UNIVERSITY OF GAZIANTEP

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INVESTIGATION OF THE EFFECT OF HEATING RATE ON

THE DOSE DEPENDENCE OF TL GLOW CURVES OF $Li_2B_4O_7$:

Mn (TLD – 800)

M.Sc. THESIS
IN
ENGINEERING PHYSICS

BY
DLGASH H. AL-ZEEBAREE
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Investigation of the Effect of Heating Rate on the Dose Dependence of Tl Glow Curves of $Li_2B_4O_7 : Mn$ (TLD – 800)

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Supervisor

Asst. Prof. Dr. R. Güler YILDIRIM

By

Dlgash H. Al-Zeebaree
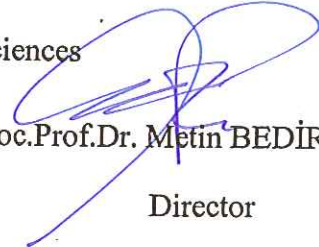
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This is to certify that we have read this thesis and that in our consensus/majority opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

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ABSTRACT

INVESTIGATION OF THE EFFECT OF HEATING RATE ON THE DOSE DEPENDENCE OF TL GLOW CURVES OF $Li_2B_4O_7$: Mn (TLD – 800)

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In this study, the effect of the heating rate on the dose dependence of TL glow curves of Li_2B4O_7: Mn (TLD-800) have been investigated by using the supralinearity function, f(D). The dose response functions have been evaluated at different linear heating rates between 1 $^oC/s$ and 20 $^oC/s$. The results of this study have showed that, the main peak shows high linear behaviour at linear heating rate of 3 $^oC/s$ and 4 $^oC/s$. Also the peak temperatures of all peaks are shifted higher temperatures and the integrated peak area of the curves decreases by about 65 %. as the heating rate increases as expected in theory because of the thermal quenching effect.

Keywords: Li_2B4O_7: Mn (TLD-800), *f(D)*, thermal quenching.

ÖZ

ISITMA HIZININ Li$_2$B$_4$O$_7$: Mn (TLD-800) KRİSTALİNİN IŞIMA EĞRİLERİ ÜZERİNE ETKİSİNİN İNCELENMESİ

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58 Sayfa

Bu çalışmada, ısıtma hızının Li$_2$B$_4$O$_7$ (TLD-800) kristalinin ışıma eğrileri üzerine etkisi doz-cevap fonksiyonu ve doğrusallık fonksiyonu f(D)kullanılarak araştırılmıştır. Doz-cevap fonksiyonu, 1 ^{o}C/s and 20 ^{o}C/s arasında lineer ısıtma hızları kullanılarak elde edilmiştir. Elde edilen sonuçlar ana dozimetrik pikin 3 ^{o}C/s ve 4 ^{o}C/s ısıtma hızlarında en yüksek doğrusallık değerine sahip olduğunu göstermiştir. Ayrıca ısıtma hızının artmasıyla toplam eğri alanı %65 oranında azalmıştır ve bu azalma teoride belirtildiği gibi termal sönümleme ile açıklanmıştır.

Anahtar Kelimeler: Li$_2$B4O$_7$: Mn (TLD-800), *f(D)*, termal sönüm.

To the of my great father. And to my mother, sisters, brothers and my wife, my son

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CHAPTER 1

INTRODUCTION

Thermoluminescence can be defined as the physical phenomenon where a solid sample takes up energy while irradiated at a given temperature, and then they release this energy as light form while heating the sample. The emitted light is recorded as intensity vs. temperature in the shape of one or more TL peaks. Then under favourable conditions, the emitted thermoluminescence light density is less the absorbed dose, using a proper standardization, we can evaluate the applied dosage in the given radiation area. The TL signs of density could reach to its maximum or the just under the TL glow peak, that are mostly proportional to each other. Furthermore, people can suitable materials in regular dosimetric applications, reproducible results in frequent measurements, linear dose dependence for the kind of radiation in question plus dose-rate independence and long time stabilization. Thermoluminescence (TL) is observed under condition of steadily increasing temperature. In the usual TL experiments, the TL systems is irradiated at room temperature (RT) and later heated through a temperature range where the luminescence is bright until a temperature level at which all the charges have been thermally excited out of their metastable levels and the luminescence completely disappears. If the light emission is plotted as a function of temperature or time the graphs is known as glow-curve [1].

The first application of the (TL) phenomena which has been known for a long time for dosimetric purposes was from Daniel et al [2]. Many natural crystals exhibit thermoluminescence properties that are suitable for TL dating. TL has become an important tool for dating of archaeological and geological materials [3]. Thermoluminescence method is a relative complex process since it involves a trap and a luminescence center. The substances like insulators or semiconductors is exposed to ionizing radiation at room or low temperature, electrons are released from the valance band to the conduction band. This leaves a hole in the valance band. Both

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types of carriers become mobile in their respective bands until they recombine or until they are trapped in lattice imperfections in the crystalline solids. These lattice imperfections play very crucial role in the TL process. The trapped electrons may remain for a long period when the crystals are stored at room temperature or they can be released due to the sufficient energy given to the electrons when the crystal is heated. These electrons may move in the crystalline solid until they recombine with suitable recombination centres that contain hole with the emission of TL light. This process of light emission by thermal stimulation from a crystalline solid after irradiations is called as "thermally stimulated process" or simply "thermoluminescence". This thermally stimulated light contains information about the trap structure of the TL material and its previous exposure to the ionizing radiations.

