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THERMOLUMINESCENCE CHARACTERISTICS OF $Zn(BO_2)_2$:Ce^{3+} UNDER BETA IRRADIATION

Nil Kucuk[1,*], Ilker Kucuk[1], Mehmet Yüksel[2] and Mustafa Topaksu[2]
[1]Arts-Sciences Faculty, Physics Department, Uludag University, Gorukle Campus, Bursa 16059, Turkey
[2]Arts-Sciences Faculty, Physics Department, Cukurova University, Adana 01330, Turkey

*Corresponding author: nilkoc@uludag.edu.tr

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In this study, the thermoluminescence (TL) characteristics of undoped and various Ce^{3+}-doped $Zn(BO_2)_2$ powder samples excited by beta irradiation are reported for the first time. $Zn(BO_2)_2$:Ce^{3+} powder samples were prepared by the nitric acid method (NAM) using the starting oxides [zinc oxide (ZnO), boric acid (H_3BO_3) and doped element oxide (CeO_2)]. The formations of the obtained samples were confirmed by an X-ray diffraction study. Dose responses of Ce^{3+}-doped $Zn(BO_2)_2$ samples were investigated after the beta irradiation in the dose range from 143 mGy to 60 Gy. All TL measurements were made on using an automated Risø TL/OSL DA-20 reader. TL emission was detected through a filter pack (Schott BG-39 and Corning 7–59) transmitting between 330 and 480 nm. TL glow curves were obtained using a constant heating rate of 5°C s^{-1} from room temperature (RT) to 450°C in an N_2 atmosphere. The dose response and minimum detectable dose (MDD) values of the samples were determined. The dose responses of all the samples tested exhibited a superlinear behaviour. MDD value of 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample, which shows a high temperature peak at about 230°C, was determined as 96 mGy. MDD values for 1, 2, 3, 5 and 10 % Ce^{3+}-doped $Zn(BO_2)_2$ samples were also determined as 682, 501, 635, 320 and 824 mGy, respectively. The trap parameters of undoped and 4 % Ce^{3+}-doped $Zn(BO_2)_2$ samples were estimated by the computerised glow curve deconvolution method.

INTRODUCTION

Thermoluminescence (TL) is one of the most important methods used in applications for radiation dosimetry[1, 2], which spans areas of health physics and other biological sciences, radiation protection and personnel monitoring. TL experiments give some information related to defects and impurities in solids. In these experiments, a phosphor is excited with some radiations for a desired period of time after which the exciting radiation is removed. Then, the material is heated at a constant rate, and the light output is measured as a function of temperature of the phosphor, and a TL glow curve is plotted. The position of the peaks on the temperature scale is a measure of the energy depth of the trapped electrons in the solid, while the area under the peak often indicates the number of electrons transferred into these traps by exciting radiations[3]. The exciting source may be alpha rays, beta rays, gamma rays, UV rays, ion beams, neutrons, etc. The knowledge of traps with their distribution in the band gap of solids is essential to understand the luminescence process that can be obtained by TL studies. Furthermore, the dosimetric characteristics of TL materials mainly depend on the trap parameters quantitatively describing the trapping–emitting centres responsible for the TL emission[4].

The borates represent a very stable chemical compound. Doped borate phosphors indicate high sensitivity, linearity and good storage properties. These tissue-equivalent phosphors are very convenient for application in radiation dosimetry, particularly in clinical applications and radiation therapy. Many investigations on them have been performed, e.g., $LiCaBO_3$:RE^{3+}[5, 6], $LiSr_4(BO_3)_3$:Ce^{3+}[7], $Li_2B_4O_7$:Cu, Ag[8], LiB_3O_5:Al[9], $Li_2B_4O_7$:Mn, Ag[10], $Li_2B_4O_7$:Cu, Ag, P[11], $LiSrBO_3$:RE^{3+}[12], $KSr_4(BO_3)_3$:Ce[13], $NaSr_4(BO_3)_3$:Ce^{3+}[14], $Sr_2Mg(BO_3)_2$:Tm, Tb, Dy[15], SrB_4O_7:Dy[16], SrB_6O_{10}:Tb[17], MgB_4O_7:Dy, Na[18], MgB_4O_7, MgB_4O_7:Mn, MgB_4O_7:Cu[19], CaB_4O_7, CaB_4O_7:Cu, Mn[20], BaB_4O_7:Ce[21], BaB_4O_7:Dy[22], $Ba_2Ca(BO_3)_2$:Tb[23], $Zn(BO_2)_2$:Tb[24], $Zn(BO_2)_2$:Dy[25], ZnB_2O_4:La[26], $Na_2B_4O_7$:Ag and MgB_4O_7:Cu, Ag[27]. Furthermore, researches on these issues are being carried out for a better understanding and improvement of the material characteristics as well as to develop new TL materials. Cerium being a rare earth ion has the propensity to capture both electron and holes and can exist as Ce^{4+} or Ce^{3+} ion, thus making the study more interesting and important. Ce^{3+} is a popular activator of luminescent materials[28, 29]. As far as it is aware, the luminescence and dosimetric properties of $Zn(BO_2)_2$:Ce^{3+} samples by beta irradiation have not been reported in the literature thus far. A need for knowledge about the results of Ce^{3+} doping has thus motivated the present work, and the true potentialities of these materials for the applications are still unexplored. In the present study, first the TL characteristics by beta irradiation of undoped and various Ce^{3+}-doped $Zn(BO_2)_2$ samples [namely, $Zn(BO_2)_2$,

