Study of radioluminescence in $LiMgPO_4$ doped with Tb, B and Tm

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ABSTRACT

Lithium magnesium phosphate ($LiMgPO_4$, called LMP) crystals doped with different concentration of Tb, B and Tm were tested in regard to their radioluminescence (RL) emitted during irradiation. LMP crystals were grown as rods with a micro-pulling down method at IFJ PAN. The samples in the form of thin slices were found very sensitive to the ionizing radiation. Differently doped LMP samples were tested in a fiber optic measurement system (called PORTOS) which was constructed and adapted to the specific spectral properties of LMP. The LMP detector placed at the end of a 15 m fiber connected to the PORTOS device was tested with gamma rays emitted from Co-60 Theratron 780E as well as with a Cs-137 source in a Laboratory for Calibration of Radiation Protection Instruments at IFJ PAN. The RL signal was roughly proportional to the dose rates in the wide range of the dose rates. The highest RL signal was recorded for LMP crystals doped with 0.8 mol% of Tb, as well as those doped with 0.8 mol% of Tm. The radioluminescence signal is discussed in regard to the thermoluminescence (TL) glow curves and optically stimulated luminescence (OSL) decay curves for 14 differently doped LMP crystals.

Keywords: $LiMgPO_4$ (LMP), radioluminescence (RL), optically stimulated luminescence (OSL), thermoluminescence (TL),

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HIGHLIGHTS:

1. $LiMgPO_4$ emits strong radioluminescence during irradiation
2. Radioluminescence is proportional to the dose rate
3. Among all tested samples the highest RL was recorded for Tb doped LMP

1. INTRODUCTION

The growing presence of ionizing radiation in all areas of life requires development of various measurement methods to ensure maximum radiation safety. Aside from industrial, scientific and nuclear energy applications, ionizing radiation is increasingly present in medical diagnosis and cancer treatment. The adequate dose of radiation in each radiotherapy session is measured at the time of planning the therapy, within phantom trials and at the time of the actual irradiation through placement of a detector on the patient's body or even inside the body (in vivo). The verification of therapeutic dose can be done with a passive detector (read in a laboratory after the end of the radiotherapy session), but for obvious reasons, real-time measurement would be more effective.

A new real-time measurement method proposed several years ago was based on luminescence of crystalline aluminum oxide doped with carbon (Al_2O_3:C) located at the end of the long optical fiber connected to a photomultiplier for signal recording and a laser for detector stimulation [Marckmann, 2006]. The method uses radioluminescence (RL) and optically stimulated luminescence (OSL). These two phenomena are associated with emission of light that accompanies transitions between energy levels in a crystal. Both phenomena are different because RL occurs spontaneously immediately upon exposure to ionizing radiation and is dependent on the dose rate, while OSL requires stimulation with light and might occur a long time after exposure thereby giving information about the absorbed dose. The fundamental requirements for detectors used for this purpose are a very high sensitivity to radiation and a small size, so that its presence does not interfere with the field of radiation or, if required, it can be placed inside the body.

The research on a real-time dosimeter system has been concentrated on commercially available Al_2O_3:C [Yukihara et al., 2015; Andersen et al., 2010; Andersen et al. 2011, Buranurak and Andersen, 2016] and BeO [Bulur, 2014] materials used as OSL detectors. One of the most promising new materials is $LiMgPO_4$. Several groups have been working on the development of $LiMgPO_4$, usually doped with Tb and B and produced in a form of powder [Zhang et al., 2010; Kumar et al., 2011; Dhabekar et al., 2011; Menon et al., 2012; Bajaj et al., 2016]. Recently, investigations have also been performed on crystals grown from LMP powder by micro-pulling down (MPD) method [Wróbel et al. (2014); Kulig (Wróbel) et al., 2016a; Kulig (Wróbel) et al., 2016b; Kulig et al., 2017; Marczewska et al., 2016; Gieszczyk et al., 2018] or thin foils [Menon et al., 2018; Malthez et al., 2018]. LMP shows a very high sensitivity to ionizing radiation, good repeatability of the OSL signal and linear dose response but also relatively high spontaneous fading in time after exposure. The fading can cause even 20–40% loss of the signal in the first two weeks, which is an obvious obstacle to its use in radiation protection. On the other hand, high sensitivity to the ionizing radiation even at high fading favors LMP for application in real-time dosimetry.

Our preliminary experiments on LMP doped with Tb, Tm and B showed that LMP crystals doped with Tm or Tb emit the strong radioluminescence during irradiation and this is highly dependent on the concentration of dopants [Marczewska et al., 2019]. Therefore, the aim of the present work was the more systematic study of the radioluminescence in LMPs differently doped with Tb; Tb+B; Tm; Tm+B; Tb+Tm and Tb+Tm+B in connection with their OSL and TL signal, as well as the investigation of dose response for selected crystals irradiated in a wide range of dose rates with Cs-137 and Co-60 sources.