The thermoluminescence is a process differs from the light emitted spontaneously from a substance when it is heated to incandescence. Say over 200 $^\circ$C (or at high temperatures) a solid substance emits (infra-red) radiation of which the intensity of it increases with increasing temperature. This process is called (thermal) or (black body radiation). In thermoluminescence phenomena can be found the three essential ingredients necessary for the productions of thermoluminescence. Firstly, the material must be an insulator or a semiconductor-metal do not exhibit luminescent properties. Secondly, the material must have at some time absorbed energy during exposure to radiation. Thirdly, the luminescence emission is triggered by heating the material. In addition, there is one important property of thermoluminescence which cannot be inferred from this statement as it stands at present. It is a particular characteristic of thermoluminescence that, once heated to excite the light emission, the material cannot be made to emit thermoluminescence again by simply cooling the specimen and reheating. In order to re-exhibit luminescence the material has to be re-exposed to radiation, whereupon raising the temperature will once again produce light emission. The fundamental principles which govern the production of thermoluminescence are essentially the same as those which govern all luminescence process, and in this way thermoluminescence is merely one of a large family of luminescence phenomena [4].

When the material is heated TL material is consequently can be known during exposure to ionizing radiation absorbs some energy or stored, output energy visible light. They were excited in a different method in stimulation of luminescence that TL does not point out to thermal excitation. Simply cooling the sample and reheating it another TL material cannot conversion light. Be re-exposed to ionizing irradiation before it makes light again it must first for dosimetric applications of a TL material the storage ability makes it suitable.

The main characteristics required for making a material a good TLD (TL dosimeter) candidate are [5].

- Luminescence density is linear with absorbed dose Long period. The majority of substances, superlinearity and decay of the TL intensity at big doses the linear interval is limited. The useful range the linear dose dependence is set.

- On the incident radiation energy Low reliance of the TL response. It can be the energy dependence partially compensated by metallic filters, if necessary.

- A high TL sign per unit absorbed dose, i.e., high sensitiveness, is crucial for use in environmental radiation monitoring and personal dosimetry.

- The capability to save dosimetric data for a long time, i.e., Low fading.

- Simple TL curve (with one isolated peak).the dosimeter heating protocol is complicated, if some peaks are present.

- The luminescence spectrum must suit the maximum spectral sensitivity of photomultiplier.

- The TL dosimetry material has to be radiation resistant, mechanically tough and chemically inert.

Usually, peaks of emitted light of one or more the form of the glow curve overlap possible. The format of the glow curve especially depends especially on its spectral

response and the light sensitive tool. As well as, the shape of the TL glow curve relies on the heating rate used; even with a given tool the glow curve may look different if different filters are inserted between the detector and sample. When personnel monitoring as fast as possible to doses are evaluated, the largest number of TLD cards in the short time with high density reader systems having fast heating rates are used. Subsequently, investigations of TL dosimetric, to record the TL signs during a short time uses rates higher environmental dosimetry and the routine personnel 10 $^{\mathrm{o}}$C s^{-1}. It is familiar many changes in location, size and shape of the glow curves heating by changes in rate that the dosimetric features of many TL materials are affected [6].

Another of glow curves important characteristics is their dependence on the excitation dose, archaeological dating of ceramic samples and the applications in dosimetry, dependence on the dose in broad ranges is to a large extent desirable, but this is by no means always found to be the case. Especially, the dose dependence curve, at high doses reaches slightly to saturation value as regard to low doses, is quite common linear dose dependency, however, sometimes appears the lowest doses of dependence in superlinear (or supralinear), though quite rarely in several cases, to take into consideration the dependency upon dose rate [5] Frequently appear in nonlinearities depend the dose of thermoluminescence. Usually when there is an approach to saturation in the dose dependency contain sublinearity, in the literature supralinearity termed superlinearity. Various in the area researchers have saw the effect of supralinearity/superlinearity somewhat from two different opinions from the point of view has to do the dose with rate of change of the dose dependency function.

The other ways are related more to the applications on TL road dosimetry, geological and archaeological, situations where in extrapolation in basically they have to do with the correction where supra (super) linearity appears following like a linear range and an essential linear dose range. The word superlinearity index, *g (D)*, in the dose ranges where the growth is more than linear and when extrapolation is not the main issue. The word supralinearity index, f *(D)*, is used in cases where the feature of interest is the deviation from linearity, namely, when the correction in the extrapolation is the main issue [7].

The effective atomic number of the Lithium tetra borate ($Li_2B_4O_7$) is (Z_{ef} =7.3) and it is very close to human tissue (Z_{ef} =7.4) and it is therefore an appropriate material for (TL) dosimetry [3]. $Li_2B_4O_7$: Mn phosphor was the first material which was presented in radiation dosimetric from the lithium tetraborate [4]. But it gives low thermoluminescence sensitivity, and partly caused the emission in the (600nm) region of the spectrum, which is still far from an exemplary wavelength zone for most commercial photomultipliers (approximately 400 nm). There are a various ways to prepare these thermoluminescence materials which have subsequently been developed, but various thermoluminescence characteristics comes from different activators [5].

CHAPTER 2

MODELS AND ANALYSIS OF THERMALLY STIMULATED PROCESS

2.1 Models for Thermoluminescence

It was understood at the beginning of the nineteenth century that conductivity phenomena and luminescence were closely related (Garlick, 1958) and it became obvious that photoconductivity arose from the release of charge holds during luminescence. These concepts were essential in establishing the energy band model as a means of interpreting luminescence phenomena in a lot of phosphorus because this model is particularly useful in providing an understanding of processes which involve transportation of an electronic charge through the lattice. In this section the trapping and recombination phenomena which have been characterized till now will be used to discuss possible mechanisms for thermoluminescence [62].

