



# The effect of synthesis and doping procedures on thermoluminescent response of lithium tetraborate

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## ARTICLE INFO

### Article history:

Received 24 May 2010

Received in revised form

22 September 2010

Accepted 8 November 2010

Available online 13 November 2010

### Keywords:

Lithium tetraborate

Synthesis

Doping

Characterization

Thermoluminescence

## ABSTRACT

Lithium tetraborate has been a scientific focus since 1960s by the courtesy of the thermoluminescence property it possesses. Moreover, it is utilized in surface acoustic wave apparatuses, in sensor sector and in laser technology owing to its non-linear optical characteristics. For the uses in thermoluminescence dosimetry lithium tetraborate is activated by addition of a variety of metals as dopants.

This study includes the synthesis of lithium tetraborate by two methods (high temperature solid state synthesis and water/solution assisted synthesis), doping of activators into the matrix material synthesized and characterization of the products. Lithium tetraborate is readily commercially available in TL (Thermoluminescence) dosimetry; hence, the main aim in this study was to specify the effect of synthesis and doping methods on the TL response.

The heating temperature for the synthesis was 750 °C and the retention time as selected as 4 h for both methods. The synthesis stages were followed by doping step where the compounds of Cu, Ag and In in different proportions were doped in lithium tetraborate by solid state and solution assisted doping techniques. Characterization of the product was achieved by X-ray diffraction (XRD), Fourier transform Infra Red Spectroscopy (FT-IR) and Scanning Electron Microscopy (SEM) techniques. All samples prepared displayed TL response and the best TL signal was obtained from the sample produced by solid state synthesis and doped by solution assisted method with 0.1% Cu and 0.004% Ag.

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## 1. Introduction

The luminescence characteristic of materials has attracted attention of human beings since very old times [1,2]. This very initial concern about luminosity grew into an enormous curiosity resulting in incalculable investigations regarding light emission of materials. The kind of luminescence displayed by the objects that release the energy, stored from irradiation, in the form of light when exposed to heat is defined as *thermoluminescence* (TL).

A number of thermoluminescent materials are available for the use in radiation dosimetry, (clinical, radiotherapy, personnel) [3], and are functional in archeological and geological dating. The intensive studies carried out by researchers on TL materials brought their fruits as a bunch of different dosimeters. Among these materials, what make lithium tetraborate an attractive candidate for personnel dosimetry are its effective atomic number linear dose response up to high doses, low fading, simple annealing procedure, etc.

None of the commercial dosimeters except for the one containing lithium tetraborate are tissue equivalent. However, it is important that the dosimeter used to measure human exposure to radiation show similar atomic properties with human tissue. The effective atomic number of lithium tetraborate is (7.42), which is almost the same as that of the biological tissue.

Moreover, in contrast to commonly used TL materials, lithium tetraborate possesses perfect linearity as a particularly useful TL property, when doses up to 10<sup>3</sup> Gy are applied. Linearity can be observed rarely over 10 Gy which makes lithium borates rather distinct among other dosimeters [2,4,5]. El-Faramawy [6] determined the minimum detectable dose for lithium tetraborate as 20 μGy. However this value went down to 6 μGy in the investigation executed by Prokic [4].

Fading refers to the reduction in radiation induced signal, i.e., intensity of a peak in a time span in the thermoluminescence terminology. Certainly, material to be used for TL dosimetry purposes is required to have low or no fading. Thus, fading can be regarded as a quality indicator for the dosimeter. The product of El-Faramawy [6] was declared to fade 11% at the end of the third month. The sample of Furetta's [2] study displayed similar fading with a percentage of 10% in three months. Prokic [4] obtained samples for which the

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**Table 1**  
Glow peaks of lithium tetraborate.

Dopant	Researcher	Main peak temperature (°C)	Shoulder peak temperature (°C)
Cu–In	Furetta [2]	210	125
Cu	Manam [9]	175	290, 325
Cu–Ag–In	Prokic [7]	190	125, 225
Cu–Ag–In	Prokic [7]	210	125
Cu–Ag–P	Prokic [4]	185–190	135
Cu	Cuong [3]	230	

main peaks faded 10% in the first three months which is a quite slow rate of fading. In this study the peak at the low temperature region faded in 48 h as it may be expected.

The practicality of annealing is important when successive read-outs should be made. Annealing is a heating procedure applied to erase the previous radiations from the material and to prevent interference of its effect on the measurement. The annealing procedure is quite simple for the case of lithium tetraborate [2,7,8].

Simple glow curve is another important property for the production of dosimeters. The glow curves are plotted by a software connected to TL measurement systems and illustrate the thermoluminescence intensity versus temperature. The glow curves are particularly important in the sense that they are the main indicators of whether a material can be used for TL dosimetry purposes or not. Generally it is desired that the glow curve gives a simple, if possible single, peak around 200 °C. Table 1 summarizes the glow curve peaks for lithium tetraborate recorded by different researchers.

To date, lithium tetraborate was synthesized in different forms, namely: powder, pellet, single crystal, glass. The lithium tetraborate in powder form can be used for thermoluminescence dosimetry purposes. In spite of the fact that powder lithium tetraborate was claimed to be inconvenient for the use in this area due to contamination risk which may be prevented by utilization of badges as made by Panasonic that produced TLD badges with lithium tetraborate powder in [7]. Pradhan [10] emphasized the possibility of contamination of the powder as a demerit of this form. However, once the powder product is placed on badges, they are covered with a thin, translucent layer of film which protects the dosimeter from getting polluted. The main advantage of powder production against other types is that the energy consumed during the process is expected to be less than that for glass and crystal formation since in latter cases higher temperatures are employed to melt material first.