2. MATERIAL AND METHODS

LMP crystals were grown by a micro-pulling down method. The preparation of raw material as well as the process of the crystals growth in the Cyberstar device are fully described in the paper of Gieszczyk et al. [Gieszczyk et al, 2019]. As the starting feedstock for production of the crystals, LMP powder prepared according to the standard procedure of solid state reaction in air was used. Lithium hydroxide (LiOH), hexahydrate magnesium nitrate ($Mg(NO_3)_2.6H_2O$) and ammonium dihydrogen phosphate ($NH_4H_2PO_4$) were used as the substrates. The solid state chemical reaction was interrupted by several annealing cycles at temperatures ranging from 200 to 750 ^oC and mixing of the reaction products. Boric acid (H_3BO_3) or borax ($Na_2B_4O_7.10H_2O$) was used for doping the phosphors with boron (B) ions. Tb_4O_7 and Tm_2O_3 oxides were used for doping the phosphors with terbium and thulium ions, respectively. The chemical composition and concentration of dopants are given in Table 1.

Table 1. The chemical composition and concentration of dopants of the LMP crystals

Type and concentration of dopants in investigated $LiMgPO_4$ [mol%]		
Tb	B	Tm
0.5	-	-
-	-	0.5
0.8	-	-
-	-	0.8
1.2	-	-
-	-	1.2
0.5	1	-
-	1	0.5
-	10	0.8
0.2	-	0.6
0.2	10	0.6
0.8	10	-
0.8	1	-
0.8	0.5	-

The crystals were grown in the MPD laboratory at IFJ PAN in Krakow. The growth process was conducted in the graphite crucible which was additionally covered by an Mo overlay in order to improve the heating properties of the applied thermal setup. After melting the raw powder the pulling of the crystals was performed through a hole in the bottom of the crucible. The crystals had the form of rods with a 3 mm diameter and they were up to 60 mm in length. The crystals were cut by means of a diamond wire cutting saw into slices 1mm thick. The picture of an LMP crystal and a single sample lying on a Risø cup is shown in Fig. 1.

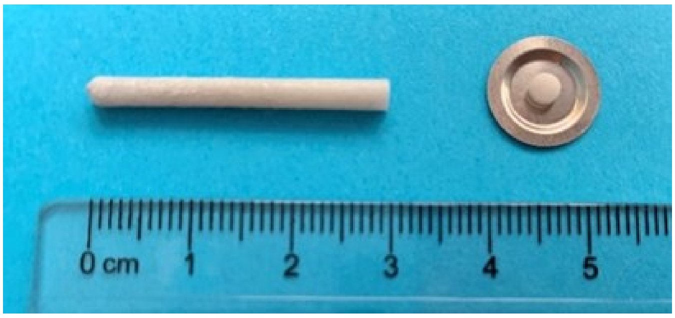

Fig. 1 Part of the LMP crystal rod obtained by MPD method and a slice of this crystal lying on a Risø cup.

The standard TL and OSL measurements were performed in an automatic Risø-TL/OSL-DA20 reader with a blue LED stimulation module (470 nm), photomultiplier tube (EMI 9235QB) and Sr-90/Y-90 beta source used for irradiation. A detailed specification of the reader was recently described by Bilski et al. [Bilski et al. (2014)] and Wróbel et al. [Wrobel et al. (2015)]. TL and OSL measurements were carried out by using Hoya U-340 filter. LMP samples were annealed by heating them up to 500 ^{o}C with a constant heating rate of 5 ^{o}C/s followed by bleaching by the OSL measurement with blue LED stimulation (90% power) during 600 s in the Risø-TL/OSL-DA20 reader before usage. Standard TL readouts of the irradiated LMP crystals were carried out at the constant heating rate of 2 ^{o}C/s from room temperature (RT) up to 500 ^{o}C, while standard OSL measurements were performed at RT, with 90% power blue LED stimulation during 600 s. TL and OSL signals were normalized to the weight of the LMP crystals.