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$Zn(BO_2)_2$:0.01Ce, $Zn(BO_2)_2$:0.02Ce, $Zn(BO_2)_2$:0.03Ce, $Zn(BO_2)_2$:0.04Ce, $Zn(BO_2)_2$:0.05Ce and $Zn(BO_2)_2$:0.10Ce], synthesised by the nitric acid method (NAM), have been reported. Additionally, the trap parameters are introduced using the computerised glow curve deconvolution (CGCD) method.

EXPERIMENTAL PROCEDURE

Sample preparation

The NAM was employed for the synthesis of undoped and various Ce^{3+}-doped $Zn(BO_2)_2$ samples. The undoped $Zn(BO_2)_2$ sample was prepared as given in Ref. [26]. Similarly, the $Zn(BO_2)_2$:Ce^{3+} samples were also prepared using zinc oxide (ZnO, with a minimum purity of 99.99 %), boric acid (H_3BO_3, with a purity of 99.99 %) and cerium oxide (CeO_2, with a purity of 99.9 %). The stoichiometric quantities of ZnO, H_3BO_3 and CeO_2 powders were separately weighted to prepare the $Zn(BO_2)_2$:Ce^{3+} samples. The starting materials were mixed using a magnetic stirrer at 80°C in 1 M nitric acid solution (HNO_3). During this process, boric acid and all oxides were converted into metallic nitrates [i.e. $Zn(NO_3)_2$, $B(NO_3)_3$ and $Ce(NO_3)_3$]. The expected reactions are as follows:

$$ZnO + 2HNO_3 \rightarrow Zn(NO_3)_2 + H_2O \quad (1)$$

$$H_3BO_3 + 3HNO_3 \rightarrow B(NO_3)_3 + 3H_2O \quad (2)$$

$$CeO_2 + 3HNO_3 \rightarrow Ce(NO_3)_3 + 3/2H_2O + 1/4O_2 \quad (3)$$

Stirring was continued until the dry precursor was obtained. The dry precursor was finely powdered by an agate mortar for about 15 min. The powder sample was transferred into a porcelain crucible, put into an electrical furnace and heated at 450°C for 5 h to remove possible organic compounds, and then cooled[26, 30]. The powder sample pelletized under the pressure of 3 tons was annealed at temperatures from 700 to 900°C for 2 h. Then, the sample was cooled to room temperature (RT), was re-ground in an agate mortar and was placed in an Eppendorf tube.

Sample analysis

In order to characterise the $Zn(BO_2)_2$:Ce^{3+} samples and identify the effect of doping on the structure of undoped $Zn(BO_2)_2$ sample, X-ray diffraction (XRD) method was used. The XRD patterns were recorded using a Rigaku Ultima IV X-ray diffractometer with Cu-K_α (40 kV, 3 deg. min^{-1}, 30 mA, $\lambda = 0.15405$ nm) radiation. Scanning was done between $10° < 2\theta < 90°$.

All TL measurements were carried out on three aliquots of 10 ± 0.1 mg samples using an automated Risø TL/OSL DA-20 reader. TL emission was detected through a filter pack (Schott BG-39 and Corning 7–59) transmitting between 330 and 480 nm. TL glow curves of all the samples were obtained using a constant heating rate of 5°C s^{-1} from RT to 450°C in an N_2 atmosphere. TL glow peak intensities were determined from the areas under dosimetric peaks. The dose responses of all the samples exposed to $^{90}Sr/^{90}Y$ beta source (40 mCi, dose rate: 143 mGy s^{-1}) were obtained in the dose range of 143 mGy to 60 Gy.