There are a few techniques to obtain lithium tetraborate as powder. It was produced by heating of a hydrated precursor of lithium tetraborate which is  $\text{LiB}_5\text{O}_6(\text{OH})_4 \cdot \text{H}_2\text{O}$  at 600 °C. Another alternative in the same method was starting with a mixture of lithium borates such as  $\text{LiB}_2\text{O}_3(\text{OH}) \cdot 3\text{H}_2\text{O}$  and  $2\text{LiB}_5\text{O}_6(\text{OH})_4 \cdot 3\text{H}_2\text{O}$  or a mixture of  $\text{LiB}_2\text{O}_3(\text{OH}) \cdot \text{H}_2\text{O}$  and  $\text{H}_3\text{BO}_3$  [11]. The second technique applied to produce powder lithium tetraborate is the wet reaction. This method was applied by Furetta [2] who performed wet reaction with stoichiometric amounts of  $\text{Li}_2\text{CO}_3$  and  $\text{H}_3\text{BO}_3$ . The same method was undertaken by Prokic [7] in 2001. Thirdly, solid state synthesis option was performed by Sangeeta [12] whose production procedure was milling and homogenizing  $\text{Li}_2\text{CO}_3$  and  $\text{H}_3\text{BO}_3$  powders and sintering the mixture at 700 °C for 48 h. Solid state synthesis was employed also by Jubera [13] who aimed at producing lithium rare earth borates with initial reactants of  $\text{H}_3\text{BO}_3$  and  $\text{LiBO}_2$ ,  $\text{LiOH} \cdot \text{H}_2\text{O}$ . The heating route tracked was keeping the mixture at 400 °C for 2 h and at 700 °C for a time span during which all the nitric vapors in the system, due to dissolving the constituents in nitric acid, could be evaporated. Then after, different sintering temperatures are chosen for different products.  $\text{Li}_2\text{B}_4\text{O}_7$ :Cu in form of powder was prepared to study thermally stimulated luminescence materials in the radiotherapy [3]. The product in the powder form

can be pelletized for a better practicability and ease in laboratory measurements. On the other hand, the final products in pellet form are quite fragile and are vulnerable against breakage [10].

Researchers up to date have not stucked to a group of elements but they have studied on numerous different metals. Table 2.2 summarizes a number of dopants studied.

Manganese doped lithium tetraborate was studied by Wall [14], Lorrain [15], Park [16] and Ignatovych [17]. The main activator in our case, i.e., Copper is the most notable metal as dopant. Copper nitrates, chlorides and oxides have been employed as activator depending on the method of doping. Copper was added as 0.24, 0.40, 0.38, 0.01–1, 0.007–0.05, 0.08–0.37, and 0.03% by weight of the matrix in different studies. Sometimes copper was assisted by another metal which is named to be the co-activator. These elements added in a smaller portion down to 10% of the activator are proved to increase the response intensity. There are a few co-activators utilized with copper. For instance, copper and silver are proclaimed to be a very effective couple considering the thermoluminescence intensity. Moreover, Indium can be added solely or can serve as a luminescence center together with silver when the activator is copper [7,18,14,19].

Although lithium tetraborate is an extensively studied material in terms of thermoluminescence, no communication investigating the relationship between TL response and synthesizing and doping techniques have been encountered. Hence this study is undertaken to determine whether or not the TL response is affected by these procedures and to investigate TL responses for different dopant combinations.

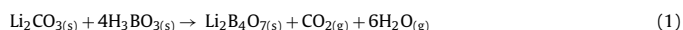
## 2. Materials and methods

### 2.1. Production of lithium tetraborate by high temperature solid state synthesis

Stoichiometric quantities of  $\text{Li}_2\text{CO}_3$  and  $\text{H}_3\text{BO}_3$ , after mixing homogenously were put into furnace in porcelain crucible and heated up to 400 °C from room temperature with a heating rate of 400 °C/h. Retention time at this temperature was selected as 2 h. After this preliminary heating stage, the sample in crucible re-blended in agate mortar. Afterwards, the furnace was adjusted to heat the sample up to 750 °C and keep it at this temperature for 2 h. An additional regrinding stage was done at this step. Finally, the mixture was re-heated at the same temperature for another 2 h to reach the best quality lithium tetraborate.

Upon the completion of the heating cycle and cooling followed, the sample was grinded and stored in desiccators. The equation given below was the expected reaction from the stoichiometric amounts of the reactants.

The expected reaction is given below.



**Table 2**

List of dopant metals used in lithium tetraborate.

Dopant metal	Weight % of the matrix	Mole % of the matrix	Form of dopant
Mg	0.1	0.1	MgO
In	0.07/0.5		Metallic/ $\text{I}_2\text{O}_3$
Ni	0.5		NiO
Ce	0.1, 1.0, 2.0		CeO
Eu	0.016–0.008		$\text{Eu}_2\text{O}_3$
Ti	0.5		
Pr		0.01, 0.1, 0.2, 0.3, 0.5, 1.0	
K		0.1–4	
Cr	0.13 and 2.5		
Co	10		
Dy	2		
Fe		0.1	
La		0.1	
Th		0.1	
P	0.8		

**Table 3**

Dopant amounts for double doping experiments.

