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Ge-doped Silica Fibre Proton Beam Measurements: Thermoluminescence Dose-Response and Glow Curve Characteristics

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ABSTRACT

Present investigations concern germanium (Ge) doped (2.3 and 6.0 mol%) silica preforms fabricated into cylindrical- and flat-shaped fibre radiation dosimeters. When subjected to 150-MeV proton beam irradiation, the fibres are observed to produce sensitive dose response. The fibres are fabricated via a modified chemical vapour dopant deposition technique and subsequent pulling process. Prior to irradiation, a thermal annealing process was carried out to erase any pre-irradiation signals potentially existing in the samples. For radiation dose in the range from 1 up to 10 Gy, these optical fibres exhibit an excellent linear relationship, offering coefficients of determination (R^2) better than 0.99, suggesting reliable calibration and utilisation. The general structure of thermoluminescence (TL) glow curve is presented as a broad peak, differing from that of the phosphor-based TLD-100 dosimeter. The maximum in the TL glow curve peak manifests at a temperature within the readout range 231- to 350 °C. The relatively high glow-peak temperatures allow the fabricated Ge-doped optical fibres to be measured between room- and high-temperature conditions. In terms of shape, the TL glow curve remains unchanged at different irradiation dose within the investigated range of interest (1 to 5 Gy). The TL properties studied herein provide an important advance in understanding the TL mechanism in the fabricated Ge-doped silica fibres, shown to be viable for use in proton beam dosimetry.

Keywords: Fabricated Ge-doped Optical Fibre, Thermoluminescence Glow Curve, Radiation Dose Linearity, Proton Beam Dosimetry.

1. INTRODUCTION

Over the past few years, numbers of materials have been investigated as candidate dosimetric, media, including glass-based media, subsequently proposed as new radiation dosimeters with properties and sensitivity that improve upon existing capabilities. In such context, a particular form of glass-based media that has attracted considerable and growing attention are silica-based optical fibres. These fibres, either commercially available or locally fabricated, have been

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investigated for their thermoluminescence (TL) utility in radiotherapy and diagnostic ranges, including via irradiation to protons [1], diagnostic X-rays [2], photons [3], alpha particles [4], electrons [5], synchrotron radiation [6] and fast neutrons [7]. Their performance has also been demonstrated in radiation-based applications, such as in environmental monitoring [8], quality audits and radiation dose inter-comparisons [9], postal radiotherapy dose audits [10], radiation dose verification in intensity modulated radiation therapy [11], the food irradiation industry [12] and positron emission tomography-computed tomography myocardial perfusion examination [13]. In all such studies, the fibres have yielded promising results in terms of radiation dose detection.

In TL dosimetry, the key factor in determining the stability of any TL material prior to their use in dosimetric applications is the temperature at which the peak of the glow curve occurs. Such study is essential, providing important understanding of the electron-trapping system, being associated with the irradiation conditions and time-temperature profile. As an instance, typically lithium fluoride-based (LiF-based) dosimeters such as TLD-100 demonstrate a TL glow curve, the shape of which is formed of a close series of typically undifferentiated narrow-peaks [14]. This condition suggests that the defects in TLD-100 give rise to single localised levels, a classical characteristic for a crystalline media. It is known that LiF-based dosimeters possess one of the most complicated TL glow curves of all known dosimetric TL materials due to its complex energy trapping system [15]. At least ten glow peaks have been identified from this LiF material in the deconvoluted TL glow curve, ranging from room temperature to 400 °C [14]. In addition, the TL glow curve peak for TLD-100 is located at relatively high temperatures (a few hundred degrees C), reflective of deep electron traps. As such, the traps remain stable over a relatively long period of time, accommodating the practicality of readout delay post-irradiation.