RESULTS AND DISCUSSION

XRD patterns

XRD analysis showed that the best crystallisation of Ce^{3+}-doped $Zn(BO_2)_2$ samples occurred at 900°C annealing temperature. The XRD patterns of $Zn(BO_2)_2$ and $Zn(BO_2)_2$:Ce^{3+} samples produced at 900°C are shown in Figure 1. The XRD patterns of the samples were seen to be consistent with that reported in JCPDS Card No. 39–1126. The XRD data showed that the $Zn(BO_2)_2$:Ce^{3+} samples were almost pure phase with high crystallinity. The XRD patterns showed a decrease in the crystallinity of $Zn(BO_2)_2$:Ce^{3+} with increasing Ce dopant content. This can be attributed to the variation in the charges of dopants that results in the defect formation in the lattice. Little impurity phases (viz. H_3BO_3 and B_2O_3) were seen at $2\theta = 16.30°$, 21.54°, 28.38°, 31.18° and 35.66°.

Thermoluminescence (TL) study

The TL properties of undoped and various Ce^{3+}-doped $Zn(BO_2)_2$ samples [viz. TL glow curves, TL dose responses, minimum detectable dose (MDD) values and trap parameters] were given as described in the following subsections.

TL glow curves

Figure 2 shows the TL glow curves of undoped and 2, 4 and 10 % Ce^{3+}-doped $Zn(BO_2)_2$ samples in the beta dose range of 143 mGy to 60 Gy. Main peaks of the samples were observed at around 190–230°C. The glow curve of the undoped $Zn(BO_2)_2$ sample exhibits one strong TL peak at around 191°C when heated at a constant heating rate of 5°C s^{-1} (Figure 2a). Also, the glow curves of 2, 4 and 10 % Ce^{3+}-doped $Zn(BO_2)_2$ samples exhibit a main peak having the maximum temperature at around 210°C with low intensity shoulders at about 320°C (Figure 2b), 230°C with low intensity peak at about 340°C (Figure 2c) and 215°C (Figure 2d), respectively. The glow peak intensities of the main peaks for Ce^{3+}-doped $Zn(BO_2)_2$ samples increase with increasing dose levels. It has shown that traps for these glow peaks are further filled with an increase in the dose. Then, these traps release their charge carriers on

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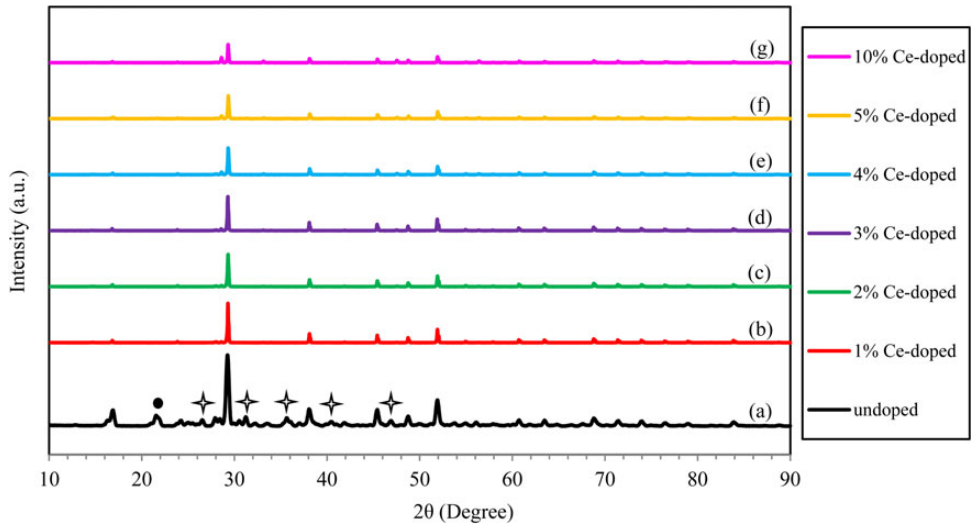


Figure 1. XRD patterns of (**a**) $Zn(BO_2)_2$, (**b**) $Zn(BO_2)_2$:0.01Ce, (**c**) $Zn(BO_2)_2$:0.02Ce, (**d**) $Zn(BO_2)_2$:0.03Ce, (**e**) $Zn(BO_2)_2$:0.04Ce, (**f**) $Zn(BO_2)_2$:0.05Ce and (**g**) $Zn(BO_2)_2$:0.10Ce; (✦) H_3BO_3, (●) B_2O_3.

thermal stimulation to finally recombine with their counterparts, and thus giving rise to different glow peaks[31]. The glow peak intensities of undoped and 2, 4 and 10 % Ce^{3+}-doped $Zn(BO_2)_2$ samples for the 30 Gy increased by approximately 33, 39, 169 and 90 times according to the 1.43 Gy, respectively.

