



## Thermoluminescence studies on Cu-doped $\text{Li}_2\text{B}_4\text{O}_7$ single crystals

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### ABSTRACT

Lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ) is a tissue equivalent material and single crystals of this material doped with Cu are promising for dosimetric applications. In the present study highly transparent single crystals of lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ) doped with Cu (0.5 wt%) have been grown using the Czochralski technique. The  $\text{Li}_2\text{B}_4\text{O}_7$ :Cu crystals were studied using photoluminescence, X-ray diffraction (XRD), UV–vis transmission, time resolved fluorescence and thermoluminescence (TL) techniques. The TL readout of  $\text{Li}_2\text{B}_4\text{O}_7$ :Cu crystals showed two well-defined glow peaks at 402 K (peak-1) and 513 K (peak-2) for a 4 K/s heating rate. While the low temperature TL peak-1 fades completely within 24 h at room temperatures, the main dosimetric peak-2 remains the same. The TL sensitivity of the grown single crystal is found to be 3.3 times that of a conventional TL phosphor, TLD-100. The  $\text{Li}_2\text{B}_4\text{O}_7$ :Cu crystals showed a linear TL dose–response in the range from 1 mGy to 1 kGy. The TL analysis using a variable dose method revealed first order kinetics for both the peaks. Trap depth and frequency factor for peak-1 were found to be 0.81 eV and  $5.2 \times 10^9 \text{ s}^{-1}$ , whereas for peak-2 the values were 1.7 eV and  $1.7 \times 10^{16} \text{ s}^{-1}$ , respectively.

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### 1. Introduction

Lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ; abbreviated as LTB) is one of the preferred materials for personal thermoluminescence (TL) dosimetry. This is a tissue equivalent material as its effective atomic number  $Z_{\text{eff}}=7.25$  closely matches with the  $Z_{\text{eff}}$  of human tissue [1]. LTB has very low photon energy dependence and low detection limit ( $\approx 10 \mu\text{Gy}$ ) along with a simple annealing procedure but low TL sensitivity compared with other common TL phosphors. Several dopants and codopants were tried to increase TL sensitivity [1–8]. Lithium borate doped with Mn was the first material to be reported for TL dosimetry [2] but, it has problem of low TL sensitivity and an emission in the 600 nm region, far from the good response region for most of the photomultipliers. Other dopants as alternative to manganese were tried in order to produce a blue-emitting phosphor. But initially only silver doped LTB (emission at 290 nm) resulted in a comparable thermoluminescence efficiency [7]. The doping with a rare earth element improved the linearity but not sensitivity [8]. Copper doped LTB as a TL phosphor was suggested for radiation dosimetry by Takenaga et al. [3]. Its TL sensitivity is reported to be 20 times higher than that of  $\text{Li}_2\text{B}_4\text{O}_7$ :Mn produced by Schulman

et al. [2]. So far copper doped LTB has been found to be the most successful phosphor. Recently it was reported that co-dopants such as In, Ag and Ag+P help in further enhancing TL sensitivity of the LTB:Cu phosphor, as well as in making its response to photons nearly tissue equivalent [4,9]. The LTB:Cu phosphor is widely studied for its TL dosimetric properties and also is used (in the form of sintered pellets) in commercially available Panasonic TL dosimeters. Different methods of preparation of this phosphor result in rather different TL characteristics, including TL glow curve, sensitivity, dose linearity, etc. [10–14]. Table 1 gives a brief review and TL characteristics of  $\text{Li}_2\text{B}_4\text{O}_7$  based phosphors prepared using different methods. However; until now in most of the studies TL properties of sintered polycrystalline and glassy phosphor have been reported.

It is reported that single crystals show higher TL intensity, thermal stability and low hygroscopicity as compared with polycrystalline materials [14–16]. Shahare et al. [17] also showed that crystalline LTB:Cu has good TL sensitivity compared with glassy LTB. Senguttuvan et al. [18] has reported that Cu-doped LTB has the most intense emission for UV excitation. The LTB single crystals are transparent to visible light, making them very effective for collection of emitted light from the inner part of the sample. The resistance to humidity due to its stoichiometric composition and easy handling compared with powder samples make the  $\text{Li}_2\text{B}_4\text{O}_7$  single crystals an ideal material for radiation dosimetry. LTB:Cu single crystals with a small concentration of Cu

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**Table 1**Comparison of characteristics of doped  $\text{Li}_2\text{B}_4\text{O}_7$  TL phosphors prepared using different methods.

