



Thermal and structural properties of magnesium tetraborate produced by solid state synthesis and precipitation for use in thermoluminescent dosimetry

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ABSTRACT

Magnesium tetraborate is a phosphor with relevant characteristics for radiation dosimetry purposes. One of the main challenges on this material is still the understanding of its structure and luminescent processes involved in dosimetric performance. In this work, all the steps of the material production through DTA/TG, XRD and SEM techniques were evaluated to understand the main physical and chemical changes during the thermal process (calcination and sinterization) to produce the MgB_4O_7 samples. Two synthesis processes of MgB_4O_7 were studied: precipitation (P.S) and solid state (S.S). Finally, the luminescence efficiency of the pellets was investigated in function of different sintering temperatures. The initial stage of DTA/TG curves of the material just after the mixing of starting materials, regardless the synthesis, is marked by endothermic events caused by dehydration, decomposition of hydroxyl groups and hydrated borate phases. At higher temperatures, the curves are marked by exothermic events related to crystalline transitions, but the peak obtained for the S.S is better defined in comparison to P.S. Moreover, the DTA/TG analyses of the pellets prior and after the sinterization, showed that the process is more relevant for the MgB_4O_7 pellets produced by S.S (950 °C), dramatically reducing its hygroscopicity. The SEM images reinforce the importance of the sintering process to create a resistant detector and with a more homogenous and cohesive surface, therefore increasing its luminescence properties. The thermoluminescent (TL) sensitivity of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets produced via S.S, sintered at 950 °C, is 4 times higher compared to the ones sintered at 950 °C, via P.S. The materials produced via P.S did not present relevant changes in their TL sensitivity for sintering temperatures above 400 °C.

1. Introduction

Since 1980, borates are of great interest for thermoluminescent dosimetry (TLD), once they present better sensitivity (~ 10 times higher) compared to the commercial TLD-100 (LiF:Mg,Ti), when exposed to gamma and beta radiation (Prokic, 1986, 2007). Some borate-based materials also can detect thermal neutrons ($E < 0.25$ eV), when produced with enriched boron (boron-10) (Yukihara et al., 2017). MgB_4O_7 , for example, has an effective atomic number (Z_{eff}) for photoelectric absorption equal to 8.4, which is comparable to 7.4, for water and soft biological tissue, which is crucial for personal dosimetry (Prokic, 1986; Yukihara et al., 2017). Among the borates, the most useful for radiation dosimetry are barium borate (BaB_2O_4), lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$), lithium triborate (LiB_3O_5) and magnesium

tetraborate (MgB_4O_7) (Anishia et al., 2011; Pekpak et al., 2010; Souza et al., 2017). The last one has been produced with different rare earths dopants and alkali metals, such as Dysprosium, Terbium, Cerium, Samarium, Thulium and Sodium (Prokic, 1993, 2007; Annalakshmi et al., 2013; Kawashima et al., 2014) to improve its luminescent efficiency.

MgB_4O_7 has excellent dosimetric properties, such as linear dose responses over broad absorbed dose range (Pekpak et al., 2010; Souza et al., 2017); TL glow curves generally with a single peak; low fading, around 8% in three months for $\text{MgB}_4\text{O}_7:\text{Dy}$; and less than 5% in one month for $\text{MgB}_4\text{O}_7:\text{Ce,Li}$ (Souza et al., 2017). Furthermore, due to their high chemical stability (melting point above 1000 °C) and wide bandgap (9.5 eV), it is possible to produce a large variety of stable trapping and color centers in these matrices (Oliveira et al., 2016). The $\text{MgB}_4\text{O}_7:\text{Ce,Li}$ has recently been suggested as optically stimulated

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dosimeter (OSL) (Yukihara et al., 2013, 2014, 2017; Souza et al., 2015, 2017).

It is already known that the synthesis process may change the luminescent efficiency of a detector and consequently its dosimetric properties, recently, it was reported how two different routes, solid state synthesis and precipitation synthesis (wet reaction), affect the dosimetric properties (e.g. dose response) of the $\text{MgB}_4\text{O}_7\text{:Dy}$, upon beta irradiation (Souza et al., 2015, 2017).