The part of theoretical solids and especially with the effects of impurities and lattice irregularities TL is related to the band structure can be appear when ions of either signals move away from their original places these may call as centers, Consequently departure vacancy states ,can be interact with holds free charge and trap, instead of ions could interstitial positions of spread and smash locally the lattice geometry ideal ,lastly, can be ions impurities perturb the lattice order, because of their valances and sizes ,generates differ in their neighbor. As well as, can be interact extrinsic defects with the intrinsic ones, and after that either of them may aggregate in more complicated configured. From a viewpoint atomic can be defect characterized the number of charge holds and means of the signal, perhaps the eventual interacts with existence of excited states, to such

characterization characteristic energy for each center corresponds. Amount of energy is defined the trapped charges free when supplied to set, therefore, the center and restoringsituation   devastating of local order. Therefore it is virtual therefore to

characterize structure in term of the valence band and conduction bands, below the conduction band at various depths are represented the defects as places lied which parted from each by forbidding gap, where free charge holds of either signal can be trapped [8].

2.1.1 Randall - Wilkins model (First Order Kinetics)

In 1945, Randall and Wilkins extensively used a mathematical representation for each peak in a glow curve, starting from studies on phosphorescence. Their mathematical treatment was based on the energy band model and yields the well-known first order expression. Consider a substance containing flaws which increase to a single electron trap level, having trap depth or activation energy (E) containing (n)electrons at a time (r) and at temperature T (in Kelvin). The energy distribution of electrons within the trap will be described by Boltzmann distribution, and hence that is given freeing of trapping probability (p) by the Arrhenius equation.

Radiation energy the absorption ($h\gamma > E_g$) leads to ionization of electrons valence, Creating active holes and electrons next thermalization make release holes in the release electrons and a valence band to (transmission a) conduction band . Become trapped or the release charge holds reassemble with some other at situation directly reassemble on the quantity of energy shall freeing which can stir a resplendent center. The relaxes luminescent center because of the emission of light (returns to the ground state). The insulators and in semiconductors trapped of the charge holds a certain percentage the holes at R and the electrons at T (transmission b).

Perhaps the rate of fleeing per second, by the Arrhenius equation, given by the Arrhenius equation, have a Maxwellian distribution of thermal energies is electron in the trap.

$$p = s \, exp \left(\frac{-E}{KT} \right) \tag{2.1}$$

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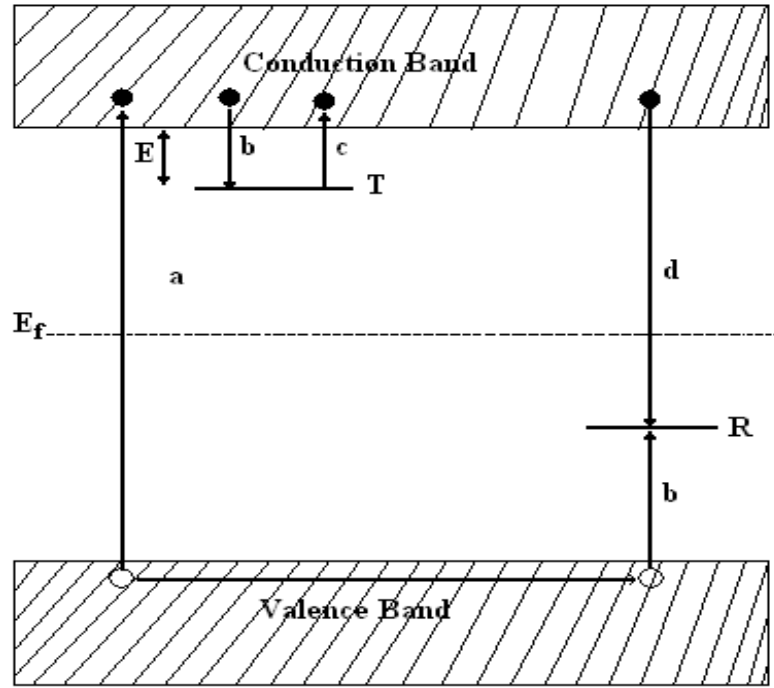
Figure 2.1 The model of the Energy band the electronic transitions the in a TL substance in accordance with the two-level model a simple (a) The obstetric of holes and electrons; (b) hole trapping and electron ; (c) electron freed because of thermal energizing; (d) Re-installation (recombination). ($\bullet$) Electrons States ($\circ$) holes show. Electron trapping is level T , center recombination is a level R , is Fermi level(E_f) . [3]

Wherever , The Boltzmann constant is (k) while attempt to escape factor having value in the order of the lattice vibration frequency or frequency factor is (s), that is 10^{12}-10^{14}s^{-1}. The rate of liberty of electrons from the trap is

$$-\left\{\frac{dn}{dt}\right\} = ns\, exp\left\{\frac{-E}{KT}\right\}$$

(2.2)

Randall and Wilkins presumed that all electrons liberated from traps undergo TSL transitions, i.e., there is no retrapping. This means that the rate of release is commensurate to the trapped charge focus , and thus termed a "first-order" reaction.

The density of the TSL glow, I(t) rely on the rate of liberation of electrons from traps and their rate of arrival at luminescence centers:

$$I(t) = -C \left\{ \frac{dn}{dt} \right\} = C \; ns \; exp \left\{ \frac{-E}{KT} \right\} \tag{2.3}$$

Wherever (C) is the constant which is related to the efficiency of luminescence

$$\beta = \frac{dT}{dt} \tag{2.4}$$

Equation (2.2) turns into,

$$\left(\frac{dn}{dT} \right) = \left(\frac{1}{\beta} \right) ns \; exp \left(\frac{-E}{KT} \right) \tag{2.5}$$

On integration, we get:

$$ln \left(\frac{n}{n_o} \right) - \int \left(\frac{1}{\beta} \right) ns \; exp \left(\frac{-E}{KT} \right) dT \tag{2.6}$$

Where the number of electrons exists in time (t_0) in the trapping is (n_0) .