| S. no. | Phosphor and method of preparation                                                                                                                                                                                                                                                                                                                                                                          | TL sensitivity relative to LiF:Mg,Ti (TLD-100)                                                                   | TL emission peak (nm) | Dose range                                           | Remarks                                                                                                                                                                                                                                                                                                |
|--------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------|-----------------------|------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1      | $\text{Li}_2\text{B}_4\text{O}_7\text{:Mn}$ (0.1 wt% using the method of Schulman et al. [2].<br>$\text{Li}_2\text{CO}_3$ and $\text{H}_3\text{BO}_3$ as starting materials are mixed along with appropriate amount of dopant, melted at 950 °C and then rapidly cooled to room temperature. The resulting glass material is reheated at 650 °C to complete crystallization Main TL peak ~200 °C            | 0.4                                                                                                              | 600                   | 0.1 mGy– $10^3$ Gy<br>(supra-linear at higher doses) | Emission at 600 nm is far from the good response region of most photomultipliers<br>The phosphor is affected by humidity.                                                                                                                                                                              |
| 2      | $\text{Li}_2\text{B}_4\text{O}_7\text{:Mn, Si}$ (0.1, 0.25 wt%),<br><br>Preparation method is similar to that of Schulman et al. [2].                                                                                                                                                                                                                                                                       | ~0.4                                                                                                             | 600                   | 0.1 mGy– $10^3$ Gy                                   | Adding silica (0.25% by weight) improves moisture resistance [10]                                                                                                                                                                                                                                      |
| 3      | $\text{Li}_2\text{B}_4\text{O}_7\text{: Ag}$ (0.1 wt%) [7]                                                                                                                                                                                                                                                                                                                                                  | Marginal improvement in the sensitivity and is comparable to that of $\text{Li}_2\text{B}_4\text{O}_7\text{:Mn}$ | 290                   | –                                                    | –                                                                                                                                                                                                                                                                                                      |
| 4      | Preparation method is similar to Schulman et al. [2].<br>$\text{Li}_2\text{B}_4\text{O}_7\text{: Cu, Ag}$ (0.02, 0.02% by weight)                                                                                                                                                                                                                                                                           | –                                                                                                                | 360                   | –                                                    | Reported to be least attractive for medical dosimetry [11]                                                                                                                                                                                                                                             |
| 5      | Preparation method is similar to Schulman et al. [2].<br>$\text{Li}_2\text{B}_4\text{O}_7\text{: Cu}$ .<br><br>$\text{Li}_2\text{B}_4\text{O}_7$ powder containing the activator compound is heat-treated in air in a platinum boat for about 1 h at a temperature of 900–913 °C, just below the melting point (917 °C) of $\text{Li}_2\text{B}_4\text{O}_7$<br>TL peak ~205 °C at a heating rate of 7 °C/s | 3<br><br>(20 times higher than that of $\text{Li}_2\text{B}_4\text{O}_7\text{:Mn}$ )                             | 360                   | $3 \times 10^{-4}$ – $10^3$ Gy [3]                   | Phosphors prepared by stoichiometric matrix composition exhibit greater moisture resistance [13]<br><br>Pradhan et al. [12] studied the suitability of various raw materials with regard to the presence of unintentional impurities, for preparation of $\text{Li}_2\text{B}_4\text{O}_7\text{:Cu}$ . |
| 6      | $\text{Li}_2\text{B}_4\text{O}_7\text{:Cu}$ (0.1 mol%). Solid state diffusion method. The thoroughly mixed powders were heated at 700 °C for several hours and then slowly cooled [17]                                                                                                                                                                                                                      | 4                                                                                                                | 369                   | 0.27–10 Gy                                           | –                                                                                                                                                                                                                                                                                                      |
| 7      | $\text{Li}_2\text{B}_4\text{O}_7\text{: Cu}$ single crystals grown by the Czochralski method [14].                                                                                                                                                                                                                                                                                                          | Sensitivity higher than those of the powder samples                                                              | 366                   | $2 \times 10^{-4}$ –20 Gy                            | Minimum detectable dose (MDD) is 10 µGy. Together with higher sensitivity, single crystals are much less hygroscopic.                                                                                                                                                                                  |
| 8      | $\text{Li}_2\text{B}_4\text{O}_7\text{: Cu}$ sintered pellets. Main TL peak ~210 °C. [1]                                                                                                                                                                                                                                                                                                                    | 0.5                                                                                                              | 360                   | $10^{-4}$ – $3 \times 10^3$ Gy                       | MDD is 20 µGy. During preparation $\text{SiO}_2$ was added for preventing effects of humidity                                                                                                                                                                                                          |
| 9      | $\text{Li}_2\text{B}_4\text{O}_7\text{: Cu, In}$ (0.07, 0.05 Wt%), sintered (~850 °C) pellets. Main TL peak ~210 °C. [4]                                                                                                                                                                                                                                                                                    | 0.7–1.0                                                                                                          | 360                   | $10^{-4}$ – $3 \times 10^3$ Gy                       | MDD is 10 µGy. During preparation $\text{SiO}_2$ was added.                                                                                                                                                                                                                                            |
| 10     | $\text{Li}_2\text{B}_4\text{O}_7\text{: Cu, In, Ag}$ (0.07, 0.07, 0.05 wt%). Main TL peak ~190 °C [4]                                                                                                                                                                                                                                                                                                       | 2.0                                                                                                              | 360                   | $10^{-4}$ – $3 \times 10^3$ Gy                       | MDD is 10 µGy                                                                                                                                                                                                                                                                                          |
| 11     | $\text{Li}_2\text{B}_4\text{O}_7\text{: Cu, Ag, Mg}$ (0.02% by weight each) prepared by the sintering technique [9]<br><br>Main TL peak ~219 °C                                                                                                                                                                                                                                                             | –                                                                                                                | 421, 380, 350         |                                                      | The proper concentrations of Ag and Mg can enhance the TL of $\text{LTB:Cu}$ . Dopant Ag can enhance the intensity of TL peak at ~219 °C and reduce that of TL peak at ~130 °C [10]                                                                                                                    |

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