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Thermoluminescence kinetic features of Lithium Iodide (LiI) single crystal grown by vertical Bridgman technique

D. Joseph Daniel [a], H.J. Kim [a, *], Sunghwan Kim [b], Sajid Khan [c]

[a] Department of Physics, Kyungpook National University, Daegu, 41566, South Korea
[b] Department of Radiological Science, Cheongju University, Cheongju, 28503, South Korea
[c] Department of Physics, Kohat University of Science and Technology, Kohat, 26000, Pakistan

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ABSTRACT

Single crystal of pure Lithium Iodide (LiI) has been grown from melt by using the vertical Bridgman technique. Thermoluminescence (TL) Measurements were carried out at 1 K/s following X-ray irradiation. The TL glow curve consists of a dominant peak at (peak-maximum T_m) 393 K and one low temperature peak of weaker intensity at 343 K. The order of kinetics (b), activation energy (E), and the frequency factor (S) for a prominent TL glow peak observed around 393 K for LiI crystals are reported for the first time. The peak shape analysis of the glow peak indicates the kinetics to be of the first order. The value of E is calculated using various standard methods such as initial rise (IR), whole glow peak (WGP), peak shape (PS), computerized glow curve deconvolution (CGCD) and Variable Heating rate (VHR) methods. An average value of 1.06 eV is obtained in this case. In order to validate the obtained parameters, numerically integrated TL glow curve has been generated using experimentally determined kinetic parameters. The effective atomic number (Z_{eff}) for this material was determined and found to be 52. X-ray induced emission spectra of pure LiI single crystal are studied at room temperature and it is found that the sample exhibit sharp emission at 457 nm and broad emission at 650 nm.

1. Introduction

Thermoluminescence (TL) is a very powerful technique used for radiation dosimetry as the energy absorbed by the phosphor on being exposed to ionizing radiation can be easily measured as light on stimulating it with heat [1]. Inorganic scintillators are widely used for the detection of radiation in research, medical diagnostics and industry. Among those, alkali iodides based scintillators (CsI, CsI:Tl, CsI:Na, NaI:Tl and LiI) have received consistent attention for applications in the field of high energy physics, nuclear physics, medicine, industry, security and environmental control devices, geology, astrophysics, etc. [2–4].

Irradiation of alkali halides by high energy ions (X-ray, Gamma ray) causes the formation, besides separated electron-hole (e-h) pairs, of anion excitons, the non-radiative decay of which leads to the creation of anion Frenkel defect pairs and of the groups of spatially correlated radiation defects [5]. And these defects can be studied by TL experimental results with the different methods of calculation. The origin and mechanism of these trap centers have been investigated by several researchers [6–9].

LiI crystallizes in face centered cubic system with space group of $Fm\bar{3}$ m (225), and the cell parameter a = 5.930 Å [10]. Recently, the scintillation properties of doped LiI crystal such as energy resolution, decay time and absolute light yield were reported by S. Khan et al. [11,12]. However, to the best of our knowledge, there is no systematic studies on TL kinetic features of LiI single crystal. In this paper, we present further results of our investigation on TL in this system. TL kinetic parameters such as order of kinetics (b), activation energy (E) and frequency factor (S) for the main peak at around 393 K have been determined and reported for the first time using standard methods such as initial rise (IR), whole glow peak (WGP), peak shape (PS), computerized glow curve deconvolution (CGCD) and variable heating rate (VHR) methods.

2. Experimental

Pure LiI Crystals have been grown by using a two-zone vertical Bridgman technique. 4N purity of LiI was used as starting materials for the single crystal growth. Powders were loaded into quartz

* Corresponding author.
E-mail addresses: hongjooknu@gmail.com, hongjoo@knu.ac.kr (H.J. Kim).

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ampoules located in a glove box with Ar atmosphere. The ampoules were vacuum sealed under a dynamic pressure of ~10^{-7} Torr. The vacuum sealed quartz ampoules were slowly translated down through an axial temperature gradient of ~5 °C/cm with a speed of 0.5 mm/h. Due to the hygroscopic nature of lithium iodide, the samples were kept in mineral oil. TL was measured using a HARSA 4500 manual TLD Reader from a sample irradiated at ambient temperature using a X-ray source. The TL reader is connected to a Personal Computer, where the TL signals were recorded and analyzed by the usage of the WinREMS software. Samples were heated using a planchet at the linear heating rate of 1K/sec, from room temperature to 623 K.