Figure 3 shows the TL glow curves of both undoped and Ce^{3+}-doped samples exposed to 30 Gy. According to this figure, the shape of the TL peaks in the glow curve structures remains almost constant, showing a concentration-independent character. The glow peak intensities and peak temperatures of Ce^{3+}-doped $Zn(BO_2)_2$ samples vary with the amount of dopant (Figure 3). It has been shown in Figure 3 that the TL peak intensity of 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample is much stronger than that of the other samples. The intensity of the TL peak (integrated peak area range 140–450°C) decreases with the increasing of the concentration of Ce^{3+} from 1 to 2 %, and arrives the maximum value at 4 %, and then decreases again with the further increasing of the concentration of Ce^{3+} (Figure 4). It is due to the concentration quenching. Moreover, the main peak temperature is shifted slightly with increasing Ce concentration level. TL emissions of undoped $Zn(BO_2)_2$ sample can be related to intrinsic defects or to some unwanted impurity in the starting powder. It exhibits less TL efficiency than that of the 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample. In such cases, TL response is found to be inadequate for reliable studies in the low doses. Furthermore, the glow curve shape of undoped $Zn(BO_2)_2$ sample is highly changed after the doping. This indicates that there are interactions between intrinsic defects and doped Ce ions.

TL dose responses

The TL dose responses of undoped and Ce^{3+}-doped $Zn(BO_2)_2$ samples can be expressed with an equation form: $TL=a(D)^k$. TL the expression $TL=a(D)^k$, where D is the applied dose, a is a proportionality factor and k is a constant. If $k>1$, the dependence is termed superlinear, whereas $k<1$ means sublinearity and $k=1$ means a linear dose dependence. [log (TL) = log (a) + k log (D)] expression can be obtained by taking the logarithm of both sides of this equation. Here, the slope of k shows the linearity factor. The linearity factor can be found with a linear fit of log (TL) versus log (D)[26, 32].

The TL dose responses of all the samples exposed to beta radiation were determined in the dose range of 143 mGy to 60 Gy. Figure 5 shows a set of the log (TL) as a function of the log (Dose) for undoped and 1, 3 and 4 % Ce^{3+}-doped $Zn(BO_2)_2$ samples, and the line through the data points is the best fit of $TL=a(D)^k$ equation. The TL dose–response curves for all the samples were observed to be superlinear 143 mGy−60 Gy region studied. Table 1 gives the peak temperature at the maximum (T_m), the background counts and the k values with its standard error (Δk). The k values of the samples were determined to be between 1.05 and 1.46. This indicates superlinear dose dependence.

Minimum detectable dose

MDD values of all the samples are given in Table 1. These were determined as a dose of three times the corresponding value of background count[18, 33, 34].

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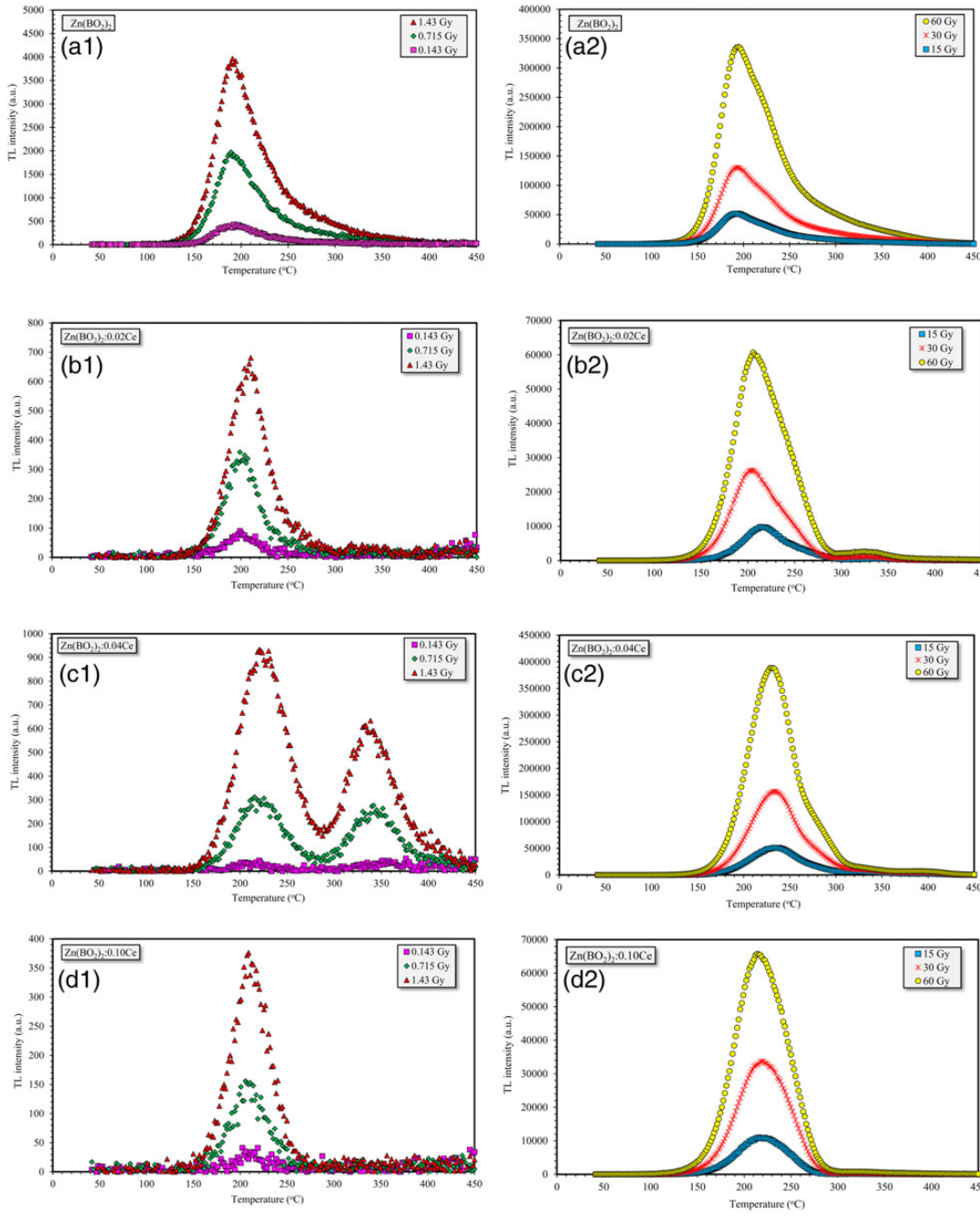