LBO Weight (g)	Cu %	Ag %	Cu %	In %
1	0.1	0.01	0.1	0.01
1	0.1	0.02	0.1	0.02
1	0.1	0.03	0.1	0.03
1	0.1	0.04	0.1	0.04
1	0.1	0.05	0.1	0.05
1	0.3	0.01	0.3	0.01
1	0.3	0.02	0.3	0.02
1	0.3	0.03	0.3	0.03
1	0.3	0.04	0.3	0.04
1	0.3	0.05	0.3	0.05

## 2.2. Production of lithium tetraborate by water/solution assisted synthesis method

For water/solution assisted synthesis, stoichiometric amounts of  $\text{Li}_2\text{CO}_3$  and  $\text{H}_3\text{BO}_3$  were mixed, and put in a beaker into which 15 ml of water was added for production of 1 g of lithium tetraborate. The solid liquid mixture was stirred in a heating magnetic stirrer until the constituents were sufficiently dispersed at temperatures lower than  $100^\circ\text{C}$ . The heating action provided by the stirrer evaporated the water included to a certain level in 15–20 min without disrupting dispersion action. This mixing step was followed by heating in the furnace. The mixture containing residual water was exposed to  $150^\circ\text{C}$  for 3 h in order to guarantee total evaporation of water. An intermittent grinding and mixing was not required in water/solution assisted production since the constituents were dispersed to a reliable level by the stirrer. Therefore the drying and heating was directly followed by the increase of the temperature from  $150^\circ\text{C}$  to  $750^\circ\text{C}$ . The reaction given in Eq. (1) is valid also for this case.

## 2.3. Doping of the samples

The doping procedures were inspired by the synthesis methods. In solid state doping, appropriate amount of dopant was mixed thoroughly with the matrix and heated at  $750^\circ\text{C}$  for 2 h but no initial heating was applied. In solution assisted doping, the dopant was dissolved in water to form a master solution from which required amount of dopant was obtained. Solution assisted doping was applied by heating the doped matrix at  $150^\circ\text{C}$  for two hours and at  $750^\circ\text{C}$  for 2 h.

Solid state doping was solely applied to high temperature solid state synthesis product and with 0.1–1% copper concentrations. The dopant used was  $\text{Cu}_2\text{O}$ .

Solution assisted doping was employed for the matrixes produced by both synthesis methods cited before. The concentration range for copper doped in the form of water soluble  $\text{CuCl}_2$  was 0.1–1%. The samples that showed promise were the ones produced by solid state synthesis method and doped by solution assisted method. Hence dopant combinations of different concentrations were experimented on these samples with the co-activators Ag and In. Table 3 displays the dopant concentrations for Cu–Ag and Cu–In double doping combinations while Table 4 includes the same information for the case where triple doping is experimented.

## 2.4. Characterization of the material

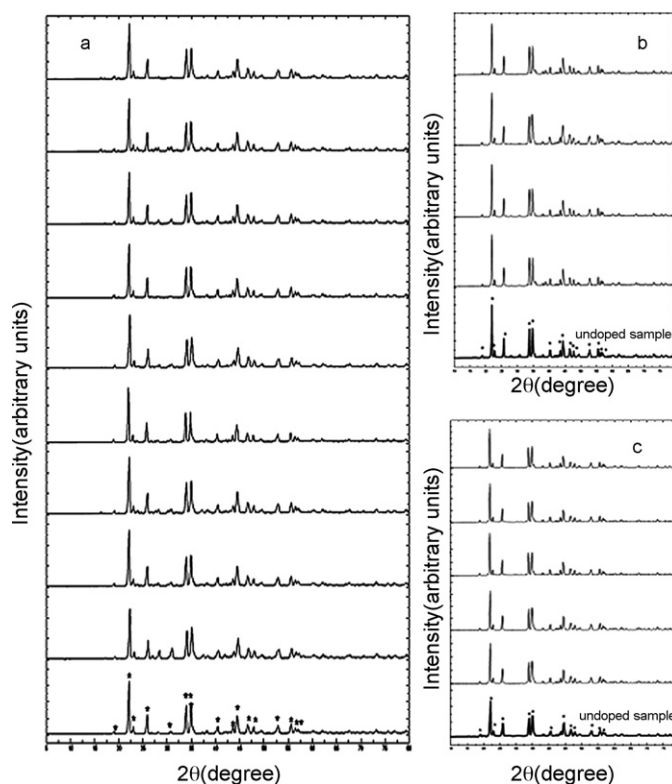
The materials produced were characterized by XRD, FTIR and SEM methods. The characterization studies aimed at detection of any structural difference caused by doping operation besides confirmation of lithium borate in the correct form namely lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ).

The X-ray Diffractometer employed for the crystal structure investigations was a Rigaku MiniFlex X-ray Diffractometer with a radiation source of  $\text{CuK}\alpha$ . The crystal structures of the lithium tetraborate were recorded within a 2 theta range of  $5^\circ$  to  $80^\circ$ . The vibrational modes of the materials produced were studied by VARIAN 1000 FTIR spectrometer between wave numbers 400 and  $4000\text{ cm}^{-1}$ . The SEM analyses

**Table 4**

Dopant amounts for triple doping experiments.