In the end, substituting for n in the equation (2.3):

$$I(T) = n_o s \; exp \left\{ -\frac{E}{kT} \right\} exp \left\{ -\frac{s}{\beta} \int_{T_o}^{T} exp \left\{ -\frac{E}{kT'} \right\} dT' \right\} \tag{2.7}$$

This is the expression for the glow density I of electron trap at a single trapping level (E). It is a Randall and Wilkins expression for first order (monomolecular) kinetic. The plot of I against T is termed as glow curve. The glow curve has a distinct asymmetrical figure to be an aspect of the low temperature broader on the high temperature aspect . A condition of maximum intensity can be found by differentiating equation (2.7) regarding T and equating the derivative to zero (i.e. (dI/dT) T=T_m = 0) which yields:

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$$\frac{\beta E}{kT_m^2} = s \exp\left\{-\frac{E}{kT_m}\right\}$$ (2.8)

Where (T_m) are glow peak temperature. From equation (2.1) and (2.8) it is concluded that greater the value of E and smaller the value of s, the greater is the thermal steadiness of the trapped electrons and thus the higher is the temperature of the glow peak.

2.1.2 Garlick- Gibson model : (Second Order Kinetics)

Garlick and Gibson presented the change of this concept in 1948, which used this same model one- recombination center one-trap but who encompassed the concept of significant trapping of the released charges. This subsequently leads to the rate of the reaction being proportional to the square of the trapped charge concentration, and consequently, we have a "second-order" reaction. Here we have:

$$\frac{dn}{dt} = -n^2 s` e^{-\frac{E}{kT}}$$ (2.9)

Where, s` = s/N, and the total focus of electron traps available is (N). The equation second-order kinetics of Garlick-Gibson equation to TSL.

$$I(T) = \frac{n^2 s` \exp\left\{-\frac{E}{kT}\right\}}{\left[1 + \frac{n_o s`}{\beta} \int_{T_o}^{T} \exp\left\{-\frac{E}{kT'}\right\} dT\right]^2}$$ (2.10)

The major characteristics of this equation are that the glow is approximately symmetrical, the curve high temperature half marginally broader than the low temperature half seems from the consider of the fact the second order reaction, (Bos, 2001) concentricity are trapped great of free electrons before they In this method of

growing to a lag in the TSL spreading and in emissions through broader range temperature. A TL equation for second-order kinetics (Garlick–Gibson), that is almost symmetrical, high temperature the curve half marginally wider than at low temperatures half seems to consider of truth the second order reaction concentricity great second-order reaction freeing electrons are trapped in this method of the increasing delays in the luminescence emission and pervasion of emission broader emission through a wide temperature extent. A primary concentricity (n_0) seems here not only multiplier constant as in the first-order situation, diversity even dose level difference of the whole curve change a shape. Appears be (T_m) drops about (n_0) rises. That it may can be retraped [14].

2.1.3 May – Partridge model (General Order Kinetics)

Derivative forms of TSL equation Randall-Wilkins and Garlick-Gibson from the use of certain hypotheses concerning the relative values trapping and recombination probabilities. But while those assumptions no hold, the proportionality the TSL peak no first- nor the second order kinetics. In 1964, made Partridge and May the next experimental for general order TSL kinetic:

$$I(t) = -\frac{dn}{dt} = n^b s^{'} \exp\left\{-\frac{E}{kT}\right\} \tag{2.11}$$

Wherever, (s`), And after from ($m^{3(b-1)}$ s^{-1}) or (b) Is general-order expressions as defined or its uncertainty (1 and 2) . The equation Integration the above $b \neq 1$ yields:

$$I(T) = s``n_0 \exp\left\{-\frac{E}{kT}\right\}\left[1 + \frac{s``(b-1)}{\beta}\int_{T_0}^{T}\exp\left\{-\frac{E}{kT}\right\}dT\right]^{-\frac{b}{b-1}} \tag{2.12}$$

Wherever, (s`` = s`$n_0^{b-1)}$) with unit (s$^{-1}$). The above equation contains the second order condition (b=2) and lowers to the equation (7) while (b $\rightarrow$1). It is worth to notice that the dimensions of (s`) are of (m $^{3(b-1)}$ s$^{-1)}$ until the distance alteration of who means of kinetics (b). Consequently, it is tough to interpret(s`) physical.

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The theoretical mechanism argued above is connected only to electrons trapped on a single trapping level. Really many different phosphors trapping levels will be present, each one because of a particular lattice defect or complex of defects. Each trapping level will increase to an associated glow peak maximum, which might or might not be resolved during readout. The area and peak height of each glow peak relies on a number of associated electron traps present. This in sequence relies on the number of lattice defects and, for real phosphors, on the type and amount of impurity atoms present, furthermore on the thermal history and treatment of the material.

2.1.4 Advanced Models

Display and phenomenon TL format the glow peak difference in the framework of the dose and heating rate is the one trap–one center model is demonstrating features. But, is no existence TL substance recognized precisely is characterized by the simple model. Has no meaning in the simple model this does not make. Conversely, Chen and McKeever the book is referred the reader, in clarification of many advantages differences models in one trap–one center which can be considered, more realistic it is tricky to speak all the progressed models in particular. [5]. for treatment a quantitative and deeper, Herein, in order that obtain information mentioned only some models around the phenomenon TL material complexity in a real.