Figure 2. TL glow curves of (**a1**–**a2**) $Zn(BO_2)_2$, (**b1**–**b2**) $Zn(BO_2)_2$:0.02Ce, (**c1**–**c2**) $Zn(BO_2)_2$:0.04Ce and (**d1**–**d2**) $Zn(BO_2)_2$:0.10Ce under beta irradiation.

MDD value of 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample was found as 96 mGy. MDD values for undoped and 1, 2, 3, 5 and 10 % Ce^{3+}-doped $Zn(BO_2)_2$ samples were also found as 130, 682, 501, 635, 320 and 824 mGy, respectively. Furthermore, the superlinearity of the TL dose response of all the samples to high radiation doses was obtained by dividing of integrated intensities observed for 60 Gy to the integrated

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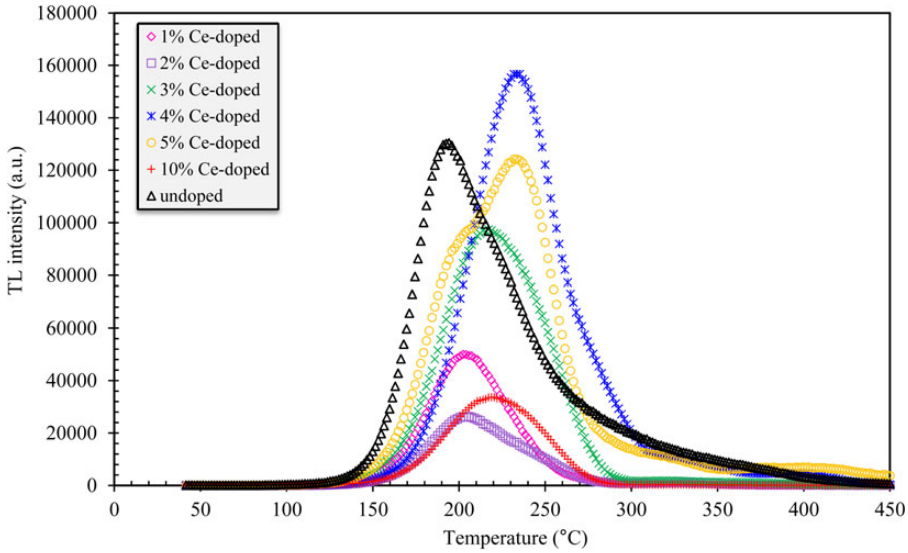


Figure 3. TL glow curves of undoped and Ce^{3+}-doped $Zn(BO_2)_2$ samples exposed to 30 Gy.