3. Results and discussion

3.1. TL studies

TL measurement was performed on pure LiI crystal and shown in Fig. 1. TL glow peak at about 393 K with a spectrum closely similar to the NaI(Tl) reported by S.C. Sabharwal et al. [13]. The LiI crystal is chemically similar to sodium iodide and also hygroscopic. The irradiation of LiI by X-rays at room temperature leads to the creation of F–H pairs of Frenkel defects via the decay of electronic excitations. Highly mobile H centers (interstitial halogen atoms $(X_2^-)_a$) undergo pair interaction with other H centers or V_K and V_F centers forming tri-halogen (Iodine molecule) (X_3^-) molecules which is stable up to high temperature. The trihalide (X_3^-) molecules occupying either, two anion and one cation lattice sites $(X_3^-)_{aca}$ or two anion sites-less stable configuration $(X_3^-)_{aa}$. This high temperature peak at 393 K is due to the thermal destruction with the release of mobile holes. Similar observations of self-trapped hole, have been reported in several alkali halides [5,14].

3.1.1. Order of kinetics (b)

The kinetics of order gives information about whether the trapped charge carriers will be retrapped or not during the process of heat. The distinction between the first and second order of the peak can be determined from the symmetry of the TL glow curve. In the case of first order kinetics, the peak has a characteristic asymmetric shape, being wider at the low temperature side than high temperature side and for second-order kinetics the main characteristic of this curve is that it is almost symmetrical.

$$\mu_g = \delta/\omega \tag{1}$$

This asymmetry can be evaluated through the geometric symmetry factor (μ_g) of the glow peak using equation (1) from the known peak shape parameters ($\delta = T_2 - T_m$) and ($\omega = T_2 - T_1$). According to Chen [15], the values of (μ_g) for first and second order kinetics are 0.42 and 0.52 respectively. The (μ_g) value obtained for the pure LiI crystal TL glow peak is 0.4019 which is very close to 0.42 and suggests that this peak obeys first order kinetics. Balarin [16] suggests the kinetic order as a function of the parameter $\gamma = \delta/\tau$, where $\tau = T_m - T_1$. This parameter ranges from 0.7 to 0.8 for a first order peak and from 1.05 to 1.20 for a second order peak. This parameter (γ) value was obtained for LiI crystal to be 0.72, which also suggest this TL glow peak belongs to first order kinetics. From the whole glow peak method, several graphs are drawn of $In\left(TL/n^b\right)$ versus $1/kT$ as shown in Fig. 2. The regression line for b = 1 (order of kinetics) gives the largest regression coefficient R^2. Within the framework of the whole glow-peak method of analysis, we can conclude that the TL glow peak of LiI crystal follow the first-order kinetics.

3.1.2. Peak shape method

According to the peak shape method, the shape of the TL glow peak are considered to find the kinetic parameters [15,17]. The E can be calculated using the general expressions given by Chen [18], which is valid for all kinetics.

$$E = C_\alpha\left(kT_m^2/\alpha\right) - b_\alpha(2kT_m) \tag{2}$$

where α stands for τ, δ and ω respectively. k is Boltzmann constant and T_m is maximum TL intensity temperature. C_α and b_α are obtained using the expressions given below:

$$C_\tau = 1.51 + 3\left(\mu_g - 0.42\right) \qquad b_\tau = 1.58 + 4.2\left(\mu_g - 0.42\right)$$
$$C_\delta = 0.976 + 7.3\left(\mu_g - 0.42\right) \qquad b_\delta = 0$$
$$C_\omega = 2.52 + 10.2\left(\mu_g - 0.42\right) \qquad b_\omega = 1$$