In regard to the silica-based fibres (a predominantly amorphous media, potentially with microcrystalline inclusions), previous studies have reported that Ge-doped fibres exhibit a broad range of thermal excitation after irradiation with photon beams [16-18]. In terms of their shape, flat-form fibres have been observed to be represented by a bi-peaked TL glow curve, regardless of optical fibre dimensions [19-20]. This double-peaked TL glow curve is indicative of a microcrystalline structure to the predominant amorphous media and deep-trap phenomenon related to the strain the fibres sustain in collapse into a flat shape. In other words, the double-peaked condition implies that the flat fibres probably have two trapping systems with different recombination energies (electrons re-trapping may occur). In comparison to cylindrical optical fibre, this form is also found to develop a secondary small-peaked, growing its intensity with increment in irradiation dose [20]. This suggests that deeper traps and electron re-trapping are also possible occurrences in the cylindrical fibres. As before, the temperature at which the peak of the TL glow curve occurs is an important factor, ensuring the stability and suitable properties of the TL material. It has been reported that the optimum TL intensity of fabricated Ge-doped fibres subjected to X-ray irradiations is peaked at around 267 °C [16]. The position of maximum TL intensity at high glow-peak temperature is well-suited to TL dosimetry applications.

Although TL glow curves of fabricated Ge-doped fibres for various types of radiation beams have been reported in the literatures, their investigation in proton beam measurements has not yet been extensively studied. In present work on fabricated Ge-doped cylindrical- and flat-shaped fibres, we investigate the fundamental characteristics of the TL glow curve and radiation dose response. In this way, the intention is to obtain an improved understanding of the behaviour of the TL mechanism in respect of proton beam irradiations.

2. MATERIALS AND METHODS

2.1. Fabrication of Optical Fibres Doped with Germanium

Fabrication of the doped silica fibres employed in this study included initial use of a standard modified chemical vapour deposition (MCVD) technique. The whole fabrication process involves several phases, comprised of: (1) fusing, (2) etching, (3) soot deposition, (4) sintering, (5) collapsing, and (6) pulling. At the initial phase in the fabrication process, ultra-pure silica (i.e. Suprasil F300) glass tube (the substrate) was fused with another low-quality silica hollow tube, which acts as an exhaust to collect residue from the vapour deposition. In the subsequent phase, the inner surface of the substrate tube was cleaned by several etching passes using sulphur hexafluoride (SF_6) to improve chemical bounding between the doping elements and the substrate tube. The etching process was then followed by deposition of silica tetrachloride ($SiCl_4$), germanium tetrachloride ($GeCl_4$) bubbler and oxygen (O_2) vapours inside the rotating substrate tube at a temperature of 1900 °C. At this stage, two germanium concentration were prepared at 2.3 and 6.0 mol% of weight gas flow rate. When sufficient deposition was achieved, the soot from the deposited vapours was then sintered at higher temperature, as a result, changing the preform into a fine transparent glass. Subsequently, the Ge-doped glass tube was collapsed at a much greater temperature (approximately 2200 °C) to finally obtain a solid Ge-doped-preform rod. These processes were applied for two germanium concentration (i.e. 2.3 and 6.0 mol% of weight gas flow rate) to fabricate two collapsed-Ge-doped-preform rods. Utilising the same fabrication process, another two copy of similar Ge-doped-preform rods was fabricated, except that these two preform rods were left in an un-collapsed state.

Table 1 Details of research samples

Sample	Outer Dimension	Length	Manufacturer / Provider
Fabricated 2.3 mol% Ge-FF*	643 × 356 μm^2	6 mm	MMU-UM consortium [Malaysia]
Fabricated 6.0 mol% Ge-FF*	272 × 69.5 μm^2	6 mm	MMU-UM consortium [Malaysia]
Fabricated 2.3 mol% Ge-CF*	481 μm [diameter]	6 mm	MMU-UM consortium [Malaysia]
Fabricated 6.0 mol% Ge-CF*	486 μm [diameter]	6 mm	MMU-UM consortium [Malaysia]
Commercial 4.0 mol% Ge-CF	125 μm [diameter]	6 mm	CorActive High-Tech Inc. [Canada]
TLD-100	3.2 × 3.2 × 0.89 mm^3	-	Thermo Fisher Scientific Inc. [USA]

*Dopant concentrations are based on values in the preforms

In the pulling process, the collapsed-Ge-doped-preform rods were then pulled (drawn-down to the final desired cross-sectional dimensions) to produce conventionally shaped cylindrical fibres (hereinafter referred to as 'Ge-CF'), while the un-collapsed Ge-doped-preform rods were pulled to produce novel flat fibres (hereinafter referred to as 'Ge-FF'). The detailed procedures of this pulling process have been elaborated by Noor *et al.* [21] for the Ge-CF, and Fadzil *et al.* [22] for the Ge-FF. Table 1 shows the details of the fabricated Ge-FF and Ge-CF, including commercial optical fibres and TLD-100 dosimeters.