Generally, TL material real considers one or more trap a single electron. When the sample is heated and some traps on the temperature range that inert. When disconnected a thermally for trap which can be from during radiation filled with electrons but Trap depth is much larger than the trap of Active such that only electrons trapped in the trap of active (AT) when heated sample is freed from the shallow trap (ST) (see Fig.2.2 (a)) . Deeper level electron trapping are unaffected and in result. In this profound an electron the trap (indicated the Figure.2.2 (a) DET) of thought that disconnect the heat. However, the presence is an impact on the trapping filling and after that in the form of from glow peak [16]. In section 2.1.1 assume that through heating release electrons trapped while settled holes trapped in recombination center. A special the release holes and unite in the center through a

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steady heating of electrons mathematically identical. But if you are editing both electrons and holes from trapping state will change in time heat and the same period from the center are also as places for recombination reason holes are freed electrons heat release heat (see Fig.2.2 (b))

Differential equations drafted new. Display a TL glow curve in analysis of the complicated kinetic model which conserves the simple Randall–Wilkins (Eqn. (2.7)) Or Garlick–Gibson (Eqn. (2.8)). Shape, according upon the selected values of the parameters. However, the (E) and s values used in Eqn. (2.7) and Eqn. (2.8) to this model appropriate on this complicated kinetic to get them we need much explanation. Another operation which may happen is a recombination without a transmission of the electron into the conduction band (Fig.2.2(c)) Herein the electron is thermally activated for1 triggered state from which a transmission in the recombination center is allowed. Seems to be the trapping must be in the close of a center.
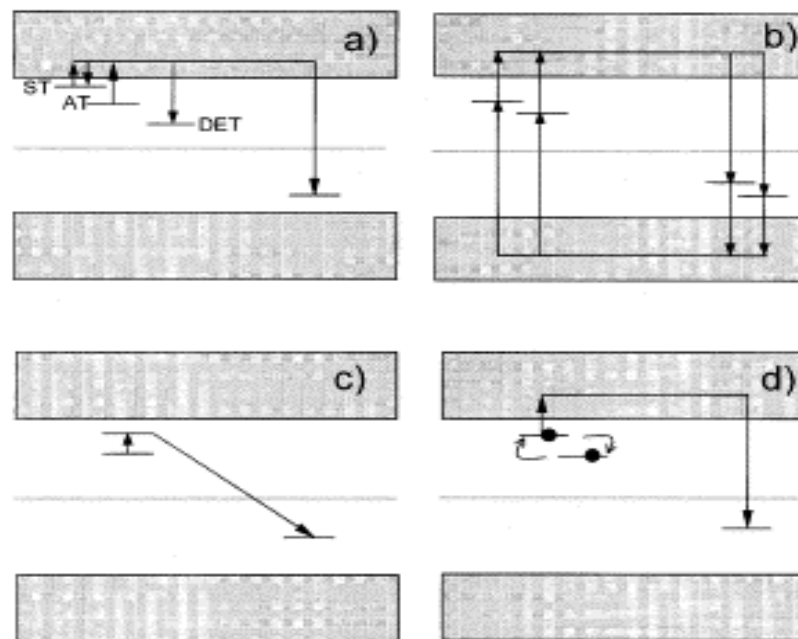
Figure 2.2 Advanced models distinction the thermally activated freed trapped charged holds It Comes: (a) a active trap (AT), a deep electron trap (DET), and a shallow trap (ST); (b) two effective traps and two recombination centers; (c) Local transmission; (d) defect interaction (trapping center interacts with another defect) [11].

May be the possibility of transmission depends largely on the extent between the two center. Could be under specific assumptions stems from the term density TL [16] that

has the same shape as Eqn. (2.7) but with(s) replaced by a quantity linked to the probability of recombination. This means that governed under Transformations by first-order kinetics.

Ultimately, we shall indicate the probability that has trapped the electrons is unstable but is participating in the process of interaction with another defect (Fig.2.2 (d)). Can be result that at low temperature the trap changed when the concentrate trapped electron is stable. Participating the electrons in two processes at higher temperatures: In addendum, (Piters and Bos) [17] have defect reactions inserted to the rate equations and glow curves simulated. It seems that the simulated glow curves can be very well fitted by Eqn. (2. 7). It is obvious that the fitting parameters have no the simple meaning of trap depth and escape frequency.

2.2 Thermoluminescence Analysis

Mechanism of systematic analysis of TL glow curves of providing the perfect summary.
First of all, Chen and McKeever [1], by following these steps:

(1) By optimizing the thermal contact between the sample and the heating element, a during the TL glow peak is a precise guarantee that the temperature measurement during.

(2) By employing a thermal cleaning process, remove the probability of close overlapping peaks. Corrected using theoretical considerations, if present, thermal quenching influences should also be accounted. Furthermore, during the TL glow curve and the study of emission spectra during supply precious data on the TL process.

(3) Which characterize glow peak secluded by valuation three parameters (E, s, and b) other methods for measuring Analysis. Over methods based on only a few points on the glow curve must be using methods separated whole glow peak. For example samples irradiated at various doses by research.

(4) A perfect convention between many approaches of analysis it is decisive getting. Ought to be testing any variations in additional detail.

(5) Earn additional data about the processes implicated and in system to solve variations , for glow peaks measured beneath unlike heating rates must be performed the analysis, many radiation doses, bulk samples and powdered, etc.

(6) Based on various physical processes acquired additional data designated using empirical approximation , such as isothermal decay techniques, excitation and emission spectra, simultaneous TL-TSC (thermally stimulated current) measurements and dose dependence measurements . [1]

2.3 Methods of Analysis

2.3.1 Computerized Glow Curve Deconvolution (CGCD)

Computer Glow Curve Deconvolution (CGCD) is one of the most important methods to determine trapping parameters from TL glow curves. This method has the advantage over experimental methods in that they can be used in largely overlapping-peak glow curves without resorting to heat treatment. In this model for analyzing these TL glow curves supposes a set of hole traps (recombination center) and a set of discrete electron traps .The entire glow curves of unlike heating rates are exposed to CGCD in the kinetic formalism. The glow peaks following appropriate and computerization dependent the general order kinetic[12] including the eminent first order [15] and Kirsh [22], the amendment introduction here is the chosen of the peak temperature within $\pm 2°C$.