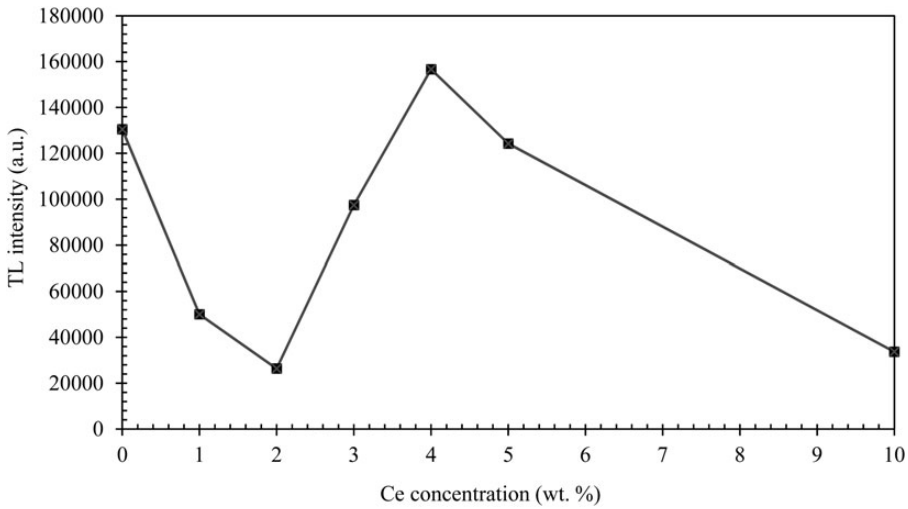


Figure 4. Variation in TL intensity with cerium concentration for 30 Gy.

intensities observed for 30 Gy (viz. I_{60}/I_{30}). These results are also given in Table 1.

Analysis of trap parameters by the CGCD method

Evaluation of trap parameters associated with the glow peaks of TL glow curves is one of the most important aspects of these studies. Trap parameters give valuable information about mechanism responsible for the TL emission in material. So, reliable dosimetric studies of any TL material should be based on a good knowledge of its trap parameters as well. Therefore, the trap parameters of the TL peaks of undoped and 4 % Ce^{3+}-doped $Zn(BO_2)_2$ samples have been estimated using the CGCD method. This method has become very popular to obtain these parameters. It has great advantages over the experimental methods (i.e. initial rise, peak shape, etc.) owing to simultaneous determination of trap parameters of all peaks without additional thermal treatments and experimental repetitions. Furthermore, this method uses all data points in the whole glow

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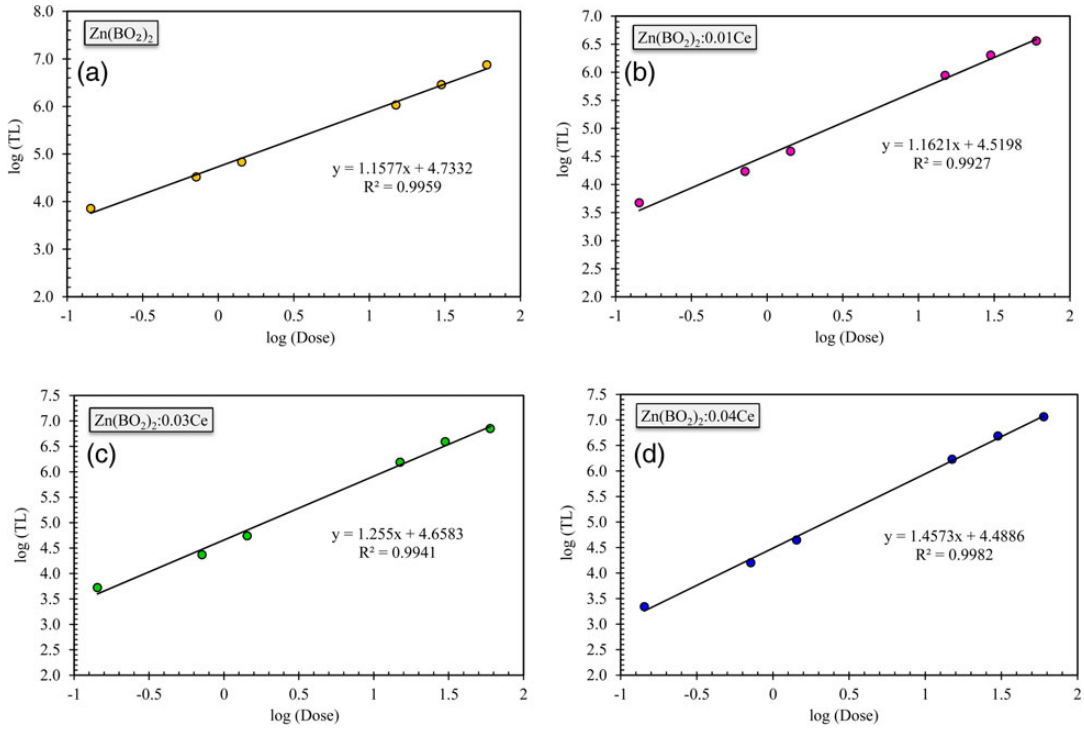


Figure 5. TL dose–response curves of (**a**) $Zn(BO_2)_2$, (**b**) $Zn(BO_2)_2$:0.01Ce, (**c**) $Zn(BO_2)_2$:0.03Ce and (**d**) $Zn(BO_2)_2$:0.04Ce after exposure to beta radiation in the dose range of 143 mGy to 60 Gy.