LBO Weight (g)	Cu %	Ag %	In %
1	0.1	0.04	0.01
1	0.1	0.04	0.03
1	0.1	0.04	0.05
1	0.1	0.05	0.01
1	0.1	0.05	0.03
1	0.1	0.05	0.05
1	0.3	0.04	0.01
1	0.3	0.04	0.03
1	0.3	0.04	0.05
1	0.3	0.05	0.01
1	0.3	0.05	0.03
1	0.3	0.05	0.05



**Fig. 1.** a) Undoped lithium tetraborate produced by high temperature solid state synthesis. b) Lithium tetraborate doped by solid state doping method. c) Lithium tetraborate doped by solution assisted doping method.

were performed using Zeiss SUPRA 50 VP with has magnification between 12 and 900,000, variable pressure between 2 and 133 Pa, acceleration voltage of 0.1–30 kV.

## 2.5. TL response measurement

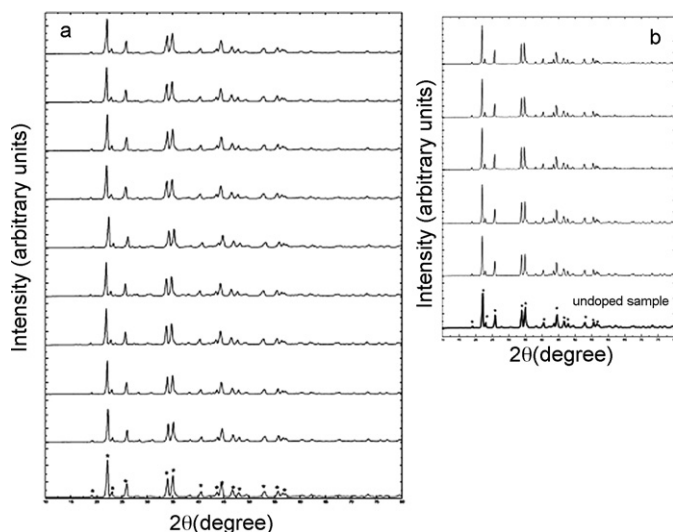
The dosimetric properties of the samples were determined by using Harshaw TLD Reader Model 3500. The heating rate was chosen as the lowest possible for the measurements to be most reliable. As a result, the heating rate of  $1^\circ\text{C/s}$  was used from  $50^\circ\text{C}$  to  $400^\circ\text{C}$ . The examined dosimeters were exposed to Beta, 90Sr–90Y radiations at room temperature for 5 min while the radiation given was  $0.5\text{ Gy/min}$ . The sample amount was 10 mg which is within the reliable range determined by Wall (1982).

## 3. Results and discussions

### 3.1. XRD analyses

The XRD patterns obtained for the material produced showed that production of lithium tetraborate was successful since all of the main peaks of lithium tetraborate coincide with those of the material produced. Most intense peaks of the XRD pattern of lithium tetraborate sample were assigned by the help of JCPDS card no: 18-0717 data of lithium tetraborate with tetragonal structure, and cell parameters  $a=9.477$ ,  $b=9.477$ , and  $c=10.286\text{ Å}$ . Lithium tetraborate belongs to I41cd space group (point group 4mm) [20]. Moreover, it was seen that the dopant added did not interfere with the crystal structure. Fig. 1 displays the XRD results for lithium tetraborate produced by high temperature solid state synthesis and doped by solid state doping and solution assisted doping respectively.

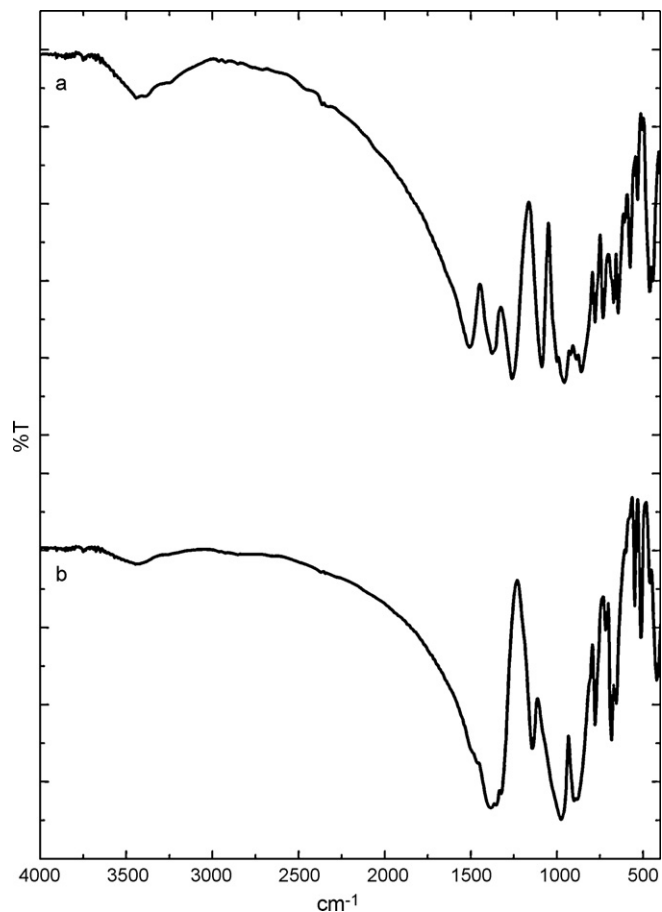
Likely, Fig. 2 belongs to the lithium tetraborate synthesized by water assisted production method and doped by solution assisted methods. As it can easily be detected addition of the activator did not have any significant effect on the crystal structure of lithium tetraborate produced.