Hereby, the present work aimed to evaluate this change in a different perspective and to a deeper understanding why the synthesis changes the final TL properties in the MgB_4O_7 matrices. With this purpose, solid state and precipitation syntheses (S.S and P.S, respectively) were used to prepare the tetraborate; it was analyzed from the initial stage, which is the mixing of starting reactants for the MgB_4O_7 production, to the final stage, consisting in the pellets' sinterization. To characterize these steps, differential thermal and thermogravimetry techniques (DTA/TG) were used. DTA/TG are important techniques for monitoring the chemical and physical changes of materials under increasing temperature. X-ray diffraction (XRD) was used to confirm the crystalline phase of the MgB_4O_7 matrices. The morphological characteristics of the pellets were examined through scanning electronic microscopy (SEM). Finally, the effect of different sintering temperature was checked in the final TL emission of $\text{MgB}_4\text{O}_7\text{:Dy}$ produced by both S.S and P.S.

2. Materials and methods

For the solid state synthesis, high temperatures are used to promote the formation of the desired phase of MgB_4O_7 . In the precipitation synthesis, the compounds are obtained using aqueous solutions and so lower calcination temperatures are needed (Souza et al., 2015; Yukihara et al., 2013, 2014; Chiang et al., 1997; Subanakov et al., 2014).

For both routes, the starting materials used were: MgO (Merck, 99.9% purity) and H_3BO_3 (Merck, 99.9% purity). For doped samples ($\text{MgB}_4\text{O}_7\text{:Dy}_{0.5\%}$), dysprosium oxide (Dy_2O_3) (Sigma-Aldrich, 99.9% purity) was utilized. The stoichiometric proportion of raw materials at S.S and P.S were the same: 87.21% of H_3BO_3 and 12.79% of MgO. For doped MgB_4O_7 , 0.5% of Dy_2O_3 was used. Calcination of the materials was carried out in an oven with air atmosphere.

For the P.S, the reactants and dopants, when it is the case, were diluted in nitric acid (HNO_3) with the main purpose of accelerating the formation of the MgB_4O_7 . At the end of the reaction, a pasty fluid material was achieved, to remove possible residual acidity; consecutive washings with distilled water were carried out in the materials and finally it remained in an oven for 24 h at 100 °C for drying. For the S.S, the starting materials were mixed and milled using a mortar and a pestle to obtain a homogenized mixture.

After the initial synthesis, part of the powder obtained was used to perform the DTA/TG analyses, in order to track the main physical and chemical transformations of the samples upon heating. The analyses were performed at SDT 2960 TA INSTRUMENTS equipment, from 25 °C up to 1000 °C, with a heating rate of 10 °C/min, and under dry synthetic air flow.

The other part of the powder was submitted to a calcination at 800 °C/2 h, for the P.S, and to 900 °C/7 h, for the S.S case. The calcination is required in order to achieve the desired crystalline phase of MgB_4O_7 ; the temperatures and the times needed for both syntheses are well defined in the literature (Subanakov et al., 2014; Souza et al., 2014). It is important to mention here, that although the P.S is a more cumbersome route, the reduced temperature and time required for the calcinations is a strong advantage compared to the S.S.

The crystalline phases of the MgB_4O_7 prepared by both syntheses were identified by powder X-ray diffraction in a Rigaku RINT 2000/PC diffractometer in the Bragg-Bretano geometry using $\text{CuK}\alpha$ radiation, with the tube operating at 40 kV/20 mA in the step scan mode in steps

of 2 θ (°) = 0.02, in the scan interval 2 θ from 10° to 80°, and 10 s per step.

The DTA/TG analysis of the samples, in pellet form, was also performed prior and after sinterization. Generally, the TLD or OSL detectors are used in pellets form (Prokic, 1986; Souza et al., 2015, 2017) for easy handling, and here the pellets were produced through cold compaction of calcined powder in a hydraulic press, with 100 kgf/cm². Each pellet had final dimensions of 3 mm of diameter and 1 mm of thickness. Afterwards, the MgB_4O_7 pellet was submitted to sinterization, which consists in a thermal treatment with different temperatures. As a default, the sinterization treatment for P.S and S.S were done at 850 °C and 950 °C, both for 2 h, respectively. To evaluate the effect of the sintering in the pellets surface the SEM images (FEG-SEM/JEOL-JSM- 7500F) were done, prior and after the pellets sinterization.

