



# Ge and B doped collapsed photonic crystal optical fibre, a potential TLD material for low dose measurements



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## HIGHLIGHTS

- We study Ge and GeB doped Photonic Crystal Fibre (PCF) dosimeters for low dose measurements.
- The irradiation response of PCF dosimeters to X-rays and gamma-rays are described.
- The presence of boron manifestly improves the sensitivity of the PCF dosimeters.

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## ABSTRACT

Offering a number of advantageous features, tailor-made silica-based fibres are attracting attention as thermoluminescence (TL) dosimeters. We have performed a detailed study of the TL properties of Ge-doped and Ge-B-doped collapsed photonic crystal fibres (PCFc), most particularly with regard to their potential use for the environmental and X-ray diagnostic dose monitoring. Extrinsic doping and defects generated by strain at the fused inner walls of the collapsed fibres result in the PCFc-Ge-B and PCFc-Ge fibres producing markedly greater TL response than that of the phosphor-based dosimeter TLD-100, by some 9 and 7 ×, respectively. The linearity of TL yield has been investigated for X-ray doses from 0.5 mGy to 10 mGy. For a dose of 1 Gy, the energy response of the PCFs and TLD-100 has been studied using X-rays generated at accelerating potentials from 20 kVp through to 200 kVp and for the 1.25 MeV mean gamma-ray energy from <sup>60</sup>Co. The effective atomic number,  $Z_{eff}$  of PCFc-Ge and PCFc-Ge-B was estimated to be 12.5 and 14.4, respectively. Some 35 days post-irradiation, fading of the stored TL signal from PCFc-Ge-B and PCFc-Ge were found to be ~15% and 20% respectively, with mean loss in TL emission of 0.4–0.5% per day. The present doped-silica collapsed PCFs provide greatly improved TLD performance compared to that of previous fibre designs and phosphor-based TLD-100.

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## 1. Introduction

Thermoluminescence dosimeters (TLD) are widely used in support of a range of radiation-dependent endeavours, encompassing for example radiation medicine, material damage and environmental studies. For penetrating photons, the performance of the TLD requires to be matched to the mass-dependent sensitivities, doses, dose gradients and dose-rates of interest. With drawbacks existing in use of the well-established phosphor-based LiF:Mg,Ti commercial product TLD-100, including its hygroscopic nature and relatively poor spatial resolution (~mm), novel materials based on

SiO<sub>2</sub> fibres are currently being introduced (Mahdiraji et al., 2015), fabricated using the modified chemical vapour deposition (MCVD) technique. The silica-based optical fibres exhibit a number of advantageous physico-chemical characteristics, including being mechanically robust, chemically inert, biocompatible and re-usable, also being easily sterilisable via simple heating etc. A remaining challenge has been that of improving the TL yield of the fibres, a matter attracting the attention of several researchers, including Mahdiraji et al. (2015), with present work forming part of the evaluation of this particular aspect.

The work started with capillary optical fibres (COF), subsequently moving on to the collapsing down of pure silica hollow capillary fibres into flat fibres (FF), and then further still by stacking a group of pure SiO<sub>2</sub> finer-bore capillaries into an outer of glass tube to form what is known as a Photonic Crystal Fibre (PCF).

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By collapsing down the PCF assembly, to be subsequently referred to as collapsed PCF (PCFc) (Dermosesian et al., 2015), it has been shown that considerable enhancement in TL yield can be obtained, the fused inner walls generating defects beyond that existing in the unstrained media. In irradiation by 6 MeV electrons, the mass-normalized TL yield of the pure FF and pure uncollapsed PCF have been shown to be improved by  $\sim 12 \times$  and  $\sim 17.5 \times$  compared to that of pure COF (Bradley et al., 2015). Further, in making use of 20 MeV electron beam irradiation, the TL yield of pure collapsed PCF (PCFc) has been shown to be  $\sim 7.5 \times$  of that of pure uncollapsed PCF (Dermosesian et al., 2015).

The outcome of such studies can be linked with the results of previous investigations in which the focus has been on extrinsically doped tailor-made silica fibres, doped with atomic species of Ge, Al (Yaakob et al., 2011), Ba and Li, zirconium oxide ( $ZrO_2$ ) (Villa-Sanchéz et al., 2007), manganese doped calcium tetraborate ( $CaB_4O_7$ : Mn) nanocrystal (Tabatabaei et al., 2006), Cd and Zn (Tiwari et al., 2014). Compared to pure COF, the TL yield has been shown to be incremented by  $\sim 3 \times$  when doped by Ge (Mahdiraji et al., 2015) and by  $\sim 6 \times$  for Ge-doped COF collapsed into FF (Bradley et al., 2015). Most notably, when Ge and B codopants have been introduced into a preform of COF, to subsequently be collapsed down into FF, its TL response has been observed to increase by a factor of 31 (Mahdiraji et al., 2015). Although it is clear that dopants and additional defects induced during the drawing-down process help to increase TL yield, there is little information on the effect of different dopants in collapsed PCFs. As such, herein collapsed PCFs with different dopants of Ge and B have been studied. For this, collapsed PCFs and TLD-100 (the latter acting as a comparator) have been exposed to energetic photons to obtain the dose versus TL response, energy dependence, effective atomic number and fading of these TLDs. These novel media have then been proposed for environmental and X-ray diagnostic dose applications, previous work on silica and doped silica fibres being almost entirely based on studies at radiotherapy doses ( $\sim$ Gy to in excess of 10 Gy) rather than the sub mGy to mGy doses that now become of operational interest.