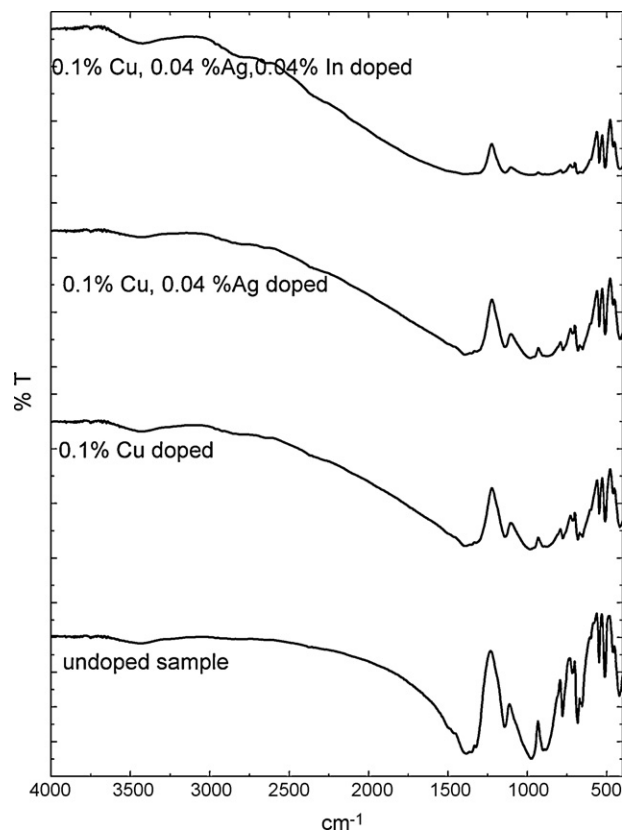
**Fig. 2.** a) Undoped lithium tetraborate produced by water assisted method. b) Lithium tetraborate solution assisted doping.

### 3.2. FTIR analyses

Fourier transform infrared spectroscopy (FTIR) was employed as an auxiliary characterization alternative. Fig. 3 displays the spectral resemblance between lithium tetraborates produced by solution assisted method and high temperature synthesis. The existence of characteristic B–O trigonals and tetragonals in the spectrum



**Fig. 3.** FTIR results for a) water assisted and b) high temperature solid state synthesized lithium tetraborate.



**Fig. 4.** Effect of doping on vibrational modes of lithium tetraborate.

confirmed the structure. An extended list of common vibrational modes expected in lithium tetraborate can be given as:

- 580 ( $\text{cm}^{-1}$ ): Borate deformation, plane bending of boron oxygen triangles.
- 900–865 ( $\text{cm}^{-1}$ ): Stretching vibrations of tetrahedral  $(\text{BO}_4)^{4-}$ .
- 1246–1807 ( $\text{cm}^{-1}$ ): Stretching vibration  $(\text{BO}_3)^{3-}$ .
- 1343–1248 ( $\text{cm}^{-1}$ ): Stretching vibrations of B–O of trigonal  $(\text{BO}_3)^{3-}$  units.

Fig. 4 displays FTIR outputs for the undoped material and lithium tetraborate doped with different amounts of different dopant combinations.

### 3.3. SEM results

Scanning electron microscopy was basically used to compare the morphologies of products of two different synthesis methods. Figs. 5 and 6 show the morphological status of lithium tetraborate produced by high temperature solid state synthesis and by water assisted method.

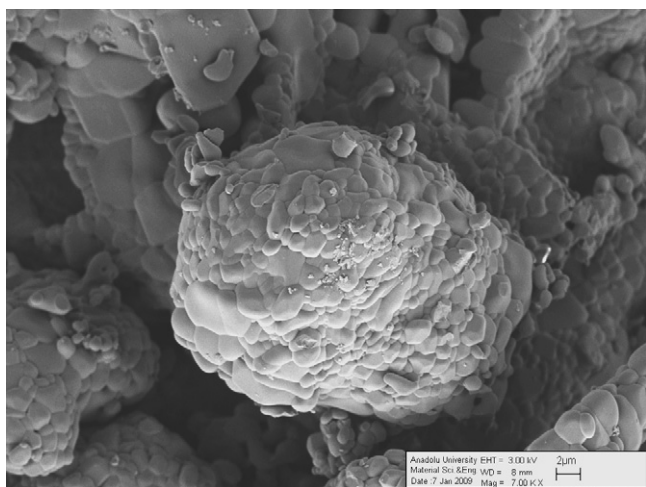
According to SEM images, the product of water/solution assisted synthesis display rather sticky characteristics. Fig. 5 shows an undulating particle surface while the image in Fig. 6 shows a smoother surface.