The effect of sintering in the TL emission of the $\text{MgB}_4\text{O}_7\text{:Dy}_{0.5\%}$ pellets was also checked. For that, the pellets were separated into batches (10 pellets per batch), each one submitted to different sintering temperatures, from 350 °C to 950 °C (S.S pellets) and from 350 °C to 850 °C (P.S. pellets). The pellets were kept 2 h at the respective temperatures and were cooled down slowly inside the oven to room temperature. Afterwards, the pellets were exposed to a beta source ($^{90}\text{Sr}/^{90}\text{Y}$) with 0.1 Gy/s of dose rate. TL measurements were carried out in a Risø TL/DA-20 reader, with pellets heated from 50 °C to 450 °C, with a heating rate of 10 °C/s, under N_2 flux.

3. Results and discussion

3.1. Thermal analyses and XRD diffraction

DTA/TG curves of the samples produced by S.S and P.S prior the calcination are shown in Fig. 1(a) and (b), respectively. The DTA (full line) represents chemical and physical changes in the samples structures during the temperature raising. The TG (dotted line) records changes in the samples mass along of this process.

The DTA/TG curves for P.S and S.S are quite similar between each other (Fig. 1 a and b), although the transitions seen along the temperature are better defined for the material produced via S.S. It is possible to divide the DTA/TG curves in four thermal stages and regardless the synthesis, the stages I and II are marked by strong endothermic events with an abrupt loss of mass. These stages are mainly associated with the decomposition of water molecules (dehydration step), hydroxyl groups (OH) and dissociation of hydrated borate phases, such as $\text{MgB}_6\text{O}_{10}\cdot 7(\text{H}_2\text{O})$, $\text{MgO} \cdot (\text{B}_2\text{O}_3)_3 \cdot 7(\text{H}_2\text{O})$ and $\text{Mg}_2(\text{B}_2\text{O}_5)(\text{H}_2\text{O})$, which explain the showed loss of mass.

The stage I of DTA/TG curve, ranged between 21 °C and 150 °C for the phosphorus obtained via S.S (Fig. 1 a) has an endothermic peak around 100 °C, and presented a loss of mass of 10%. At the stage II (150–300 °C) the endothermic peak (228 °C) is accompanied with a loss of 26% of mass. At the stage III (300–1000 °C) an exothermic event is observed, between 600 °C and 800 °C, peaked at 712 °C, without significant weight loss, approximately 1%. For the P.S case, at the first stage, an endothermic peak is seen at 115 °C, accompanied by a loss weight of 25% between 21 °C and 150 °C. In stage II there is an endothermic peak at 170 °C, with weight loss of 5% in the range of 152–265 °C. At the stage III an exothermic broad band is seen between 600 °C and 800 °C with another weight loss of 25%.

For both methods, stage III (Fig. 1a and b) is marked by exothermic events related to crystalline transitions (Brown, 2004), but the exothermic peak seen for the S.S [Fig. 1 (a)] is better defined in comparison to P.S [Fig. 1 (b)], where a broad band is seen, accompanied by 25% loss of mass, related to the release of residual nitric acid (NO_3) used in these syntheses. In stage IV, an initial exothermal process can be observed up to the end of the thermal cycle, but without significant weight loss, which can still be related to the crystallization process and the formation of MgB_4O_7 pure phase. Fig. 2 shows XRD graphs of the crystal produced by P.S., after calcination at 800 °C for 2 h, and the one