2.2. Samples Preparation

Commercial optical fibre (CorActive High-Tech Inc., Canada) was chosen for this study on the basis of availability. The outer plastic coating layer of this optical fibre was carefully stripped away and the remaining optical fibre was cleaned using isopropyl alcohol solution to remove any resin residues. All fibres, including those locally fabricated, were cut into nominal lengths of 6.0 (±1.0) mm. The 6.0 mm length was chosen to accommodate the fibres within the dimension of the planchet used in the TLD reader during the readout process. For ease of handling and storage, all samples were grouped into thin-walled gelatine capsules according to their types. Prior to irradiations, all samples were annealed to eliminate low-temperature TL glow curve peaks and pre-irradiation signals that possibly exist in the samples.

2.3. Proton Beam Irradiations

Table 2 Summary of parameters used for proton beam irradiation

Parameter	Setting
Energy [MeV]	150
Radiation dose [Gy]	1 to 10
Dose rate [Gy/min]	4
Source-to-surface distance [cm]	100
Beam field size [cm^2]	10 × 10

Table 2 depicts a summary of proton irradiation parameters used for exposure of the samples. A polythene-type water-equivalent phantom of cross-sectional dimensions 20 × 20 cm^2 and 14 cm in thickness (placed on top of the samples) was used to serve as the build-up medium and provide maximum absorbed radiation dose (d_{max}). Another 10 cm in thickness of the water-equivalent phantom was placed below the samples to allow for backscatter.

2.4. Signal Acquisition (Readout)

Table 3 Parameters of the time-temperature profile used for samples readout

Type of Sample	Phase	Temperature [°C]	Time [sec]	Ramp-rate [°C/sec]
Ge-FF	Preheat	120	10.0	-
	Acquisition	400	13.3	30.1
	Annealing	400	10.0	-
Ge-CF	Preheat	80	10.0	-
	Acquisition	400	13.3	30.1
	Annealing	400	10.0	-
TLD-100	Preheat	50	0.0	-
	Acquisition	260	26.7	9.8
	Annealing	260	0.0	-

A Harshaw™ 3500 TLD reader (Thermo Scientific™, USA) was used to acquire TL signals from the proton-irradiated samples. The TL signals were acquired based on the time-temperature profile as detailed in Table 3. Throughout the readout process, a nitrogen gas atmosphere of 0.5 bar was used to prevent contributions of TL signal from the chemiluminescence phenomena. The gross TL yield was normalised per unit volume to mitigate against uncertainty in TL yield (the volume for optical fibres has been calculated based on their core diameter, a region where the Ge is mainly deposited).

3. RESULTS AND DISCUSSION

3.1. Radiation Dose Linearity

Fig 1 below provides a summary of the TL signal, normalised to per unit doped-core volume, the dotted lines indicating least square fit linear trends, while the error bars portrayed are one standard deviation (1 SD) on the mean values, obtained from at least five measurements for each data point (in some cases, the error bars are smaller than the data points shown). It is observed that fabricated Ge-FF and Ge-CF offer excellent linear relationships throughout the investigated radiation dose range (1 to 10 Gy), with coefficient of determination (R^2) values of better than 0.99, providing for reliable calibration and utilisation. Similar behaviour was also observed for the TLD-100 dosimeter and commercial optical fibre (R^2 > 0.99), albeit with the TLD-100 providing much lower sensitivity than any of the fibre types. Also apparent is that the nominal 6.0 mol% Ge-FF provides the greatest response of all fibres, well-differentiated from the 2.3 mol% Ge-FF which provides the next greatest response. Identified at lower but nevertheless significant sensitivity is then the 6.0 mol% Ge-CF followed closely by the response of the 2.3 mol% Ge-CF.