The measurements of the RL signal were conducted in a newly constructed optical system called PORTOS (**port**able RL/**OS**L) which is a small portable remote device. Its scheme is presented in Fig. 2. The measuring device consists of optical filters situated in the panels between a Hamamatsu H10682-210 photomultiplier and a 15 m quartz optical fiber at the end of which was placed a detector in the form of a slice of a crystal (diameter of 3mm, thickness of 1mm). The PORTOS system also possesses a dichroic mirror, intended for OSL measurement in the future, and for changing the direction of the laser beam which can be easily attached to the system. The set of optical filters was selected on the basis of previously performed spectral investigations of the RL light emitted by Tb or Tm doped LMP crystals [Sas-Bieniarz et al., 2019]. The filters were selected so that the following transitions in Tb: $5D_3 \rightarrow 7F_6$ at the wavelength of 381nm, $5D_3 \rightarrow 7F_5$ (418 nm), $5D_3 \rightarrow 7F_4$ (440 nm), $5D_3 \rightarrow 7F_3$ (460 nm) and $5D_4 \rightarrow 7F_6$ (486 nm) as well as in Tm ($1D_2 \rightarrow 3F_4$ at 460nm) were included in the measuring window. Finally, a short pass filter transmitting wavelengths between 250÷475 nm (Edmunds Optics) and two 405/150 nm (transmission between 330÷480 nm) BrightLine single-band bandpass filters (Semrock) were applied. In the optical setup there is also a place for a notch filter for rejecting the laser light, when the device is used for OSL measurements. The PORTOS system is controlled by software enabling the signal to be recorded with the sampling time between 1μs and several s and for the measurement duration from ms up to several hours. In the present measurements the sampling time was always 0.5s.

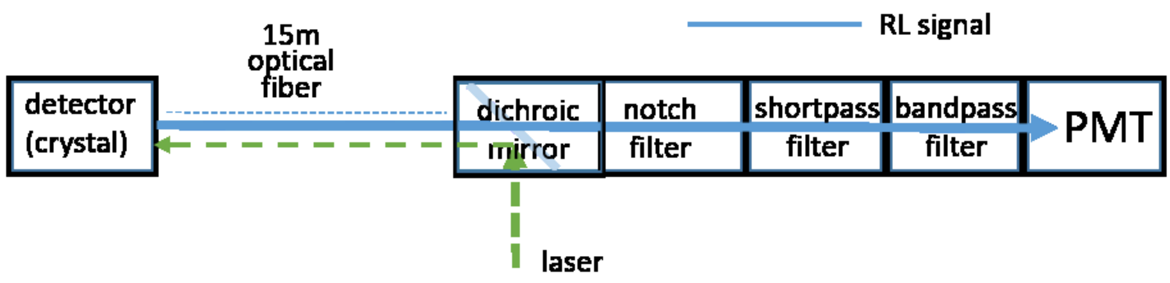

Fig. 2 Scheme of a measuring device named PORTOS. The place where the laser can be connected to the system is marked in green.

RL of LMP crystals were tested with two devices situated in IFJ PAN: a teletherapy Co-60 machine (Theratron 780E) and a Cs-137 source in the accredited Laboratory for Calibration of Radiation Protection Instruments. The Co-60 Theratron provides dose rates of 0.857 Gy/min, 1.167 Gy/min, 1.485 Gy/min, 1.934 Gy/min, 2.658 Gy/min at distances from the Co-60 source of 105 cm, 90 cm, 80 cm, 70 and 60 cm, respectively. During Co-60 irradiations the ending of the optical fiber with the LMP detector was placed in the defined position between two PMMA plates. The Cs-137 source delivers dose rates of 30 mGy/h, 49 mGy/h, 94 mGy/h, 354 mGy/h and 683 mGy/h at distances from the source of 250 cm, 210 cm, 170 cm, 120 cm and 105 cm, respectively. Before each irradiation the bare fiber (without a detector) was placed in the same position as intended for the subsequent exposures of LMP crystals in order to record the signal generated by radiation in the fiber (called 'stem'). Background signals of the LMP detector placed in the ending of the fiber and additionally fixed by a thermal shrinkable cover were also recorded, although they were very low and amounted only to several impulses. During Cs-137 irradiation the ending of the optical fiber was fixed on a movable table and the background without a detector as well as the stem signal of the irradiated fiber in each fixed position were also measured.

For analysis of RL linearity the signal was calculated as the sum of the RL signal of the last 20 channels (0.5 s each) before the end of irradiation minus the sum of the stem signals recorded in the similar manner.

3. RESULTS AND DISSCCUSION

The most common admixtures of LMP reported in the literature are Tb, Tm and B. According to the opinion of many authors [Menon et al., 2018; Tang et al., 2019], the concentrations of Tb, Tm and B strongly affect TL and OSL signals, but systematic quantitative investigations of RL signals have not been conducted so far.