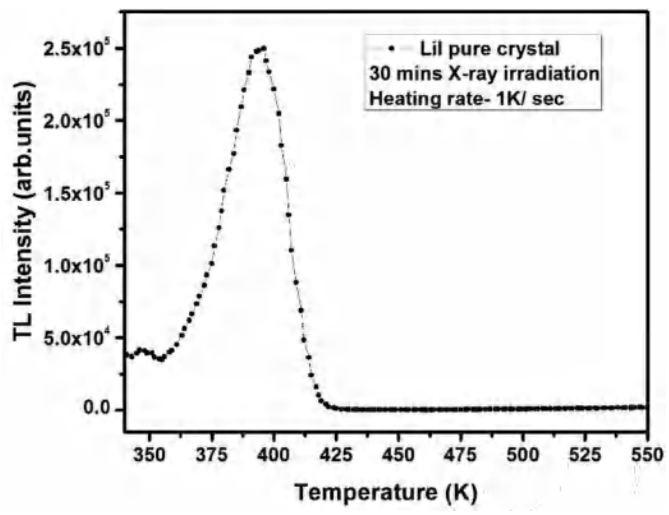

Fig. 1. TL glow curve of nominally pure LiI single crystal.

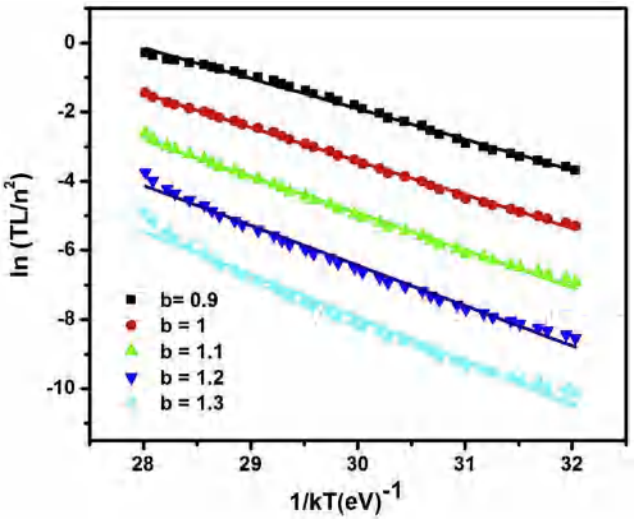

Fig. 2. Plots of 1/kT versus In (TL/n^2).

$$E = 0.976\, kT_m^2 \Big/ \delta \tag{3}$$

$$E = 1.51\left(\frac{kT_m^2}{\tau}\right) - 3.16\, kT_m \tag{4}$$

Calculation of E by Lushchik [19] and Halperin-Braner [20] method also has been carried out for the pure LiI crystal using the first order kinetics equations (3) and (4). After obtaining the order of kinetics (b) and E, S can be calculated using the following general expression by substituting the obtained values of E & b.

$$s = \frac{\beta E}{kT_m^2} exp^{\left(E/kT_m\right)}\left[1 + (b-1)\left(\frac{2kT_m}{E}\right)\right]^{-1} \tag{5}$$

where β is the heating rate and k is the Boltzmann constant. Using equations, (2, 3, 4) and (5) the E and S were determined for pure LiI single crystal and the mean values of E_δ, E_τ, E_ω and s_δ, s_τ, s_ω are shown in Table 1. It shows clearly that there is no much deviation in the calculated values of E_δ, E_τ, E_ω. This shows that the TL peak of LiI crystal is simple and well divided from the low temperature satellite peak.

3.1.3. Whole glow peak method

For well-defined TL glow peak, the whole glow peak method is suitable to calculate the trapping parameters. The expression for the general order kinetics,

$$In\left(I/n^b\right) = In\left(S^{-1}/\beta\right) - E/kT \tag{6}$$

where n is the integral area under the TL glow curve, b is the order of kinetics, β is the heating rate. For a specific value of b, the plot of $In\left(I/n^b\right)$ against $^1/_{kT}$ is linear with slope E. Fig. 2 shows the semi logarithmic plots for some values of b. The plots in Fig. 2 corresponding to b = 0.9, 1.1, 1.2 and 1.3 clearly deviate from straight lines, and must be rejected. The plot was found to be most linear for b = 1.0 (R^2 = 0.99829). The S is calculated from the y-intercept of the linear plot of equation (6) and it is found to be 1.68×10^{11}.