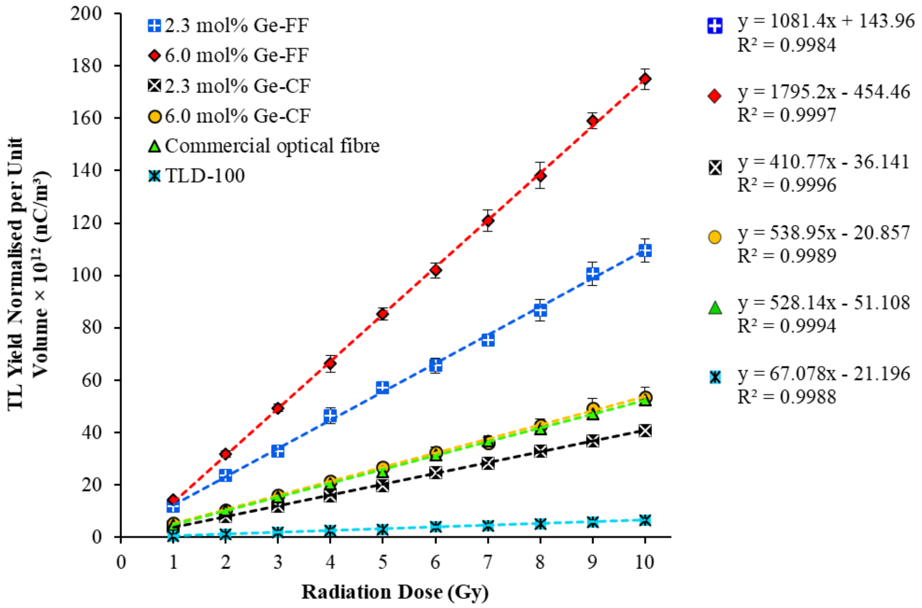

Figure 1. Radiation dose response of the investigated samples after irradiation using a proton beam of energy 150 MeV, for a range of radiation doses from 1 Gy up to 10 Gy.

3.2. Shapes of TL Glow Curves

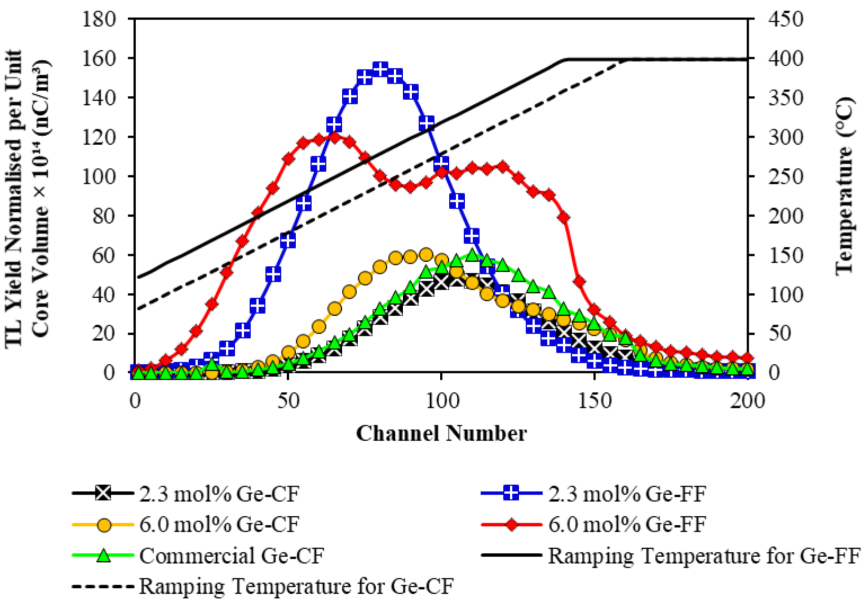

Figure 2. TL glow curves for the range of fibres studied herein, irradiated with a 150 MeV proton beam to a dose of 5 Gy.

Fig. 2 shows the TL glow curves for the Ge-FF and Ge-CF obtained subsequent to proton beam irradiation. The broad TL distribution to be observed across the range of temperatures results from the spread of electron trap levels in the silica, reflecting the structure and properties of amorphous media. Conversely, the TLD-100 glow curve forms a narrow-peak same as reported by Hassan *et al.* [23], this narrow-peak formation indicates the point defects or other such localised electron trap levels being characteristic of the crystalline material itself. In Fig. 2, the major contribution to the accumulated electrical charge (TL signal) acquired during the readout process is seen to reside within the channel number range 50 to 150 (the region of interest).