### 3.4. TL response results

#### 3.4.1. TL response results of single dopant case

The TL response graphs, i.e., glow curves are the major tools to decide on the TL dosimetry potential of a material. Hence the influence of production procedure on the TL response of lithium



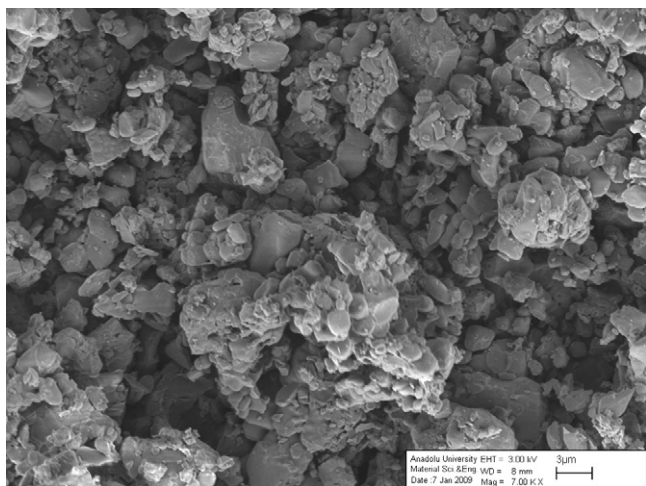


**Fig. 5.** Lithium tetraborate produced by high temperature solid state method-magnification = 7.00 K X.

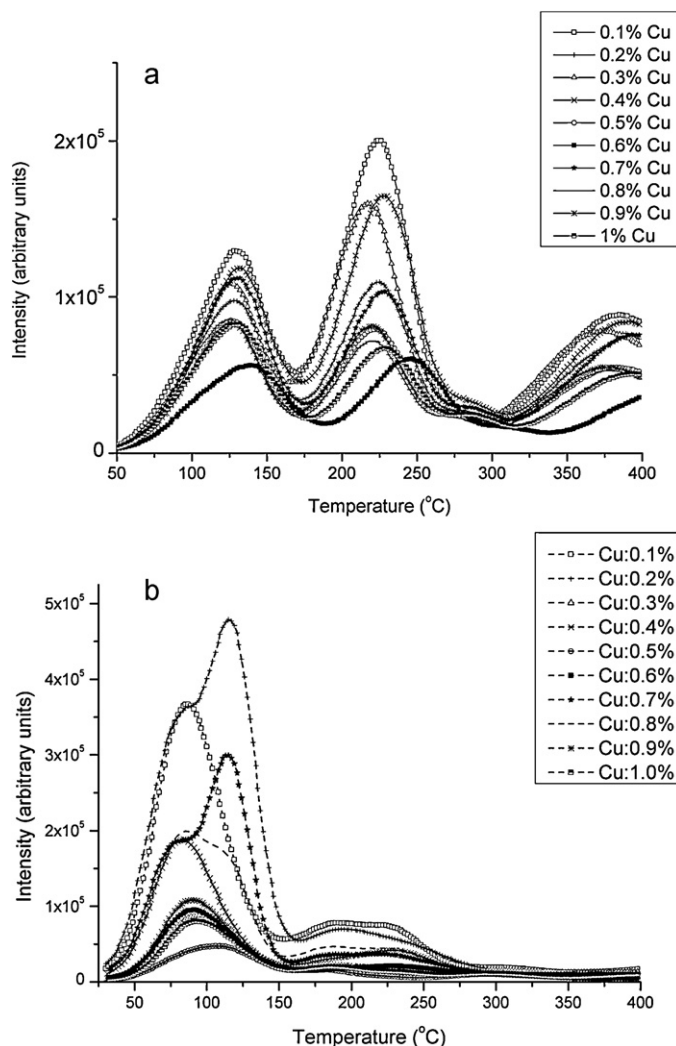
tetraborate was studied on these plots. Figs. 7–10 display the glow curve sets of different products for doped samples.

As Fig. 7 displays the glow peak intensity was inversely proportional to the amount of dopant added within the concentrations under investigation for solid state produced and solution assisted doped samples. Although there were two shoulder peaks, the major peak at 200 °C could be caught. The best result was obtained with 0.1% copper concentration with TL intensity around  $10^5$ . Hence, this quantity was adopted as the basis for double and triple doping experiments. However 0.3% copper was also experimented not to miss out any interaction possibility of major dopant and the co-dopant.

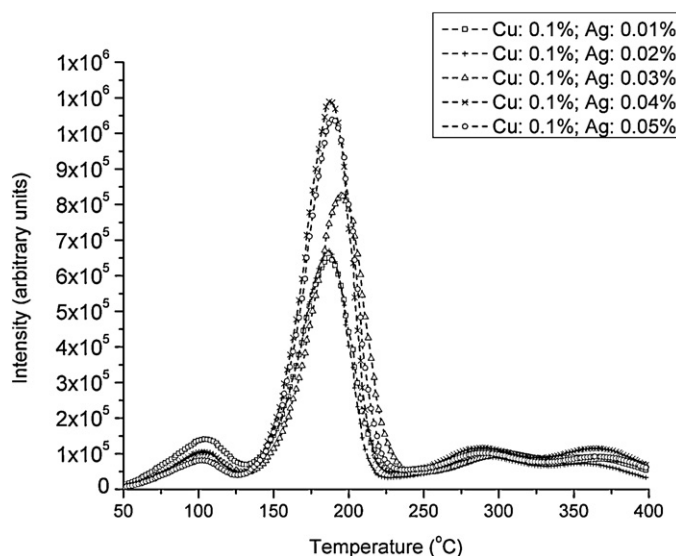
No noticeable trend could be detected in the glow curve set of samples produced and doped (0.1–1% Cu) by solid state method. On the other hand lithium tetraborate produced and doped by water/solution assisted method, in spite of giving more meaningful results than solid state synthesis and doping product, yielded broad low intensity peaks around 200 °C which is not a desired case. These products displayed higher intensity peaks around 100 °C which are widely known to be fading peaks. When the shoulder peaks around 200 °C are concerned it was observed that the highest peak was given by the sample including lowest quantity of copper dopant.