Moreover, the minimum value of the origin means square perversions. In the CGCD program, the general order kinetics for the TL intensity I (T) depend on the absolute temperature T as developed by Chen is given as:

$$I(T) = -\frac{dn}{dt} = s`\, nb\ \exp\left(-\frac{E}{KT}\right) \qquad (2.13)$$

Where, (no) the initial focus of trapped electrons, s $(= s'n_0^{b-1})$ is the frequency factor,

(E) The activation energy, (b) the kinetic order, and (k) is the Boltzmann's constant.

For a linear heating profile:

$$T = To + \beta t \tag{2.14}$$

Wherever is the initial temperature (T) the temperature at the time(t) , and (β) the linear heating rate.   We get Randall and Wilkins equation, From Eqs (2.1) and (2.2), (b= 1)

$$I(T) = n_o s \exp\left\{-\frac{E}{kT}\right\} \exp\left\{-\frac{s}{\beta}\int_{T_o}^{T}\exp\left\{-\frac{E}{kT'}\right\}dT`\right\} \tag{2.15}$$

And general order kinetic equations (b ≠ 1)

$$I(T) = n_0 s \exp\left\{-\frac{E}{kT}\right\}\left[1+\frac{s(b-1)}{\beta}\int_{T_0}^{T}\exp\left\{-\frac{E}{kT}\right\}dT`\right]^{-\frac{b}{b-1}} \tag{2.16}$$

However, they succeed integration by parts over a limited number of terms, Chen and Winer[17] approximated the integral as:

$$e^{\int -\frac{E}{kT}dT`} \simeq \frac{kT^2}{E}\ e^{\left\{\left(-\frac{E}{KT}\right)\left(1-2\frac{E}{KT}\right)\right\}} \tag{2.17}$$

Then, the TL density may be written on the general form as:

$$I(T) = I_m\ b^{\frac{b}{b-1}}\ e^{\frac{E}{kT}}\ \left(\frac{T-T_m}{T_m}\right)x\left[(b-1)\left\{1-2\frac{KT}{E}\right\}x\left(\frac{T-T_m}{T_m}\right)x\left(\frac{T^2}{T^2 m}\right)e^{\frac{E}{kT}}\left(\frac{T-T_m}{T_m}\right)\right] \tag{2.18}$$

As, (I_m) and (T_m) are the maximum intensity and peak temperature, respectively.

Additionally, one may follow the frequency factor (s) as well:

$$s = \left(\frac{\beta E}{kT_m^2}\right) \left\{ \frac{1}{1 + \frac{(b-1)2\,kT_m}{E}} \right\} e^{\left(\frac{E}{kT}\right)} \qquad (2.19)$$

But Chen's general order kinetic transient does permit the order of kinetic (b) behind two (i.e. b > 2), but traditional analysis mentioned in the literature [23] shows $1 \le b \le 2$.

Measured TL glow curve retested best fit using χ^2-test of normality[24-27]. Whose measurement goodness of appropriate around normality of the error distribution. As a cross check (FOM) [28,29] was also calculated. To calculate to avert error in the kinetic standards report by CGCD is the active heating rate (β_{eff}) between the heating element and the thermoluminescent sample through the TL readings in the reader (using contact heating) is least important point. Was used as a simple way of heating correction reduce careful active heating rate of the TL sample by using the equation to avert this affair:

$$\beta_{eff} = \frac{\left(T_g - T_o - \Delta T\right)}{T_g - T_o}\,\beta = \frac{(T_m - T_o)}{T_g - T_o}\,\beta \qquad (2.20)$$

Wherever $(\Delta T = T_g - T_m, \;\; T_g)$, $(T_o$) is room temperature (25°C) , Note Peak temperature (K) and (T_m) is a the genuine temperature peak (i.e., with thermal lag correction).

2.3.2 Various Heating Rate (VHR)

From the maximum stipulation of the first-order kinetics Eq. (2.3), Can be as it is written:

$$\frac{\beta E}{kT_m^2} = s\,exp\left(\frac{-E}{KT_m}\right) \qquad (2.21)$$

Or

$$ln\left(\frac{\beta}{kT_m^2}\right) = ln(sK/E) - \left(\frac{E}{KT_m}\right) \qquad (2.22)$$

A plot of In (T_m^2/β) versus ($1/T_m$). Should yield a straight line with a straight line with a slope (E/k) then E is calculated[16]. The value of (s) is studied by induction of the straight line to ($1/T_m$) = 0. As such is separate of the order of kinetics and usable for any heating rate[31]. Similarly in the situation of CGCD, last significant point that was taken in mind in the appraisal of the trapping parameters by VHR style is the temperature was between the phosphor and the heating element through the TL read out in a reader using contact heating. The thermal lag between the phosphor and the heating element through the TL readout in the reader is studied by the style developed by Kitis and Tuyn [30]. The determined temperature accurate heating rates peaks differed by using the equation:

$$T_m^j = T_m^j - ln\left(\frac{\beta_i}{\beta_j}\right) \qquad (2.23)$$

Where (T_m^j) and (T_m^j) are the extreme temperatures of the glow peak with heating rates (β_i) and (β_i). Accordingly, (c=ln(sK/E)) is a constant which is generally rated by using two very low heating rates preferably below 1°Cs^{-1} in that the temperature lag can be considered to be negligible[32]. In this work, the heating rates 0.5° Cs^{-1} and 1.0°Cs^{-1} were used to decide the constant (c).