## 2. Materials and methods

### 2.1. Sample preparation

Using the modified chemical vapour deposition (MCVD) process, fabrication has been made of silica-based PCF doped with 8.5% by weight of Ge or Ge-B. The glass tube enclosing the stacked capillaries was drawn into fibre using the fibre pulling tower located at the Department of Electrical Engineering, University of Malaya. During pulling, a vacuum pressure was applied from the top of the glass tube in order to collapse the tube into a flat shape. This causes the inner walls to fuse, generating defects. Later, the preform was re-pulled into 125  $\mu$ m and 140  $\mu$ m fibre size (matching with typical values for commercially available optical fibres that have been investigated as TLDs in the past (Dermosesian et al., 2015)), as shown in Table 1. Herein, 0.5  $\pm$  0.1 cm of PCF rod was cut using a diamond-cutter and weighed using an electronic balance.

**Table 1**  
Respective types, diameter and mass of fibres used.

Sample	Diameter ( $\mu$ m)	Mass (mg)
PCFc-Ge	125 $\pm$ 10	0.1 $\pm$ 0.1
PCFc-Ge-B	140 $\pm$ 10	0.1 $\pm$ 0.1

**Table 2**  
Respective results of collapsed PCFs using EDX analysis.

Element	Weight fractional contribution of each element (%)	
	PCFc-Ge	PCFc-Ge-B
O	65.78	30.25
Si	32.96	50.18
Ge	01.26	01.34
B	0	18.23

The response of the PCFs have been compared with TLD-100 chips (Thermo Fisher Scientific Inc., Waltham, MA, USA), the latter being of dimension  $3.2 \times 3.2 \times 0.89$  mm<sup>3</sup> and of mean mass 23.4 mg. For readout, use has been made of a Harshaw 3500 TL reader (USA) supported by WinREMS software. The time-temperature profile (TTP) was set to a preheat temperature of 50 °C, heating rate of 10 °C s<sup>-1</sup> and maximum temperature for data acquisition of 400 °C. A slow flow of nitrogen gas was supplied during the read out process to inhibit sample oxidation. For each sample, the TL yield was normalized to its mass, obtaining results in  $\mu$ C/g.(Table 2).

### 2.2. Annealing process

Annealing of the PCF and TLD-100 was carried out with a Harshaw furnace held at 400 °C. The samples were annealed for a period of 1 h, removing any previously stored triboluminescence and irradiation history, also stabilising the trap structure. As a control measure, upon the cessation of annealing, with the fibres remaining in the furnace, additional thermal stress has been avoided by allowing the samples to cool down naturally over a period of 24 h (Hashim et al., 2015). In order to minimize exposure to ambient light levels the samples were then retained in individual small plastic bags and placed in a light-tight box ready for subsequent irradiation.

### 2.3. Sample irradiation to provide for selection of uniformly responsive dosimeters

To provide for selection of uniformly responsive dosimeters, irradiations were made using an ERESO model 200 MF4-RWX-ray machine, with inherent filtration of 0.8 mm  $\pm$  0.1 mm Be. For the irradiations process, the dosimeters were placed on the central axis of the source at a focus to surface distance (FSD) of 38 cm. The complete set of dosimeters were given a dose of 1 Gy of X-rays, allowing for selection of only those PCFs and TLD-100 providing uniform response to dose to within  $\pm$  5% of the mean TL yield. Only the selected TLDs were used in subsequent study, as below.

#### 2.3.1. Dose response to low-doses of X-rays

For study of TL versus doses within a range typically familiar in diagnostic X-ray radiology, a nominal X-ray tube potential of 80 kVp was selected, with delivered doses ranging from 0.5 mGy to 10 mGy, measured using a calibrated PTW UNIDOS ionization chamber.

#### 2.3.2. Energy dependence

For study of energy dependence, nominal X-ray tube potentials ranging from 20 kVp to 200 kVp were selected. Identical doses of 1 Gy were employed. Again for a dose of 1 Gy, making use of a large activity <sup>60</sup>Co source located at the Physics Department, University Malaya, irradiations of the TLDs were obtained at the mean gamma-ray energy of 1.25 MeV. Due to the polyenergetic nature of X-ray beams, it is typical for the effective energy to be

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