3.1.4. Initial rise method

The initial rise method is the simplest method to calculate the trap depth of the TL materials first suggested by Garlick and Gibson in 1948 [21]. This method is based on the assumptions that in the low temperature tail part of the TL glow curve where T << T_m, the rate of change of trapped carriers population is negligible. The TL intensity is proportional to $exp^{\left(-E/kT\right)}$, assuming the S to remain the same and there is no overlapping of glow peaks.

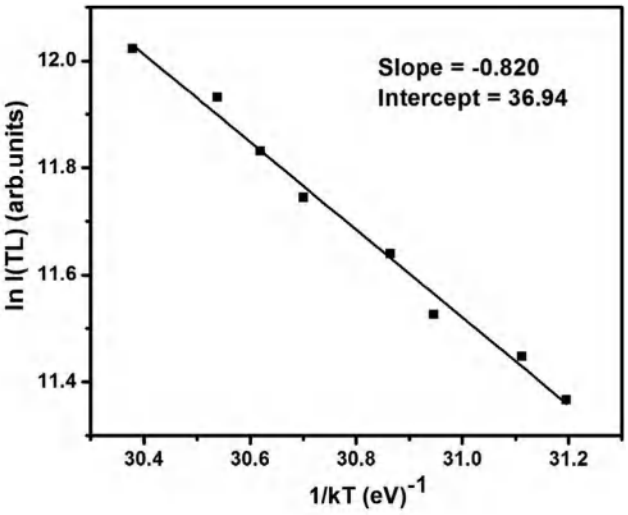

Fig. 3. Plot of ln(TL) versus 1/kT graph for TL glow peak of LiI pure crystal.

$$I(TL) = Cexp^{\left(-E/kT\right)} \tag{7}$$

where C is the constant, I(TL) is the TL intensity at any temperature T, when the sample is heated at a linear heating rate of β = dT/dt, E is the thermal activation energy and k is Boltzmann's constant. E can be calculated by plotting a graph between $In\, I(TL)$ versus 1/kT, over the initial rise region which represents a straight line. The slope of this straight line gives the value of E without any knowledge of S and order of kinetics (b). The $In\, I(TL)$ versus 1/kT graph for the main TL glow peak of LiI pure crystal is shown in Fig. 3. The value of the E for the main peak at around 393 K has been calculated from the slope of the straight line graph. From the intercept I obtained from the plot of $In\, I(TL)$ versus $^1/_{kT}$ graph for the initial rise part, one can find the value of S. The general order expression for s is given below,

$$s = antilog(I - InA - (b-1)In\left(n_{0/N}\right)) \tag{8}$$

where b is the order of kinetics, N is the total concentration of the traps, n_0 is the initial concentration of trapped electrons, I is intercept of the initial rise plot and A is the area under the TL peak. For first order kinetic TL process (b = 1) and hence the expression for s reduces to:

$$s = antilog\,(I - In(A)) \tag{9}$$

The trap depth and S of the LiI crystal TL glow peak was determined by initial rise method and found to be 0.82 eV and 1.38×10^9.

Table 1
TL kinetic parameters calculated by peak shape method for LiI crystal.

Peak shape Method	E (eV)			S (s^{-1})		
Chen's	E_δ 0.9926	E_τ 1.0028	E_ω 1.0747	s_δ 4.89×10^{11}	s_τ 6.69×10^{11}	s_ω 6.09×10^{12}
E_L (Lushchik)		0.99258			6.51×10^{12}	
E_{HB} (Halperin-Braner)		1.002776			8.82×10^{12}	
Average		**1.0131**			**4.51 × 10^{12}**	

3.1.5. Deconvolution method

CGCD technique was used to find the kinetic parameters of overlapping TL glow peaks using the Microsoft Excel Spreadsheet program [22]. This method allows the optimization of some fundamental characteristics of a complex TL glow peak system. The general order kinetics (GOK) expression is given by Kitis et al., 1998 [23].

$$I(T) = I_m b^{b/b-1} e^{\left(\frac{ET-T_m}{kT\ T_m}\right)} \left[(1-b)(1-\Delta)\frac{T^2}{T_m^2} \times e^{\left(\frac{ET-T_m}{kT\ T_m}\right)} + Z_m\right]^{\frac{-b}{b-1}} \quad (10)$$

where, $\Delta = \frac{2kT}{E}$, $\Delta_m = \frac{2kT_m}{E}$ *and* $Z_m = 1 + (b-1)\Delta_m$ was used for the fitting.