In the present experiment the crystals were intentionally doped with the Tb or Tm whose concentration was in the range of 0.5 mol% and 1.2 mol%, wherein the concentration of B varied between 0 and 10 mol%. For clarity, the results of the RL measurements are divided into 3 groups concerning Tb, Tm and Tb+Tm common doping. To determine the effect of the specific doping, the RL signal of all the crystals which were investigated was discussed together with OSL decay curves and TL glow curves. It is known from observation that TL and OSL signals can vary from sample to sample due to the inhomogeneous presence of dopants on the crystal length. To avoid such situation all investigations were performed on the same samples, in the following sequence: annealing/bleaching –TL investigation – annealing/bleaching – OSL investigation – RL at one dose rate (Co-60) – annealing/bleaching – dose rates dependence (Co-60) – annealing/bleaching – dose rate dependence (Cs-137).

3.1 OSL decay curves and TL glow curves

At the beginning of the investigation OSL decay curves were recorded for all the crystals. The crystals with Tb and B doping showed a significantly higher OSL signal (Fig. 3 a), especially LMP doped with 0.5 mol% of Tb and 1.0 mol% of B. The OSL signal is heavily dependent on B concentration and changing B content results in the change of several times of the initial value of OSL signal. Tb- doping is more effective than Tm in the case of the OSL signal. Double doped with Tb and Tm LMPs present a relatively low OSL signal due to reduced content of Tb.

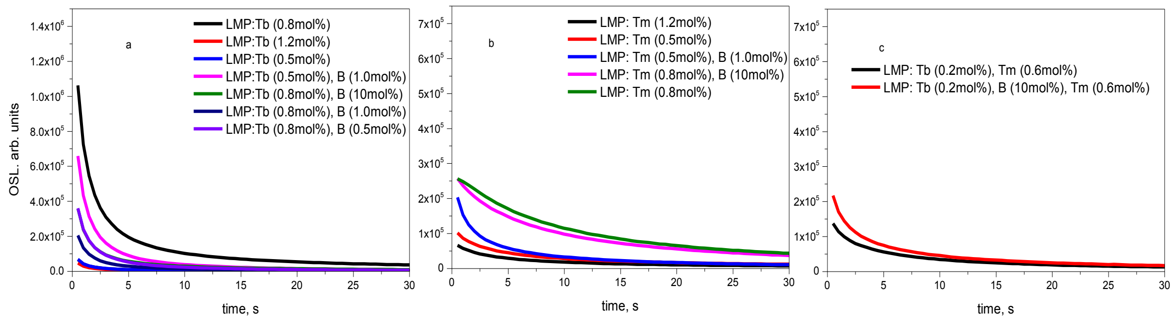

Fig. 3 OSL decay curves of crystals doped with Tb or Tb+B (a), with Tm or Tm +B (b) and Tb+Tm with and without B (c), recorded in Risø reader after irradiation with 200mGy of beta rays.

TL glow curves were also analyzed for all the LMP crystals under investigation (Fig. 4). The glow curves consist generally of 3 peaks; a low temperature peak at about 125 ^{o}C, at 250 ^{o}C (one exception is LMP with 0.8 mol%, which exhibits a peak at 300 ^{o}C) and a high temperature peak at about 400 ^{o}C. However, all of these peaks have low amplitudes. The presences of low temperature peaks and the relatively low amplitude of higher temperature peaks exclude LMP:Tb from being used as TL detectors. The presence of B is not as significant as in the case of OSL signals.

Looking at the TL glow curves for LMP doped with Tm or Tm+B, the tendency is as follows: LMP doped with 0.8 mol% Tm presents a higher OSL signal irrespective of the presence of B (Fig. 3b). The smaller and higher amounts of Tm cause the lower OSL signals. The significantly highest TL amplitude was noticed for 0.8 mol% Tm doped LMPs (Fig. 4b).

LMP:Tb,Tm crystals have a low OSL signal, similar to Tm doped crystals but the amplitude of TL peaks at about 300 ^{o}C is the highest (over 40 times higher than that of 0.8 mol% Tm).