**Fig. 6.** Lithium tetraborate produced by water/solution assisted method-magnification = 7.00 K X.



**Fig. 7.** Glow patterns for the samples produced by a) solid state synthesis method and (0.1–1% Cu) doped by solution assisted method b) water assisted method doped by solution assisted method.



**Fig. 8.** Glow patterns for 0.1% Cu with varying amounts of Ag (0.01–0.05).

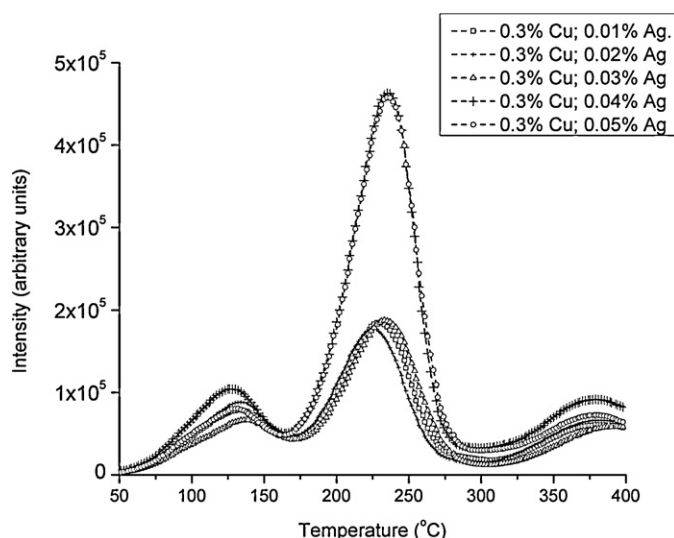


Fig. 9. Glow patterns for 0.3% Cu with varying amounts of Ag (0.01–0.05).

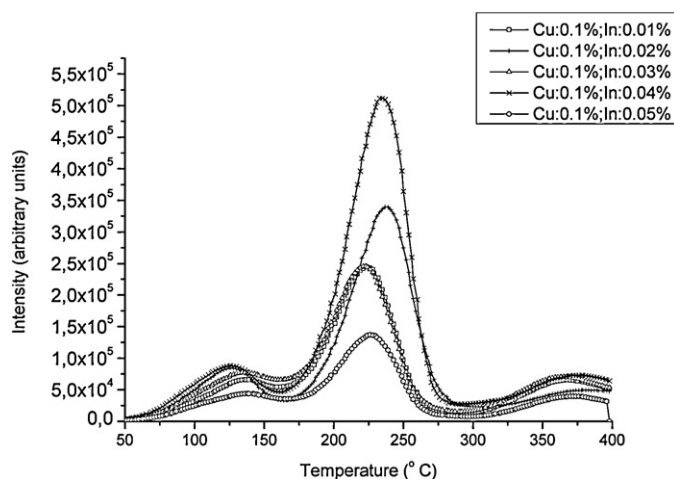


Fig. 10. Glow patterns for 0.1% Cu with varying amounts of In (0.01–0.05).

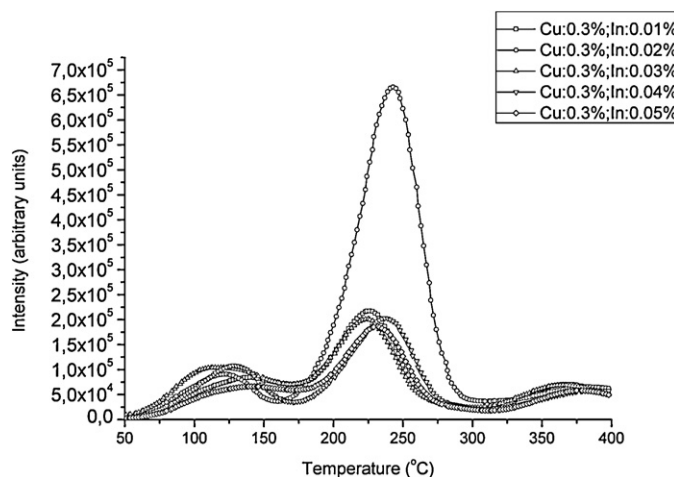


Fig. 11. Glow patterns for 0.3% Cu with varying amounts of In (0.01–0.05).