Table 1. Dose responses and MDD values for $Zn(BO_2)_2$ and $Zn(BO_2)_2$:Ce^{3+} samples.

Sample	T_m (°C)	Background counts	$k \pm \Delta k$	I_{60}/I_{30}	MDD (mGy)
$Zn(BO_2)_2$	191	5630	1.16 ± 0.04	2.6	130 ± 1
$Zn(BO_2)_2$:0.01Ce	210	12 829	1.16 ± 0.05	1.8	682 ± 33
$Zn(BO_2)_2$:0.02Ce	210	7472	1.11 ± 0.07	2.4	501 ± 43
$Zn(BO_2)_2$:0.03Ce	210	28 189	1.26 ± 0.05	1.8	635 ± 10
$Zn(BO_2)_2$:0.04Ce	230	8829	1.46 ± 0.03	2.4	96 ± 7
$Zn(BO_2)_2$:0.05Ce	230	25 546	1.05 ± 0.07	2.2	320 ± 12
$Zn(BO_2)_2$:0.10Ce	220	13 017	1.34 ± 0.07	2.0	824 ± 6

curve rather than just a few points during the curve-fitting procedures. It is apparent that if the number of data points used in the analysis increases, the potential for accurate determination of the trap parameters gets better[21].

For a constant heating rate, the peak maximum (T_m) should not be affected by other experimental parameters and thus should be completely constant within the limit of experimental errors. It is noted that the positions of peak maximum do not shift with increasing dose level for first-order kinetics. But, the peak maximum is shifted to the lower temperature region with increasing dose level for general-order kinetics ($1<b<2$). The positions of the peak at around 200°C in the glow curve of $Zn(BO_2)_2$:Ce^{3+} are within the experimental error of ± 4°C for all the doses. However, as shown in Figure 2a–d the peak temperature of glow peak at around 200°C is not shifting with high dose levels, and this point clearly indicates that this peak is of first-order kinetics[35–39]. Therefore, GlowFit software was selected to analyse the TL glow peak of $Zn(BO_2)_2$:Ce^{3+} at around 200°C.

In this study, the TL glow curves of undoped and 4 % Ce^{3+}-doped $Zn(BO_2)_2$ samples for the selected 30 Gy beta radiation dose were decomposed with the GlowFit software. Figure 6 shows the analysed glow

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curves of these samples. The analysed TL glow curves as shown in Figure 6a consist of six glow peaks. The TL glow curves of undoped $Zn(BO_2)_2$ sample exhibit three prominent glow peaks at around 185, 202 and 218°C; two low intensity peaks at around 242 and 262°C on the high-temperature sides of Peak 3; and one shoulder peak at around 321°C on the high-temperature sides of Peak 5. The analysed TL glow curves as shown in Figure 6b consist of five glow peaks. The TL glow curves of 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample exhibit one low intensity peak at around 214°C on the low-temperature sides of Peak 2, two prominent glow peaks at around 234 and 261°C, and two shoulder peaks at around 311 and 394°C on the high-temperature sides of Peak 3. The accuracy of these presented values is supportable with figure of merit (FOM) values. FOM values are 1.38 % for $Zn(BO_2)_2$ and 1.09 % for $Zn(BO_2)_2$:0.04Ce. Therefore, the trapping parameters [i.e. order of kinetics (b), activation energies (E_a) and frequency factors (s)] associated with the glow peaks in the samples were obtained by the CGCD method. The obtained trap parameters and T_m values were given in Table 2. As shown in Table 2, all of the samples have first-order kinetic. The activation energies and the frequency factors of undoped $Zn(BO_2)_2$ sample were determined as 1.38 eV and 6.43×10^{15} s^{-1} for the first peak, 1.55 eV and 1.01×10^{15} s^{-1} for the second peak, 1.19 eV and 5.59×10^{12} s^{-1} for the third peak, 1.27 eV and 6.60×10^{12} s^{-1} for the fourth peak, 0.65 eV and 1.94×10^{9} s^{-1} for the fifth peak, and 0.50 eV and 1.40×10^{7} s^{-1} for the sixth peak, respectively. The activation energies and the frequency factors of 4 % Ce^{3+}-doped $Zn(BO_2)_2$ sample were also determined as 1.28 eV and 5.40×10^{13} s^{-1} for the first peak, 1.17 eV and 1.01×10^{12} s^{-1} for the second peak, 0.92 eV and 9.91×10^{8} s^{-1} for the third peak, 0.77 eV and 6.01×10^{8} s^{-1} for the fourth peak, and 1.30 eV and 1.16×10^{10} s^{-1} for the fifth peak, respectively.