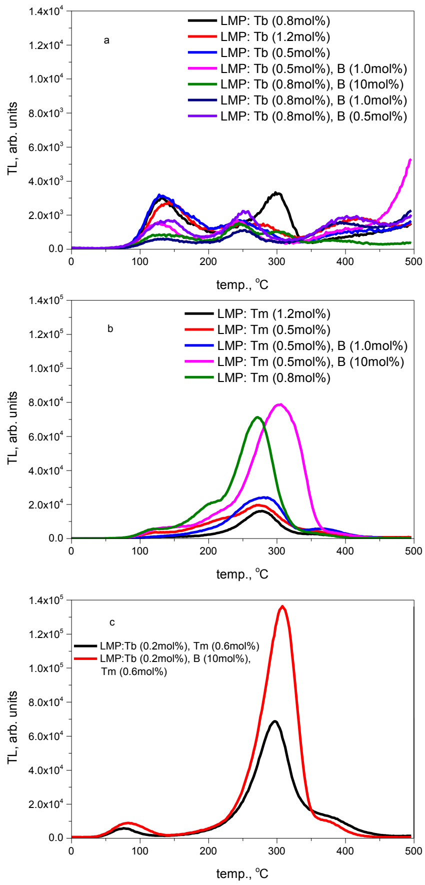

Fig. 4 TL glow curves for crystals doped with Tb or Tb and B (a), with Tm or Tm and B (b), and Tb, Tm without B and with B (c), recorded in Risø reader after irradiation with beta rays (200 mGy) and a 5 minute pause (to avoid the recording of after-glow emission).

Summarizing this part of the experiments, definitely the highest OSL was noticed for Tb+B doped crystals. However, Tm+B doped LMPs showed TL signal even 20 times higher and LMPs double doped with Tb+Tm about 40 times higher than TL measured for Tb doped LMP.

3.2 RL signal

LMP doped with Tb

The RL signals of Tb doped LMP crystals under gamma irradiation by the Co-60 Theratron were recorded in the PORTOS device. All crystals were tested at the same position at the distance of 80 cm from the source and at the same dose rate of 1.485 Gy/min. Crystals were coupled to the optical fiber which was always fixed in the same position. A stem signal of the optical fiber without any detector recorded in this position amounted to 16×10^3 impulses and as a background was subtracted during calculation.

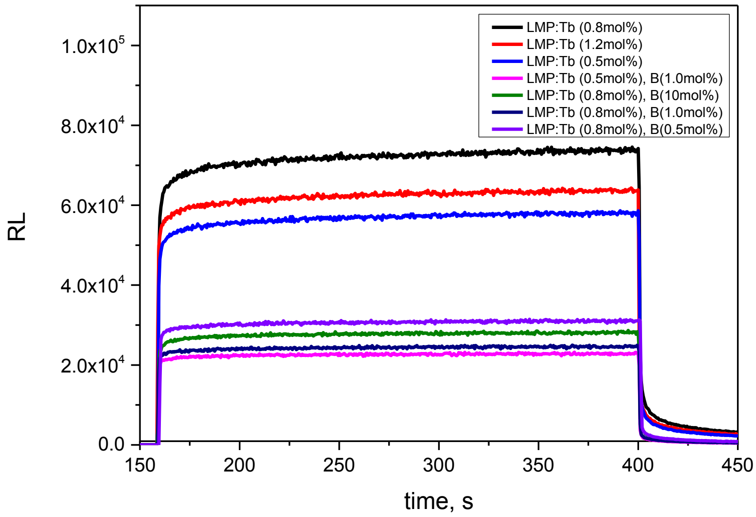

Fig. 5 RL signals recorded in PORTOS device at the dose rate of 1.485 Gy/min of gamma rays from Co-60 Theratron 780E apparatus for Tb doped LMPs.

In Fig. 5 it can be seen that the RL signals are higher for LMP crystals doped only with Tb than for those doped with Tb and B, with the highest signal observed in LMP doped with 0.8 mol% of Tb. The addition of B atoms reduces the emission of RL signal by 3 times in comparison with single Tb doping. The highest RL signal in the group of Tb and B doped crystals was emitted by LMP doped with 0.8 mol% of Tb and 0.5 mol% of B. The curves are slightly rising. The decrease of the RL signal after switching off the radiation is also worth mentioning. The RL signal of crystals doped only with Tb decreases rapidly to the level of 2000-4000 impulses and stays longer at this level which indicates the presence of after-glow emission. The RL signal of crystals doped additionally with B fades quickly to background level.

LMP doped with Tm

Similar investigations were performed for LMP crystals doped with Tm or Tm+B atoms. Figure 6 presents RL signals recorded in PORTOS device tested in the Co-60 radiation field, at the same distance from the source (80 cm, dose rate of 1.485 Gy/min). In this case the highest RL signal was achieved for LMP doped with 0.8 mol% of Tm, similar RL signals were obtained for LMP: Tm (1.2 mol%) and LMP:Tm (0.8 mol%), B (10 mol%). It seems that a smaller concentration of Tm (LMP:Tm (0.5 mol%) and LMP:Tm (0.5 mol%), B (1.0 mol%)) causes the lower RL signal, independently of the presence of B. The RL signal also has a slightly rising tendency.