2.3.3 Peak Shape Methods

Thermoluminescence phenomena basic expectations theory expressions for the special session of the experimental peaks glow TL. The theory of the first order kinetics is derived by Randall Wilkins [1]. Garlick and Gibson are derived for the kinetic theory of second order [2]. However, the kinetic system first or second mismatch there is much they glow peaks shape.These kinetic medium orders of May and Partridge and Suggest a theory proposed purely an empirical description [3]. In basic research and in TL applications , at TL glow-peak shape plays an important role . Appropriate methods for account he traps standards of distinguished distinct

energy levels during the crystal in the condition of basic TL search it is the basis . Measurements of a little point on the glow-peak these methods are based, as clarification in Fig 2.3. Chen [10] present an overall systematic and summary all found before methods deriving the coefficients of the term for first and second order kinetics case of intermediate kinetic orders too and in the case the peak shape method coefficients were of Chen [11]. By:

(i) Coefficients for first order kinetics it is calculating.

(ii) Coefficients for second-order kinetics it is calculating .

(iii) Linear interpolation way to gain it is using Expressions for the intermediate kinetic.

As a function of orders the symmetry factor (μ_g), which was found to be between 0.42 and 0.52 for first and second order kinetics. Lately, Kitis and Pagonis [12] provide the theoretical basis, and phrases on the kinetics medium Chen and labor public order kinetics of experimental model, a row, TL theory in the current situation, characterized by the region of medium the order kinetics of experimental models. [13] Glow peaks derived from model phenomena many of them (OTOR) and one of the traps and one model center recombination in this study capabilities kinetic public order and mobility mixed system to fit artificial, (IMTS) interactive multi-system trap (NMTS) non interactive multi-trap system. Glow peaks so much more successful than public order or kinetic fit (NMTS) and (IMTS) discovered kinetic mixed system, and in addition to that, the kinetic mixed system fits bad TL peaks model provided by the (OTOR). Sunta et. [13] Attributed to the fact that the model (OTOR) very simple and unrealistic physically this result. Clearly superior to public order in the kinetic properties of the tops of the experimental TL glow was the net result kinetic mixed system. New peak shape ways for evaluating the activation energy (E) The purpose of the current work ,meaningful models of mixed order kinetics which are based on the physical. A assess revitalizes energy (E) methods used the peak shape theory (TL), are depending on first and second order kinetics equations .The peak form of the middle kinetic orders depends on the empirical theory of general order kinetics . We derive peak shape methods depends on the physical meaningful mixed order kinetics model in current work , peak shape

methods existing in the TL literature be verified the derived equations are compared and their precision with another. Were the methods employed shape the peak in this work were those of Braner [10] , Chen [11] and Lushchik [9]. The glow peak parameters required for these techniques are:.

Width of methods for the report of the kinetic standards of TLDS

Display with temperature ranges half basement $(\tau),$ the high temperature half display (δ) , the full display of half maximum (ω) , and the so-called geometry factor (μ_g). The order of kinetics(b) can be evaluated by form expressions . Chen [19] detect that (μ_g) is not ticklish to changes in (E) and(s) , however its amendments to the order of kinetics (b) . illustrated in ranges of(μ_g) differs from (0.42)for b=1 to(0.52)for (b=2) In the case of linear heating In the case of linear heating (Figure 2.4).

$$\delta = T_2 - T_m \tag{2.24}$$

$$\tau = T_m - T_2 \tag{2.25}$$

$$\omega = T_2 - T_1 \tag{2.26}$$

$$\mu g = \frac{\delta}{\omega} \tag{2.27}$$
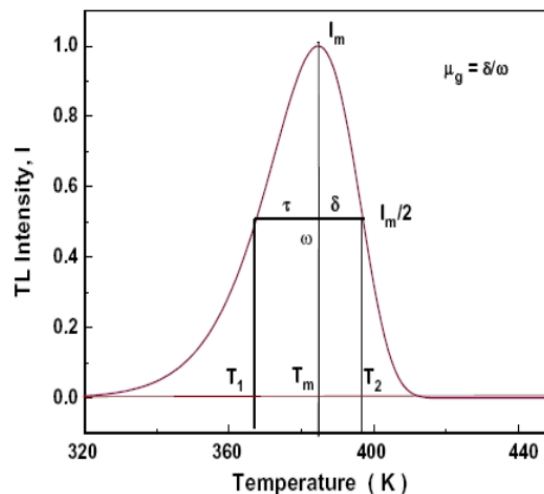
Figure 2.3 The distinguishing points on a TL glow-peak, which state the peak-shape parameters.[22].
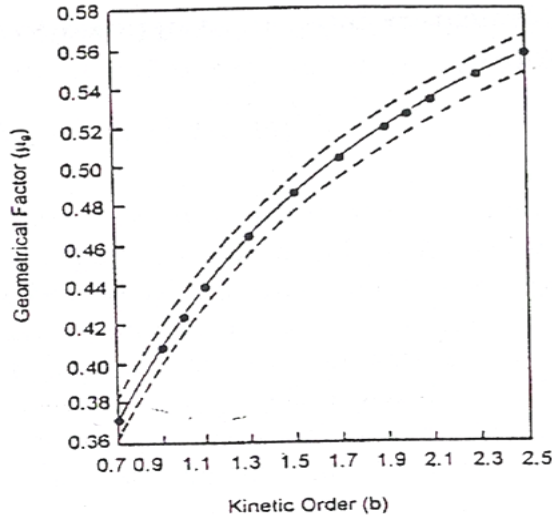
Figure 2.4 Shape expressions as a function of the given order Geometrical factor, μg [7].