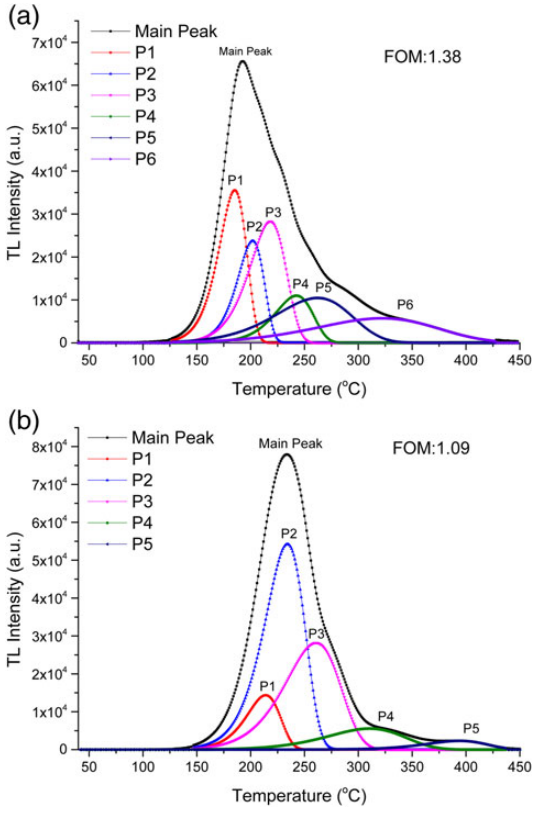


Figure 6. CGCD of TL glow curves of (**a**) $Zn(BO_2)_2$ and (**b**) $Zn(BO_2)_2$:0.04Ce for 30 Gy.

CONCLUSIONS

Undoped and various Ce^{3+}-doped $Zn(BO_2)_2$ samples were prepared by NAM. The TL characteristics of all the samples have been investigated under beta irradiation. Main peaks of the samples were observed at around 190–230°C. It has been seen that the glow

Table 2. Trap parameters obtained by the CGCD method of $Zn(BO_2)_2$ and $Zn(BO_2)_2$:0.04Ce samples for 30 Gy.

Sample	Peaks	T_m (°C)	E_a (eV)	s (s^{-1})	b	%FOM
$Zn(BO_2)_2$	1	185	1.38	6.43×10^{15}	1	1.38
	2	202	1.55	1.01×10^{15}	1	
	3	218	1.19	5.59×10^{12}	1	
	4	242	1.27	6.60×10^{12}	1	
	5	262	0.65	1.94×10^{9}	1	
	6	321	0.50	1.40×10^{7}	1	
$Zn(BO_2)_2$:0.04Ce	1	214	1.28	5.40×10^{13}	1	1.09
	2	234	1.17	1.01×10^{12}	1	
	3	261	0.92	9.91×10^{8}	1	
	4	311	0.77	6.01×10^{8}	1	
	5	394	1.30	1.16×10^{10}	1	

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peak intensities of the main peaks increase with increasing dose levels. The TL dose responses of all the samples exposed to beta radiation were determined to be superlinear in the dose range of 143 mGy to 60 Gy. MDD value of $Zn(BO_2)_2$:0.04Ce sample was found as 96 mGy. MDD values for $Zn(BO_2)_2$, $Zn(BO_2)_2$: 0.01Ce, $Zn(BO_2)_2$:0.02Ce, $Zn(BO_2)_2$:0.03Ce, $Zn(BO_2)_2$: 0.05Ce and $Zn(BO_2)_2$:0.10Ce samples were also found as 130, 682, 501, 635, 320 and 824 mGy, respectively. It has been found that the TL peak intensity of $Zn(BO_2)_2$:0.04Ce sample exposed to 30 Gy is much stronger than that of the other samples. Therefore, the optimum Ce^{3+} concentration is 4 % to obtain the highest TL intensity. The trap parameters of $Zn(BO_2)_2$ and $Zn(BO_2)_2$:0.04Ce were calculated by the CGCD method. The reported characteristics indicate that $Zn(BO_2)_2$:Ce^{3+} may be a new promising thermoluminescence dosimetry material for the use in ionising radiation dosimetry. However, the present studies are not enough to propose any concrete and meaningful TL mechanism in these samples. Hence, further investigation on the TL mechanism and parameter calculations are under progress.

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