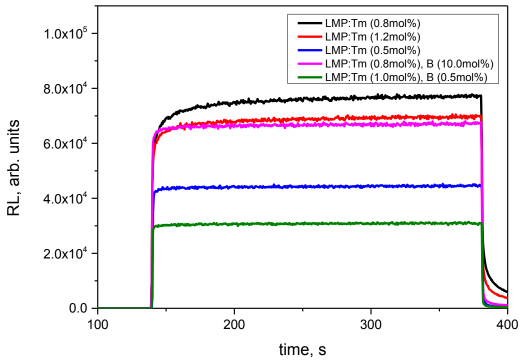

Fig. 6 RL signals recorded in PORTOS device at the dose rate of 1.485 Gy/min of gamma rays from Co-60 Theratron 780E apparatus for Tm and Tm, B doped LMPs.

LMP with Tb+Tm

The RL signal of double doped LMPs with Tb+Tm crystals is noticeably lower than that of crystals investigated previously, whereas the sample additionally doped with 10 mol% B showed even lower RL signal (Fig. 7).

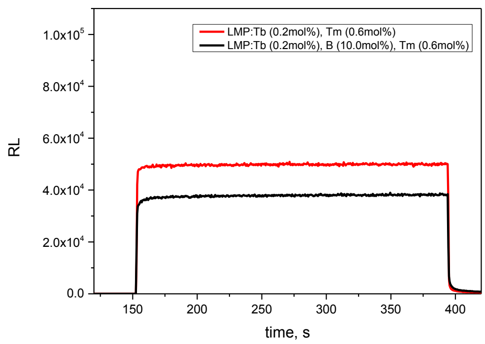

Fig. 7 RL signals recorded in PORTOS device at the dose rate of 1.485 Gy/min of gamma rays from Co-60 Theratron 780E apparatus.

Looking at RL signal it can be pointed out that among all the crystals investigated, the high RL signal was obtained both for LMP crystals doped only with Tb as well as Tm or Tm+B doped crystals. In the case of Tb doped crystals it was observed that the addition of even small amounts of B (0.5 mol%) causes the RL signal drop. If we consider Tm-doped crystals, another relationship can be seen – the RL signal is higher for higher Tm concentration (0.8 mol% or 1.2 mol%), independent of the presence of B. Crystals double doped with Tb and Tm presented a relatively lower RL signal whose reason could be the low concentration of Tb (0.2 mol%) and Tm (0.6 mol%).

Analyzing Figures 5, 6 and 7, RL signals under irradiation rise relatively quickly, reaching a “steady state” of slight increase. After stopping the irradiation the decrease of the RL signal is fast, but not in all cases does it reach pre-irradiation level. To compare curves obtained during 2- minute irradiation for all the crystals in a more quantitative way, three parameters were analyzed: rise time to reach 95% of the maximal RL signal, which was calculated as an average value of 10 last measurement points (i), signal increase measured for 200 s before interruption of irradiation (ii) and signal fall within 5 s after interruption of irradiation (iii) (see fig. 8).

The parameters shown in Table 2 indicate that in all cases the addition of boron causes faster stabilization of the RL signal, smaller increase of the RL signal during irradiation and faster fall of the RL signal after stopping irradiation.

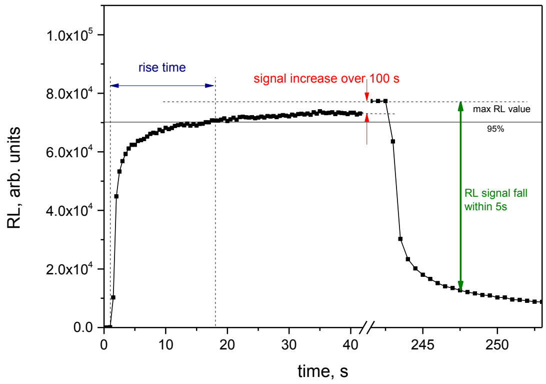

Fig. 8 Schematic illustration of the analyzed parameters (presented in Table 2).

Table 2 Three parameters: rise time to reach 95% of the maximal RL signal, signal increase expressed as % per 100 s and signal fall within 5 s after interruption of irradiation; for all investigated crystals.

Type and concentration of dopants in investigated $LiMgPO_4$ [mol%]			Parameters		
			rise time to reach 95% of the max RL	signal increase	signal fall within 5 s
Tb	B	Tm	[s]	[%/100 s]	[%]
0.8	-	-	35	2.5	88
1.2	-	-	35	2.5	89
0.5	-	-	3	2.0	89
0.5	1.0	-	10	1.5	94
0.8	10.0	-	15	1.5	91
0.8	1.0	-	10	1.5	94
0.8	0.5	-	15	1.5	91
-	-	0.8	35	2.5	63
-	-	1.2	25	2.0	88
-	-	0.5	3	1.0	98
-	10.0	0.8	5	1.0	96
-	0.5	1.0	2	1.0	97
0.2	-	0.6	2	0.5	98
0.2	10.0	0.6	5	1.0	95

3.2 RL as a function of dose rates

In the second step of the experiment three crystals were selected for the dose rates dependence investigations: LMP: 0.8 mol% of Tm, 10 mol% of B; LMP: 0.8 mol% of Tb and LMP: 0.5 mol% of Tm. These samples were chosen because of their high RL signal (the first two of them) or the almost immediate achievement of the constant RL value and fast fall to the background level. The samples were tested in the PORTOS device at Co-60 and Cs-137irradiations.