The solver is a Microsoft Excel Add-in general purpose software package [24] that minimizes the sum of the squares of the residuals to perform a least-squares fitting. The experimentally obtained values such as TL glow peak intensity (I_m), maximum peak temperature (T_m) and the guess values of E (eV) and order of kinetics (b) were used as fitting parameters. By changing the values of these parameters, the experimental glow curve was fitted. The goodness of the fitting was verified by the figure of (FOM%) is a minimum.

$$FOM = \frac{\sum_P \left|y_{exp} - y_{fit}\right|}{\sum_P y_{fit}} \quad (11)$$

where, y_{exp} and y_{fit} signify the experimentally observed TL intensity and the values of the fitting function, respectively. The experimental TL glow curve of LiI crystal was fitted to one main peak along with one satellite peak of lesser intensity. Fig. 4 shows the experimental data along with the fitted glow curves. The Figure of Merit (FOM) of the fitting is 2.44%. For a good fitting, the value of the FOM should be less than 5% [25]. The value for the E was found to be 1.16 eV, and the S was found to be 5.85×10^{13}. The values of S in the range of 10^8 ~ 10^{11} are normally encountered in TL materials. The order of kinetics was 1.10.

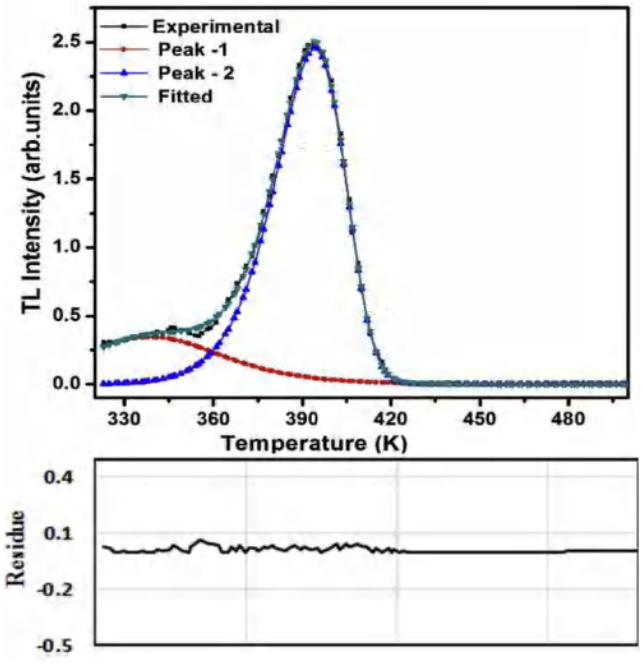

Fig. 4. TL Glow curve deconvolution of pure LiI crystal.

3.1.6. Variable heating rate method

Heating rate is an important experimental variable in TL measurements. TL glow curves of X-ray irradiated pure LiI single crystals at different heating rates from 1 to 5 K/sec are shown in Fig. 5. It has been observed that there is no change in the TL glow curve shape and structure at various heating rates. As the heating rate is increased, the position of the TL maximum (T_m) shifts toward higher temperatures. Temperature lag between the heating element and the TL sample is the important point that has to be taken into account to avoid large deviation in the kinetic parameter calculation by the VHR method. In order to avoid this temperature lag problem, Kitis and Tuyn [26] have proposed a simple method using the following expression.

$$T_{mj} = T_{mi} - C \cdot ln\left(\frac{\beta_i}{\beta_j}\right) \quad (12)$$

where, T_{mj} and T_{mi} are the maximum peak temperature with the heating rate of β_j *and* β_i respectively and C is a constant. The inset of Fig. 5 shows that the corrected maximum peak temperature using temperature lag effect.

