(D)$ values at 0.09 mGy, 483CF $f(D)$ values have a range of 0.88~1.19.

3.3 Proton Irradiation

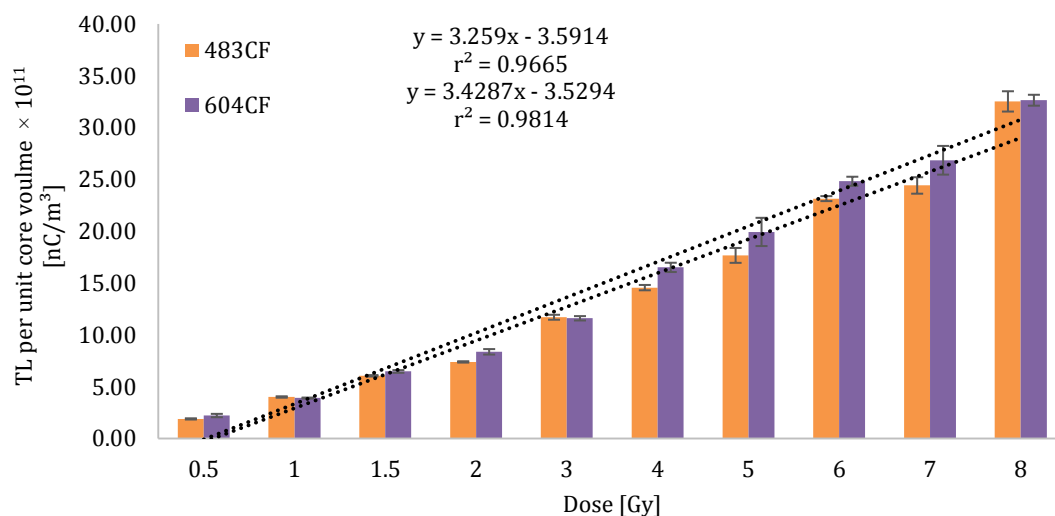


Figure 7. TL response per unit core volume vs dose of proton irradiated fibres.

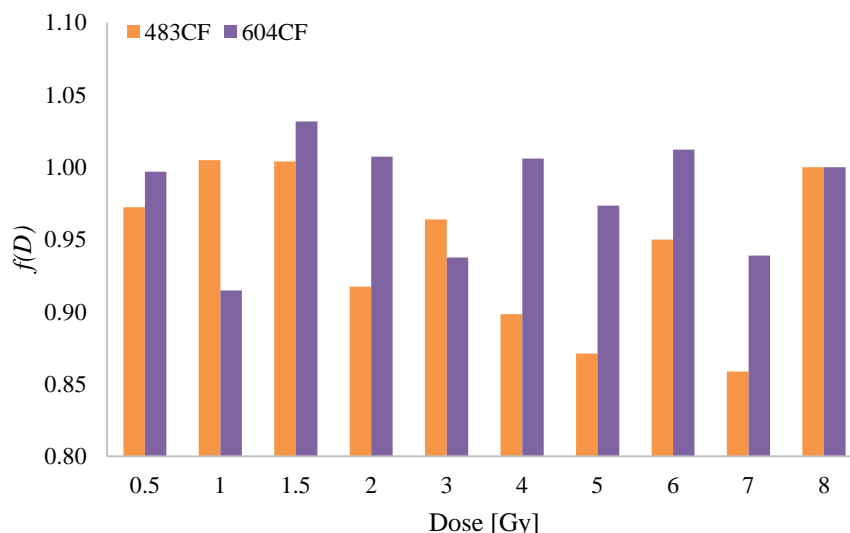


Figure 8. Linearity index of proton irradiated fibres.

From Figure 7, like beta and neutron irradiated fibres, both 483CF and 604CF show a high degree of linearity with r^2 values of 0.987 and 0.9969 respectively. 604CF with larger core volume compared to 483CF shows only slightly higher TL response compared to 483CF. While both types of fibres show high r^2 values, from linearity index of proton irradiated fibres are drawn in Figure 8, it can be seen the responses are not wholly linear throughout the irradiated doses. 604CF is sublinear and supralinear almost alternatively at doses under 8 Gy. However, 483CF are mostly sublinear at doses under 8 Gy. Only at 1.0 and 1.5 Gy had a linearity index with a value near to 1.

For beta and neutron samples, the *MDD* of 483CF is higher than 604CF which imply that 604CF fibres are more sensitive to the type of mentioned radiation. For proton irradiated fibres however, the opposite is true. Furthermore, both 483CF and 604CF seem to have higher *MDD* when irradiated using proton dissimilar to when the fibres are irradiated using beta and neutron.

Table 5 *MDD* of proton irradiated fibres

| Fibre type | Slope, $m \times 10^{10}$ [nC/mGm ³] | Bg Average $\times 10^{10}$ [nC/m ³] | Bg Deviation $\times 10^{10}$ [nC/m ³] | MDD [mGy] |
|------------|---|---|---|--------------|
| 483CF | 109.19 | 1.53 | 1.08 | 33.81 |
| 604CF | 168.48 | 2.09 | 0.57 | 19.16 |

4. CONCLUSION

In this work, the aim is to measure the ability of optical fibres to detect low dose beta, low dose neutron and proton radiation. From the high r^2 value of more than 0.97, thus in can be say that germanium doped cylindrical optical fibres is able to detect those radiations with high linearity. For beta and proton irradiations, 604CF had a lower *MDD* compared with 483CF.

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