Concerning the locally fabricated dosimeters, the TL glow curve of Fig. 2, obtained at a dose of 5 Gy, shows that the greater preponderance of trapping-centres of the 6.0 mol% Ge-FF provides a pronounced increment in TL yield over that of 2.3 mol% Ge-FF. A less clear situation arises in the observation of TL yield for the locally fabricated cylinder-shaped fibres, such that at 5 Gy and within measurement uncertainty, the 2.3 mol% Ge-CF gives a comparable TL signal to that of the 6.0 mol% Ge-CF. For both types of fibre (Ge-FF and Ge-CF), the fabrication process begins with a preform, one that results in collapsing of the cylinder walls and with it considerable strain, generating additional trapping-centres that can modify TL yield. Further strain results from the drawing down of the preform into fibres of both the shapes dealt with herein. The situation can be interpreted to be interplay between the number of trapping centres and their density, retrapping with a likelihood which increases with dopant concentration and strain related defects generation, all occurring at various depths. As such, the phenomenological response is not one that can be lightly interpreted on the basis of there existing a simple linear dependence on extrinsic dopant concentration.

In assessing TL glow curve peak, the fabricated 2.3 mol% Ge-CF and commercial Ge-CF are essentially composed of a single peak, each located centrally within the channel range. The positive skew that is brought about by a shoulder to the distribution has previously been related to strain generated defects [20]. Similarly observed is a single TL glow curve peak for the fabricated 6.0 mol% Ge-CF fibre, again with a positive skew associated with a shoulder and a shift of the distribution to lower temperatures. The 2.3 mol% Ge-FF glow curve distribution shifts further towards lower temperatures. Finally, for the fabricated 6.0 mol% Ge-FF, the TL glow curve clearly manifests as a double-peaked distribution, the previously observed shoulder growing in yield, with progressive shift of the distribution to the left. The downward shift of the TL glow curve has been associated with re-trapping of the more deeply trapped electrons [22].

Ghomeishi *et al.* [20] also found the Ge-CF to have developed a secondary small-peaked feature, starting at around 330 °C, growing in intensity as radiation dose increased (in the range from 0.5 to 8 Gy). Further reported was the observation of TL glow curve intensity terminating at a maximum temperature of 400 °C. This was interpreted to be due to the presence of deep-seated trapped electrons incompletely released due to limited activation energy. In present study no abrupt termination of TL intensity in the TL glow curves has been observed (Fig. 2). This indicates the time-temperature profile used in present work to be optimal, the TL glow curves, being free of the effects of superficial traps (flushed out by the preheat cycle). The trap parameters of maximum TL glow curve peak of all the investigated samples are shown in Table 4 below, these parameters are obtained based on their TL glow curve formation as presented in Fig. 2 and Hassan *et al.* [23].

Table 4 Trap parameters obtained at maximum TL glow curve peak after irradiated at 5 Gy

Sample	Temperature [°C]	Channel No.	TL × 10^{14} [nC/m^3]
Fabricated 2.3 mol% Ge-FF	281	81	155.30
Fabricated 6.0 mol% Ge-FF	237[a], 346[b]	59[a], 114[b]	121.24[a], 105.17[b]
Fabricated 2.3 mol% Ge-CF	289	105	47.59
Fabricated 6.0 mol% Ge-CF	263	92	60.84
Commercial 4.0 mol% Ge-CF	299	110	60.18
TLD-100	232	137	15.82

a: First TL glow curve peak
b: Second TL glow curve peak

3.3. Effects of TL Glow Curves at Different Radiation Doses

The effects of TL glow curves formation on fabricated Ge-FF and Ge-CF towards different radiation doses are shown in Fig. 3, the height of the maximum in TL glow curves increasing progressively with radiation dose within the investigated range of 1 to 5 Gy. Also observed is that the general structure of TL glow curves for all samples remains unchanged over that dose range.

In each type of the fabricated Ge-doped optical fibre, the maximum in the TL glow curve peak is seen to occur at approximately the same temperature across the investigated radiation dose range (Fig. 3).