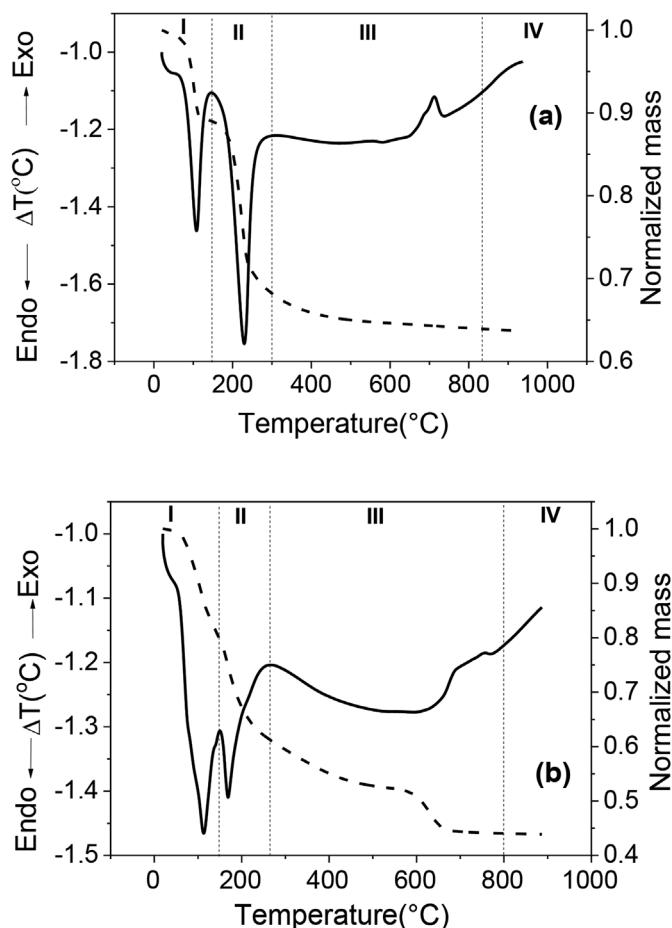


Fig. 1. DTA/TG curves after mixing reactants (MgO and H_3BO_3) in stoichiometric proportions through the (a) S.S and (b) P.S synthesis.

obtained by S.S at 900 °C for 7 h. The XRD results indicate that the crystalline phase of MgB_4O_7 is the dominating one for these calcination temperatures.

The DTA/TG analyses in the MgB_4O_7 pellets prior and after the sinterization are shown in Figs. 3 and 4, respectively. In Fig. 3 (a) is shown the thermal analyses of MgB_4O_7 produced by P.S, prior the sinterization at 850 °C for 2 h, where two weak exothermic broad bands are seen around 40 °C and 120 °C approximately. These bands are related to an adsorption process whereby water molecules adhere to the surface, due to the porosity presented by non-sintered pellets. After the sintering process [Fig. 3 (b)], the two small peaks are replaced by a broad exothermic band. The loss of mass of the pellet prior sinterization is 10% compared to 4% observed after the sinterization.

The sintering process has a higher relevance for MgB_4O_7 pellets obtained through the S.S. As can be seen in Fig. 4 (a), the DTA/TG curve of a non-sintered pellet shows two strong endothermic peaks at 114 °C and 214 °C, which is related to the occurrence of water molecules decomposition. An exothermic peak is seen at 539 °C, which can be related to the recrystallization process of the borates (Pekpak et al., 2010; Yukihiro et al., 2013). After the sintering process at 950 °C, the pellets did not present relevant thermal changes during the temperature interval of the DTA/TG. The loss of 16% of mass for these pellets without sinterization, and just after the sinterization the loss of mass is only 4%.

The DTA/TG curve of a sintered MgB_4O_7 pellet, at 950 °C for 2 h, is very similar to that obtained for a sintered MgB_4O_7 pellet prepared via precipitation [Fig. 3 (b)]; it is composed of one exothermic broad band around 100 °C and an endothermic broad band between 600 °C and 800 °C, also with insignificant loss of mass (3%).

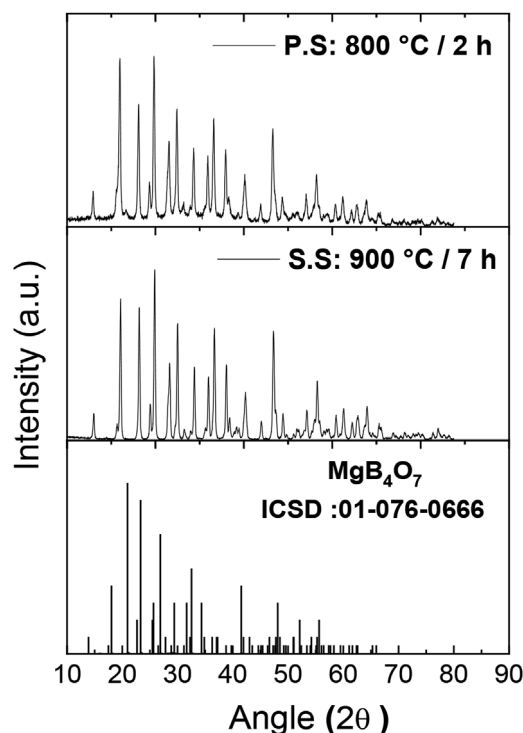


Fig. 2. Experimental XRD of MgB_4O_7 produced by the solid state and precipitation syntheses. The last XRD is the reference of MgB_4O_7 from the ICSD database (01-076-0666).