Booth and Bohun proposed a method to calculate the kinetic parameters from the different sets of heating rates and their corresponding peak temperatures [27,28].

$$E = \frac{kT_{m1}T_{m2}}{T_{m1} - T_{m2}} \ln\left[\frac{\beta_1}{\beta_2}\left(\frac{T_{m2}}{T_{m1}}\right)^2\right] \quad (13)$$

$$s = \frac{E}{k} \exp\left(\frac{T_{m2} ln\left(\frac{T_{m2}^2}{\beta_2}\right) - T_{m1} ln\left(\frac{T_{m1}^2}{\beta_1}\right)}{T_{m1} - T_{m2}}\right) \quad (14)$$

where, T_{m1} and T_{m2} are temperatures corresponding to the maximum TL intensities for heating rates β_1 and β_2 respectively. The value of S can be evaluated by substituting E in equation (14). The value of E and S of pure LiI crystal has been calculated using expressions (13) and (14) for different sets of heating rates β_1 and β_2

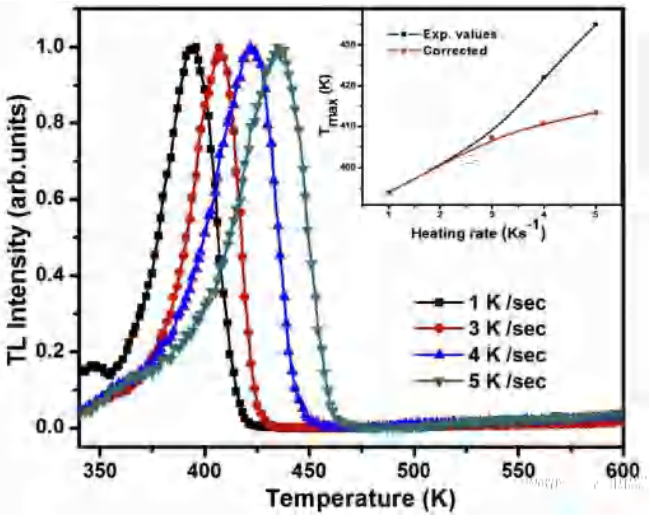

Fig. 5. Behavior of experimental T_m values of LiI crystal against the different heating rate and the inset shows the corrected Tm values using temperature lag effect.

Table 2
Kinetic parameters values of pure LiI crystal calculated by Variable heating rate method.

T_{m1} (K)	T_{m2} (K)	β_1 (K/sec)	β_2 (K/sec)	E (eV)	S (s^{-1})
407.28	393.94	3	1	1.070	3.86×10^{12}
410.77	393.94	4	1	1.080	5.18×10^{12}
410.77	407.28	4	3	1.118	1.60×10^{13}
413.48	393.94	5	1	1.087	6.47×10^{12}
413.48	407.28	5	3	1.125	1.96×10^{13}
413.48	410.77	5	4	1.134	2.56×10^{13}
Average				**1.102**	**1.28×10^{13}**

are given in Table 2.

Hoogenstraaten [29] has used various heating rates to calculate E by graphically, based on the position of T_m with corresponding heating rate β.

$$ln\left(\frac{T_m^2}{\beta}\right) = ln\left(\frac{E}{sk}\right) + \frac{E}{kT_m} \quad (15)$$

A plot of $ln\left(\frac{T_m^2}{\beta}\right)$ against $\left(\frac{1}{kT_m}\right)$ should yield a straight line. From the graph shown in Fig. 6, slope equals to E (eV) and intercept value equals to $ln\left(\frac{E}{sk}\right)$. E and S of LiI calculated from Fig. 6 are found to be at 1.08 eV and 5.80 × 10^{12} (s^{-1}) respectively.

3.1.7. Comparison of TL kinetic parameters

A comparative study was carried out with the calculated E by the various methods for LiI single crystal. The mean average value of E and S obtained from the different methods are shown in Table 3. The E of the peak is about ~1.06 eV and the S is of the order of 10^{13} s^{-1}. Value of E for the LiI crystal obtained from the initial rise method is lower than that calculated by other methods. Since, this method is based on the approximation of the integral, it leads to a systematic error so that the computed value of E becomes lower than its actual value. According to Wintle (1975), due to the presence of thermal quenching, the value of E obtained from initial rise method will be lower than the actual value by an amount 'φ' which is an energy parameter characterizing the non-radiative transitions in the luminescence centers [30]. The value of S varies in the range of 10^{11} - 10^{13} magnitude across the various methods for pure LiI crystal studied and this is consistent with what is reported in the literature. The calculated order of kinetics is in good agreement with different methods. The value of order of kinetics are in good agreement with calculated by different methods.