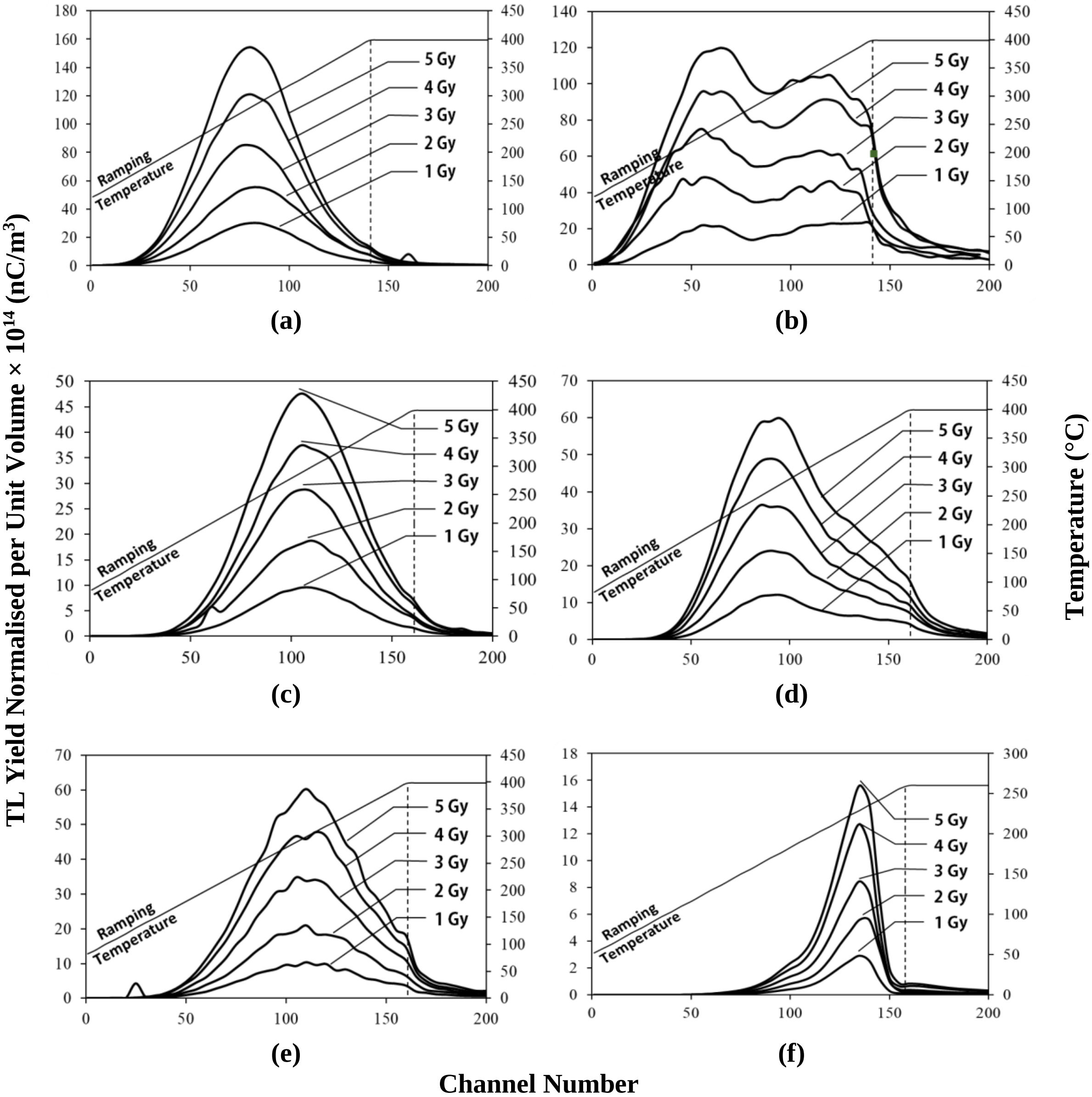

Figure 3. TL glow curves for (a) 2.3 mol% Ge-FF, (b) 6.0 mol% Ge-FF, (c) 2.3 mol% Ge-CF, (d) 6.0 mol% Ge-CF, (e) commercial optical fibres, and (f) TLD-100 after irradiated with proton beam at energy of 150 MeV for five different radiation doses (1 to 5 Gy).

4. CONCLUSION

Fundamental studies have been conducted of TL glow curve and radiation dose response of fabricated Ge-doped cylindrical- and flat-fibres subjected to proton beam irradiation. The general structure of the TL glow curves manifests as broad shapes, differing from the more narrowly peaked feature of TLD-100. The TL glow curves shapes are shown to have physical composition and fabrication dependencies, a complex interplay of defect density, trap depth and re-trapping. It is nevertheless clear that the fabricated Ge-FF and Ge-CF are able to sensitively detect proton beam doses, responding linearly within the investigated dose range, one that covers the fractionated doses of proton radiotherapy. We intend further investigating of the TL mechanism of these fibres in terms of TL glow peak and kinetic parameters based on deconvolution analysis.

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