3.2. Scanning electronic microscope images

Through SEM images it was possible to see the surface of the MgB_4O_7 pellets before and after the sintering. In Figs. 5 and 6, it is seen that the sintering reduces the porosity of the pellets and thus improve their cohesion and surface homogeneity. For dosimetric purposes pellets must have a homogeneous surface and should be as cohesive as possible, to improve the optical and thermal conductivity properties, once that reproducibility of the luminescent signal is reduced by porous surfaces (Galmarini, 2011). In addition to increasing mechanical resistance of MgB_4O_7 pellet, the sintering process is a fundamental tool to reduce the sample hygroscopicity, and the DTA/TG results obtained before and after sintering process corroborate this affirmation [Figs. 3 and 4].

3.3. Sintering effect on TL sensitivity

The influence of different sintering temperature on the final thermoluminescent (TL) emission of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ is shown in Fig. 7. In the figure, each point in the graphs represents the average value of the areas below the TL peak at 240 °C exhibited by the pellets produced through S.S and that one at 225 °C by the samples obtained via P.S (Fig. 8).

In Fig. 7 it is clear that the TL sensitivity of the pellets produced by S.S is much higher compared to the one produced by P.S, for non-sintered pellets and for all sintering temperature range evaluated. As an example, the TL emission of non-sintered S.S pellets is 6 times higher than the one produced by P.S.

The sintering at 400 °C for 2 h increases the TL emission of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ produced by P.S, in an order of two [Fig. 7 (b)]. Considering the other sintering temperature (> 400 °C), the TL emissions are within the uncertainties (type A) range.

The $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets obtained through the S.S and sintered at 300 °C for 2 h showed a similar TL sensitivity to the non-sintered pellets. For higher temperatures, from 300 °C to 950 °C, an increase was

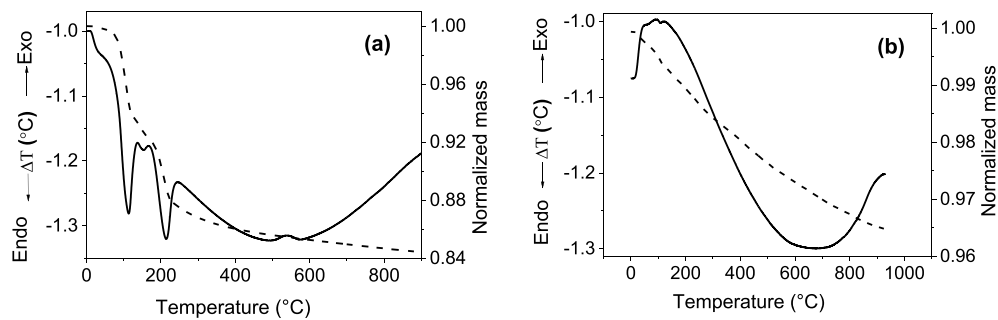


Fig. 3. (a) - DTA/TG curves of the MgB_4O_7 pellet prepared by P.S without sintering. (b) DTA/TG curve for the pellet after the sintering process at 850 °C for 2 h.

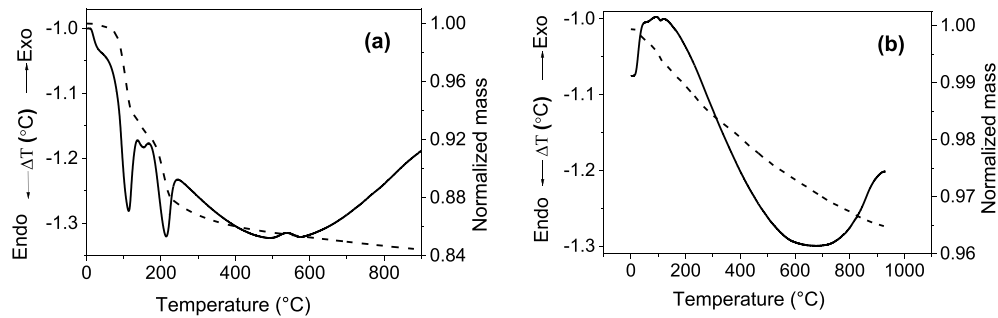


Fig. 4. (a) - DTA/TG curves of the MgB_4O_7 pellet prepared by S.S without sintering. (b) DTA/TG curve for the pellet after the sintering process at 950 °C for 2 h.