High dose-rates (Co-60, Theratron 780E)

RL signals obtained for the LMP crystals at different distances (dose rates) from the source are shown in Fig. 9. The RL signals of all crystals have slight upward trends. Fig. 10 presents the stem signal recorded for the optical fiber during irradiation at all distances from the source. For each distance, the sum of the 20 points (for 2 s) of the stem as a background signal was subtracted from the signal recorded before interruption of irradiation also 2 s. To check the linearity of the dose response, the linearity coefficient, calculated as the ratio of the signal to the dose related to the signal to the dose for small dose, was determined (Fig. 11). As can be seen, the RL signal is linear in respect to the dose rates over the range from 0.857 Gy/min to 2.658 Gy/min (51430.2 mGy/h to 159465.6 mGy/h).

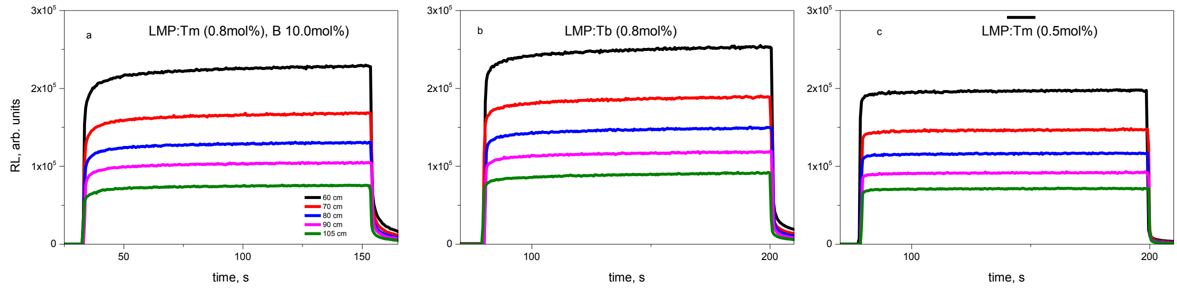

Fig. 9 RL signal of 0.8 mol% Tm and 10 mol% B doped LMP (a); 0.8 mol% Tb doped LMP (b) and 0.5 mol% Tm doped LMP (c) at different distances from the Co-60 source (Theratron 780E).

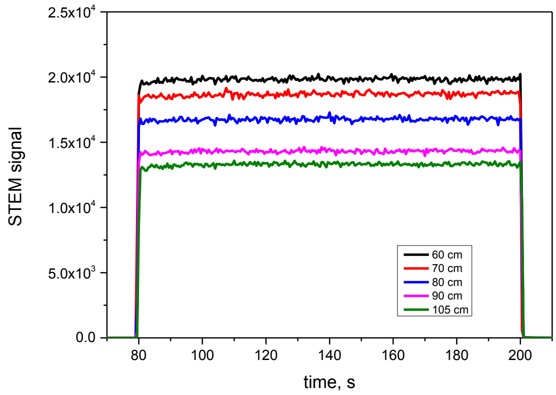

Fig. 10 Stem signal of quartz optical fiber irradiated at different distances from C-60 source (Theratron 780E).

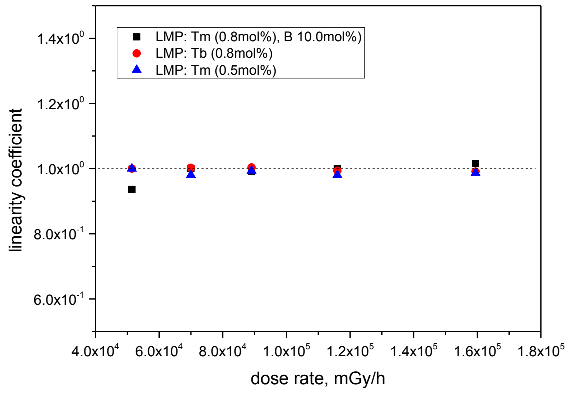

Fig. 11 Linearity coefficient for LMP:Tm (0.8 mol%), B (10.0 mol%); LMP:Tb (0.8 mol%) and LMP:Tm (0.5 mol%) calculated for RL signal measured at different dose rates of Co-60 source (Theratron 780E).