Table 3
Kinetics parameters calculated for TL glow peak of LiI single crystal using various methods.

Methods	E (eV)	S (s^{-1})
Peak shape method	1.01	4.51×10^{12}
Whole glow peak	0.98	1.68×10^{11}
Deconvolution method	1.16	5.85×10^{13}
Variable Heating Rate method	1.10	1.28×10^{13}
Graphical method	1.08	5.80×10^{12}
Average	**1.06**	**1.63×10^{13}**

3.1.8. Validation of kinetic parameters

In order to validate the calculated kinetic parameters, the TL glow curve has been numerically integrated using the experimentally calculated kinetic parameters. Numerically integrated TL glow peak generated using the following general-order kinetic expression.

$$I(t) = sn_0 exp(-E/kT)\left\{\frac{(b-1)s}{\beta}\int_{T_0}^{T} exp(-E/kT')dT' + 1\right\}^{-\frac{b}{b-1}}. \quad (16)$$

The same heating rate as that used for experimental measurements was used for the generation of synthetic TL glow curve. Numerically integrated and experimentally observed TL glow peaks for LiI crystal are shown in Fig. 7.

3.2. X-ray induced luminescence spectra

The X-ray induced luminescence measurement was carried out by using an X-ray tube from DRGEM.Co. with a tungsten anode at ambient temperature. The power setting parameters of the tube were 80 kV and 1 mA. The X-ray emission spectra were acquired by using a fiber optic spectrometer (QE65000, Ocean Optics). X-ray induced luminescence involves typical electron hole recombination

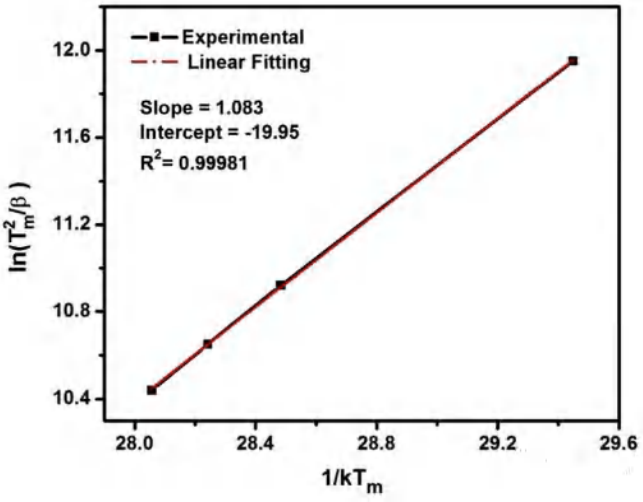

Fig. 6. Plot of $\ln(T_m^2/\beta)$ against $1/kT_m$.

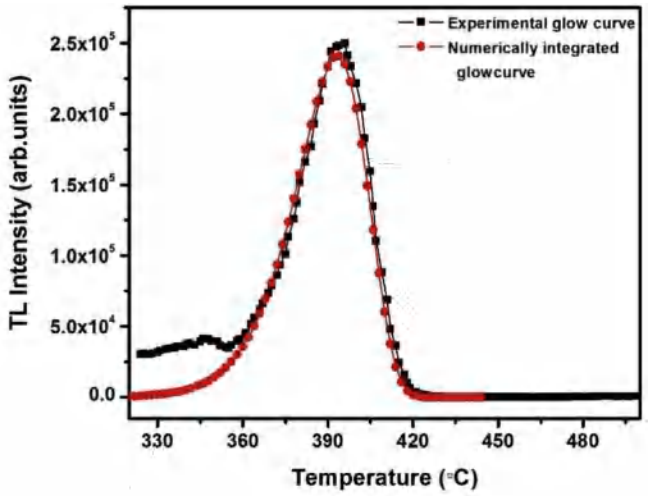

Fig. 7. Comparison of TL glow curves of LiI crystal experimental and numerically integrated.