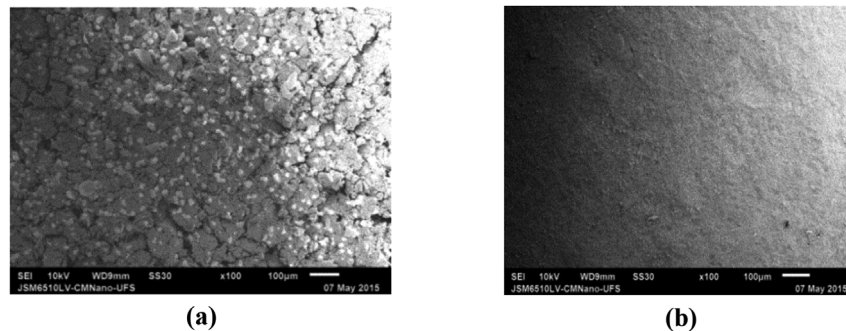


Fig. 5. Surface of a MgB_4O_7 pellet obtained via precipitation synthesis: (a) non-sintered, and (b) sintered at 800 °C for 2 h.

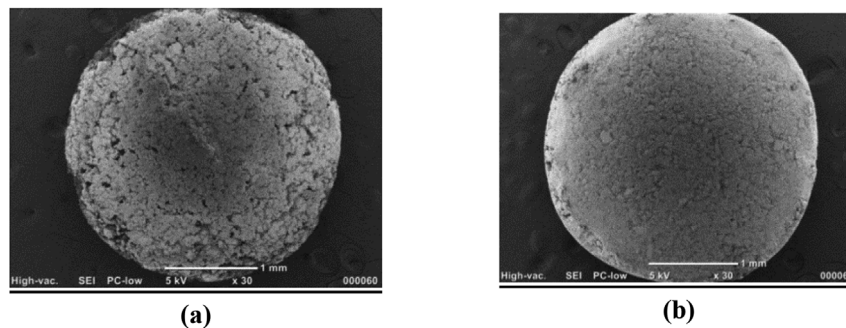


Fig. 6. Surface of a MgB_4O_7 pellet obtained via S.S synthesis: (a) non-sintered, and (b) sintered at 800 °C for 2 h.

observed in the TL efficiency by an order of 4. This behavior confirms how the sintering process is fundamental in the TL efficiency of the phosphorus, prepared by S.S.

The DTA/TG curves of non-sintered S.S MgB_4O_7 pellets [Fig. 3 (b)] exhibited endothermic events related to water release in their structure, due to its high porosity [Fig. 6 (a)]. During the TL reading, the presence of water molecules in the porous of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellet can easily absorb the luminescence emitted by the sample, creating a quenching effect and thus reducing its luminescence efficiency.

During the heating in the TL reading, the thermal conductivity among the pellets is also reduced with the presence of the porous: the transfer of heat through the pores is usually slow and inefficient, due to the presence of stagnant air and water (Yukihara et al., 2014; Somiya, 2013) and so this is another important factor which contributes to decrease the TL sensitivity of non-sintered $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets.

The sinterization process used in this work consisted in taking the tetraborate pellets to temperatures close to their fusion, which is approximately 1000 °C. High temperatures cause reduction of porosity,