Low dose-rates (Cs-137 irradiator)

A similar investigation was performed for three selected crystals, e.g. LMP:Tm (0.8 mol%), B (10.0 mol%); LMP:Tb (0.8 mol%) and LMP:Tm (0.5 mol%) at the Cs-137 source in dose rates range from 30 mGy/h to 683 mGy/h. The crystals were placed in the PORTOS device and before each irradiation the stem signal was also recorded at all distances. Fig. 12 presents RL signals at two distances from the Cs-137 source: 105 cm and 250 cm. RL signal for 250 cm is at the level of 40 impulses, while the stem signal is at 20 impulses, but it is still distinguishable.

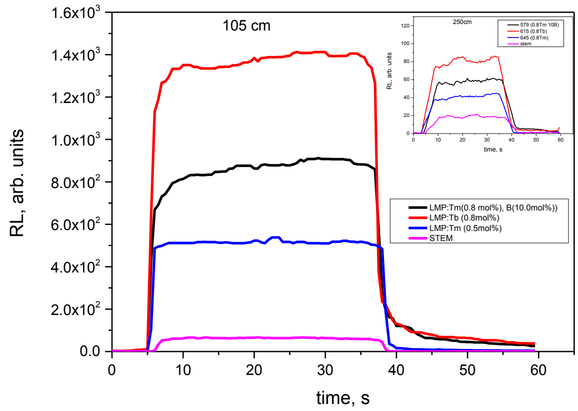

Fig. 12 RL signal of 0.8 mol% Tm and 10 mol% B doped LMP (a); 0.8 mol% Tb doped LMP (b) and 0.5 mol% Tm doped LMP (c) at the distance of 105 cm (dose rate of 683 mGy/h) from the Cs-137 source. Insert – RL signal recorded at the distance of 250 cm (dose rate 30 mGy/h).

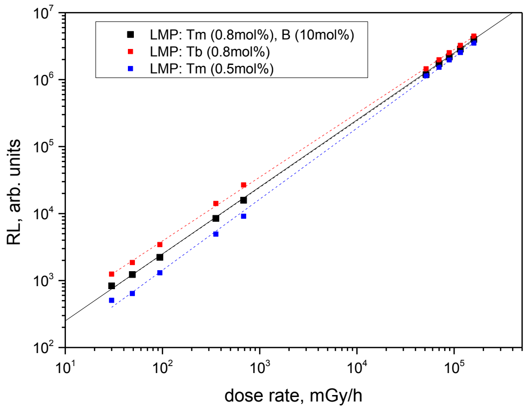

Fig. 13 Integrated RL signal as the function of dose rates (Cs-137 and Co-60 sources) for 0.8 mol% Tm and 10 mol% B doped LMP (a); 0.8 mol% Tb doped LMP (b) and 0.5 mol% Tm doped LMPs. He solid line indicates the linearity trend.

Figures 13 presents a graph of the RL signal obtained for a wide range of dose rates generated by the Cs-137 and Co-60 sources. Despite the differences in the measurement geometry and device mounting method the proportional character of the RL-dose rate function is visible, in the case of LMP:Tm (0.8 mol%), B (10 mol%) this dependence is nearly perfectly linear.

CONCLUSIONS

A small PORTOS device constructed for the measurements of RL of LMP differently doped with Tb and/or Tm was proved to be a useful tool for remote measurements. The current optical configuration of its components allows only for RL recording, but OSL readouts will be possible if a laser is attached.

OSL and TL measurements showed that among all the crystals investigated the significantly highest OSL signal is exhibited by Tb (0.5 mol%) and B (1.0 mol%) doped crystal, whereas the highest TL signal was achieved for Tb (0.2 mol%), B (10 mol%) and Tm (0.6 mol%) doped LMP.

Among all the crystals investigated the highest RL signal was noticed for Tb (0.8 mol%) doped LMP, whereas presence of B atoms causes the decrease of RL. A similarly high RL signal was found for Tm and Tm, B doped LMPs. In this case, a higher Tm content (0.8 mol% or 1.2 mol%) is more important than the presence of B because 0.1 mol% Tm doped LMP had a lower RL. On the other hand, the addition of boron causes the faster stabilization of the RL signal, smaller increase of the RL signal during irradiation and faster fall of the RL signal after stopping irradiation. The dose rate tests conducted in a wide range of dose rates indicated a proportional character of the RL – dose rate relationship. Even the dose rate of 30 mGy/h is detectable for the LMP detector operated in the current optical configuration of the PORTOS system.

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