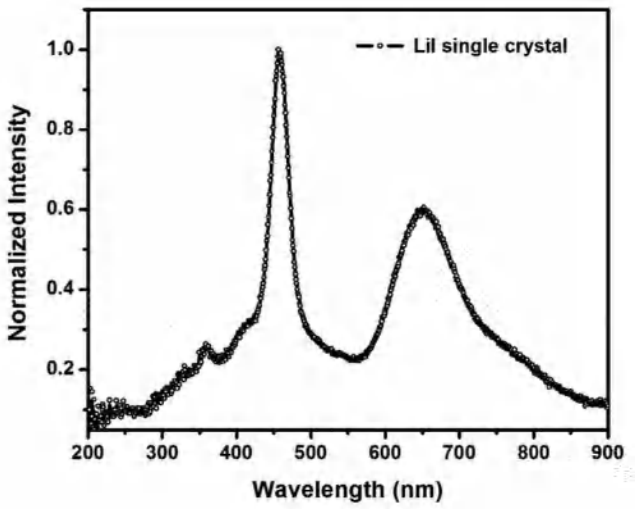

Fig. 8. X-ray induced luminescence spectra of nominally pure LiI single crystal.

events as well as electronic and ionic processes taking place during the irradiation. The exposure of alkali halide crystals to X-rays is known to create several absorption centers such as non-radiative decay of self-trapped excitons e_s^0 (STE) or non-radiative recombination of electrons e^- with self-trapped holes $(X_2^-)_{aa}$ (V_k centers) with the creation of anion Frenkel defect pairs (FD): anion vacancies V_a (α - centers) and interstitial halogen ions X_i^- (I - centers) as well as F-centers $(V_a e^-)$ and interstitial halogen atoms $(X_2^-)_a$ (H-centers) [31]. The X-ray induced luminescence spectrum of the pure LiI crystal was recorded at room temperature and shown in Fig. 8. It exhibited two well resolved peaks at 457 nm and 610 nm. After X-ray irradiation at room temperature in LiI crystal, I_3^- linear molecules arise situated at two anion and one cation lattice sites $(X_3^-)_{aca}$ centers. It is obvious that $(X_3^-)_{aca}$ centers are formed with the participation of radiation created V-type centers (trihalide quasi-molecules) and complementary H centers (X_2^- two-halide molecule in one anion site). Similar results were reported by Ch. Lushchik et al., for X- ray induced luminescence on pure alkali halide materials [14].

3.3. Effective atomic number (Z_{eff})

Z_{eff} values are of critical importance in radiation dosimetry calculations. If a TL material is to be used for certain application in the field of photon radiation, one of the main characteristics that must be known is the variation of response with energy. In the present work, we examine the effective atomic number of LiI crystal. Study of Z_{eff} provides conclusive information about the target with which the radiation interacts. The effective atomic number (Z_{eff}) of a TL material can be evaluated according to the following expression:

$$Z_{eff} = \sqrt[x]{a_1 Z_1^x + a_2 Z_2^x} \ldots \quad (17)$$

With

$$a_i = \frac{n_i(Z_i)}{\sum_i n_i(Z_i)}, \quad n_i = N_A Z_i$$

where, a_1, a_2,... are the fractional contents of electrons belonging to element Z_1,Z_2,... respectively, n_i is the number of electrons, in one mole, belonging to each element Z_i. N_A is the Avogadro's number. The x values are in 2.94 [32] and 3.5 [33] range. The values of Z_{eff} calculated using above calculation about 52.01 and 52.17.

3.4. Summary

In this present work, we have presented the results on the TL kinetics properties of LiI single crystal. Single crystal of LiI was successfully grown by vertical Bridgman technique. TL glow curve has revealed that has a very stable peak at 393 K with low temperature satellite peak at 343 K. The TL kinetic parameters of the as grown pure LiI crystal were calculated using various methods such as peak shape method, initial rise method, whole peak method and glow curve fitting method. Analysis of the main peak using various methods shows that the peak follows first order kinetics. The value of E is about ~1.02 eV and the S is of the order of 10^{12} s^{-1}. In order to validate the estimated kinetic parameters, synthetic TL glow curve has been generated by numerically using the experimentally determined parameters. It is found that the peak temperature for the computer generated and the experimental TL glow curves are good agreement in each other.

Acknowledgements

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