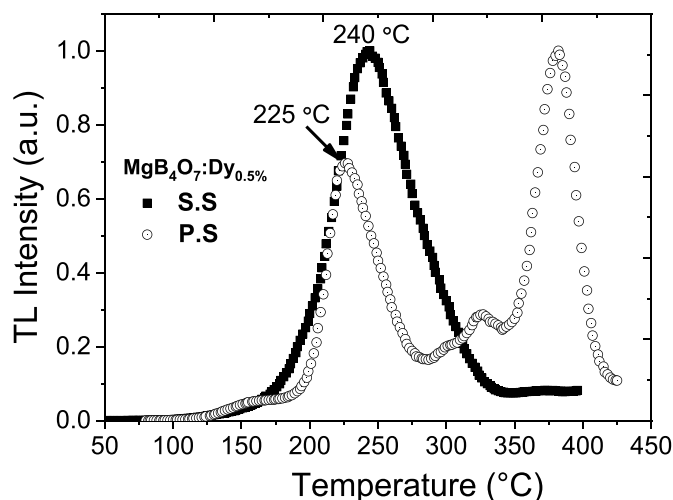


Fig. 7. (a) - TL emission of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets obtained through the S.S and P.S syntheses. (b) Enlarged image of the P.S graph. All the pellets were irradiated with 1 Gy ($^{90}\text{Sr}/^{90}\text{Y}$).

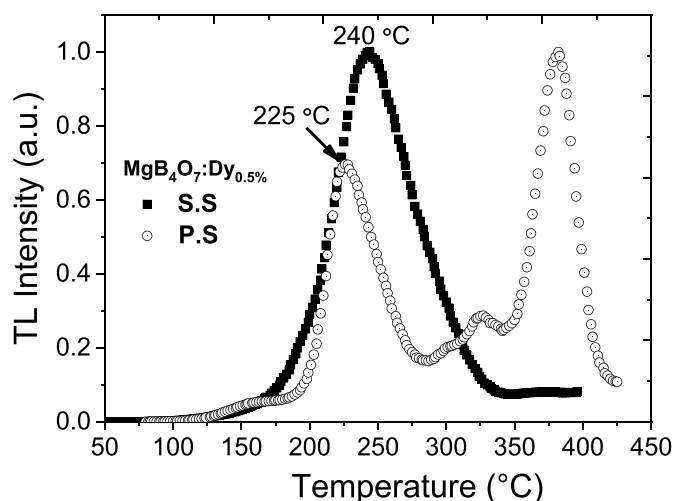


Fig. 8. TL emission from $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ produced via the S.S synthesis and the precipitation method, after irradiation of 1 Gy ($^{90}\text{Sr}/^{90}\text{Y}$).

improving the mechanical, thermal and optical properties of these pellets. Indeed, the sintering at higher temperatures causes better diffusion of the BO_3^{3-} and BO_4^{4-} vacancies at the MgB_4O_7 (Galmarini, 2011), which act as trapping centers in this lattice [21]. Thus, sintering directly imply in the increase of charge capture probability during the irradiation process, increasing the luminescence efficiency of $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets, especially for the material produced by S.S.

4. Conclusions

In this work the TL efficiency of tetraborate has been correlated with its synthesis steps, covering from the calcination process, were the borate is produced in its crystalline phase, to the sintering of the pellets. With the DTA/TG analyses of raw material, just after mixing, it was possible to identify the main physical and chemical events which occur with temperature raising. The graphs showed that the crystallization of the material prepared by P.S is accompanied by nitric acid liberation, causing the broad band seen within 600 °C and 800 °C (Fig. 1 (b)). The exothermic band at the S.S case is well defined and with a peak at 712 °C. It was also observed that the total loss of mass during the

calcination process is 55%, for the P.S, and 37% for the material produced via S.S.

The DTA/TG graphs of the non-sintered and sintered pellets emphasize the importance of the sintering process to improve the cohesion of the pellets and also to reduce the porosity in their surface. This process is more relevant for the S.S $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets in comparison to the ones produced by P.S. Such proprieties reflect directly in the TL efficiency of the pellets, and the SEM images corroborate this affirmation. Finally, it was observed that the TL sensitivity is improved with the increase of sintering temperatures. This effect was pronounced for the S.S $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ pellets, where an improving in the TL sensitivity from 350 °C to 950 °C was seen. Moreover, although the calcination temperature and time to prepare the MgB_4O_7 through P.S is much lower compared to the S.S, leading to less energy consumption, the technique is very cumbersome and the TL sensitivity of the detector is much lower compared to the TLDs produced by S.S (in an order of 6). This aspect reinforces the assumption presented by previous papers (Souza et al., 2014, 2015), that the S.S has more advantages than the P.S, when producing the $\text{MgB}_4\text{O}_7:\text{Dy}_{0.5\%}$ for dosimetric purposes.

Acknowledgments

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