### 3.4.2. TL response results of double dopant case

In double doping experiments Cu–Ag couples (Figs. 8 and 9) gave better results compared to Cu–In couples (Figs. 10 and 11). In both

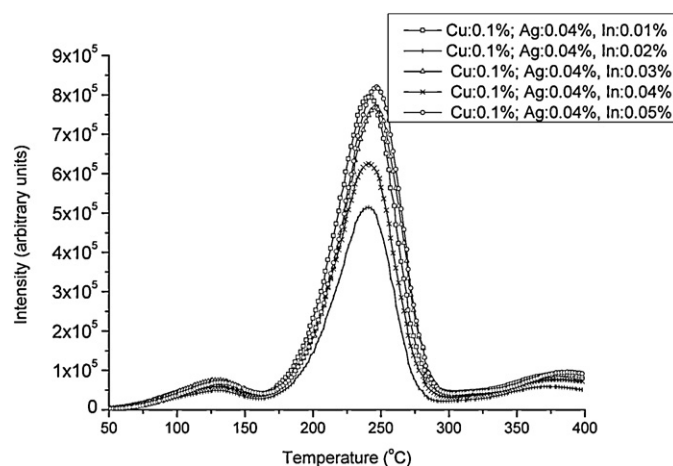


Fig. 12. Glow patterns for 0.1% Cu–0.04%Ag with varying amounts of In (0.01–0.05).

cases it can be concluded that increasing the copper content from 0.1% to 0.3% did not have a positive effect on TL response intensity in general.

It can be detected from Fig. 8 that the TL intensity increased with increasing concentration of co-dopant silver. It was obvious that addition of the co dopant exceeded the intensity reached with single dopant and the highest TL intensity obtained was in the order of 10<sup>6</sup> for 0.1% copper and 0.04% silver. Nevertheless the intensity decreased after 0.04% silver and when the copper concentration was increased to 0.3% to evaluate the interaction with the co- dopant, no better results were obtained since the TL intensity decreased down to the values of single doping case (Fig. 9). The interesting point in this second case was that within the group best results were obtained for 0.04 and 0.05% concentration of the co-dopant as in the case of 0.1% Cu.

Changing co-dopant from silver to indium did not yield better results. Moreover the trend caught for the case of silver co-dopant was also lost (Figs. 9 and 10).

Resultantly the best TL intensity was obtained for the sample doped with 0.1% Cu together with 0.04% Ag.

### 3.4.3. TL response results of triple dopant case

The triple doping experiments concentrated on the samples with 0.1% Cu with 0.04% Ag and 0.01% Cu with 0.05% Ag by adding the third co-dopant, i.e., In in differing proportions from 0.01 to 0.05% (Fig. 12). However to comply with the rest of the experiments same amounts of co-dopants accompanied increased amount of

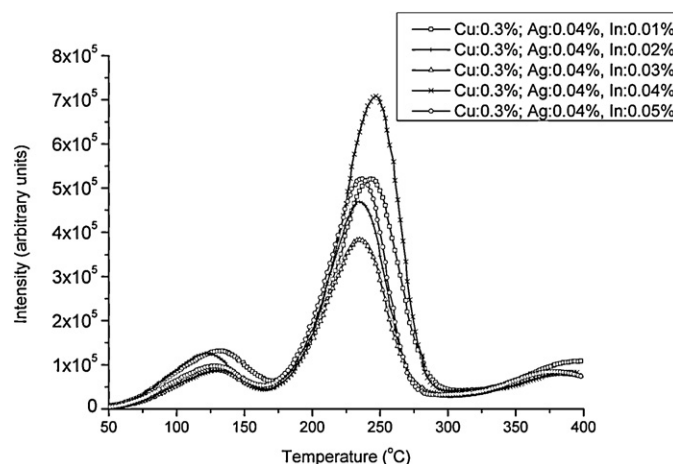


Fig. 13. Glow patterns for 0.3% Cu–0.04%Ag with varying amounts of In (0.01–0.05).

main activator (Cu) with 0.3% concentration to analyze the interaction (Fig. 13).

As it can be observed from Figs. 12 and 13 addition of third dopant did not have a positive effect on the TL intensity obtained.

#### 4. Conclusions

- 1 Lithium tetraborate was synthesized by high temperature solid state and water/solution assisted synthesis by exposing the reactants lithium carbonate and boric acid to a final heating at 750 °C for 4 h. The matrix material obtained was doped by different amounts and combinations of dopants: Cu, Ag, In. According to the XRD results it can be proposed that both water/solution assisted and solid state synthesis resulted in successful production of lithium tetraborate in powder form.
- 2 FT-IR spectrum revealed that addition of the activators Cu, Ag and In caused no noticeable change in vibrational modes of borates and the bond structure within the material.
- 3 The morphologies of the lithium tetraborates produced by high temperature solid state synthesis-doped by solution assisted doping and produced by water/solution assisted method-doped by solution assisted doping display different characteristics. The former one gave smoother surfaces with larger particles. This difference may be the root of difference in TL characteristics of the material obtained by these methods.
- 4 The TL results obtained showed that the material produced show TL response. Moreover it was realized that the lithium tetraborate produced by high temperature solid state synthesis yielded more meaningful results compared to those synthesized by water/solution assisted method. Besides, the glow curves of samples doped by solution assisted doping were more suitable for the potential use in TL dosimetry than the samples doped with high temperature solid state synthesis.

- 5 In the case where solely copper was added as dopant, sample with 0.1% Cu (lowest) dopant gave the best results among the concentration levels under investigation for both methods.
- 6 When the double doping is concerned, it was observed that co-dopant Ag increased the TL response in comparison to solely Cu doping experiments while In had no significant trend. Among the samples prepared with 0.1% Cu, 0.04% Ag coactivator gave the highest TL response.
- 7 In triple doping, addition of indium as the second co-dopant gave lower TL signals than copper, silver doped case.

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