The first peak shape method was developed by Grossweiner [45], later Chen [43] modified Halperin and Braner's equation [46] for calculating E values

$$E_\tau = \left[1.51 + 3(\mu_g - 0.42)\right]\frac{kT_m^2}{\tau} - \left[1.58 + 4.2(\mu_g - 0.42)\right]2kT_m$$

$$E_\delta = \left[0.976 + 7.3(\mu_g - 0.42)\right]\frac{kT_m^2}{\delta} \tag{2.28}$$

$$E_\omega = \left[2.52 + 10.2(\mu_g - 0.42)\right]\frac{kT_m^2}{\omega} - 2kT_m$$

Next fixing in the order of kinetics and the activation energy ,the next parameter used which the frequency factor (s) can be noted that this expression in the general order kinetic called as pre-exponential factor, and evaluation for general and first order kinetics respectively.

$$s = \frac{\beta E}{kT_m^2}\ \exp\left[\frac{E}{kT_m}\right]$$

$$s = \frac{\beta E}{kT_m^2}\left[\exp(-\frac{E}{kT_m})(1 + (b-1)\frac{2kT_m}{E})\right]^{\frac{b}{b-1}} \tag{2.29}$$

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Moreover, have determined Chen form way peak kinetic parameters as by Gartia, Mazumdar and Singh [25] of road peak and the demands of the kinetics order. The method uses any three points of a peak. According for this way it is possible to calculate energizing energy the following.

$$E_a = \frac{CkT_m^2}{\left|T_x - T_y\right|} + DkT_m \tag{2.30}$$

Where $\left|T_x - T_y\right| = \tau, \delta, \omega$. The factor ($C$) and ($D$) are found using the way of least squares for various orders of kinetics(b) in the range from 0.7 to 2.5 and for $x = 1/2$, 2/3, 4/3. For a specific value of x the coefficients consequences to be dependent on (b) and then can be expressed as a quadratic function of (b) itself. So that, the former equation can be rewritten as:

$$E_a = \frac{(C_0 + C_1 + C_2 b^2)kT_m^2}{\left|T_x - T_y\right|} + (D_0 + D_1 b + D_2 b^2)kT_m \tag{2.31}$$

CHAPTER 3

TL DOSE RESPONSE MODELS

3.1 Introduction

Great deal of practical applications in thermoluminesecence (TL) is about the dating .
And areas dosimetry of radiation protection That transmits signals to a dose of TL
substances the dependence of the Thermoluminescence . Has mentioned the researcher
several examples from this chapter of used theoretical modules which used , to clarify
the thermoluminescence of a diversity of substances against the dose response.

Exhibited by several materials nonlinear responses of this chapter Provides a survey
reported in the first section , to describe these nonlinear the terms used to describe.
Competition between traps shows how during irradiation can be the TL dose- response
result in performance to superlinear , based on in existence from contending traps of
model from a category of kinetic models , this is the second part.

In this third cares section is significance during exciting and used heating phase TL,
describes used case the most complex phenomenon in the competition.   For an example
of indicator superlinearity g(D) and the supralinearity index f (D) The quantities f (D)
and g(D) knowledge in next branch . Is mentioned pilot from TL against dose curves
in the exercise of the fourth in this chapter, and the latter.

3.2 Overview of Nonlinear Dose -Response of TL Materials and Terminology

Can be found dose response observed experimental nonlinear of TL materials to
radiation are offered some essential concepts and terminology are offered relevant in this
section , the theoretical aspects broad review diverse can be found notional respects of

the sample given in this branch , for example [30] , Through McKeever and Chen reviewed, in material [63]. Can be expressed dose response non-linear displayed many large TL materials form mathematics Equations:

$$I_{max} = aD^k \qquad (3.1)$$

Where (*k*) are constants,(*D*) about irradiation the dosage , (*I*_{max}) A TL maximum density (integral the TL). Although (*I*_{max}) Plan as a function from dosage (*D*) Register range give this equation a straight-line with a slope k can be   greater anything discovered for example, Chen and Halperin [64] , Chen and McKeever which offered dose response A slope (*k*) amidst 2 and 3 at specific Waves of UV irradiated diamonds exhibited [65], Recommended using the term superlinearity from derivative for the dosage increment of the TL reply   function   ,   term superlinearity to depict this additional than linear dosage- response و these authors recommended. If the sign measurement a TL S(D), An increase in derivative *S(D)* , About by reality the second derivative $(\frac{d^2S}{dD^2} > 0)$. Cases where$(\frac{d^2S}{dD^2} < 0)$ of feature as sublinear, and condition wherever $\frac{d^2S}{dD^2} = 0$ are feature   as a linear dose response. defined a termed these authors the following superlinearity index *g(D)*:

$$g(D) = \left[\frac{D\,s``(D)}{s`(D)}\right] + 1 \qquad (3.2)$$

As long as S`(D) > 0, a value of g(D) > 1 signifies superlinearity, while a value of g(D) = 1 denotes a linear dose response, and g(D) < 1 signifies sublinearity. In the particular case where S(D) = α D^k +β , one obtains g(D)=k . Which can fit the obtained curves dose response TL experimentally needs the knowledge of the application of the analytical the equation (3.2), Otherwise, this equation cannot be applied to the experimental data.

A dependence explain a linear dose in some ranges low dose several TL materials . While approaching saturation by of a superlinear dose range and by a sublinear range. For example by Cameron et al, as reported [66] , gamma-irradiated LiF is known to display such a behavior . Saturation behavior can be illustrated within the

structure of TL models- Based on the rivalry between traps during the excitation of the sample this kind of linearsuperlinear. A typical example is given to TL model based on competition by stirring in Exercise 3.2

Some authors Chen and McKeever [67] , This particular dose response nonlinear suggested using the term supralinearity, Disposition quantified by introducing the following dimensions function termed the supralinearity index or dose response function f(D):

$$f\ (D) = [S(D)/D]\ /\ [S(D_1)/D_1] \tag{3.3}$$

Where a normalization dose in the early linear range is (*D*). The values of *f (D) > 1* point to values of *S(D)* Above a linear early. In short , A measure of the rate of change of the dose response it is called superlinearity , and is described quantitatively by the superlinearity index *g(D).* And it is mostly used in applications TL dating and dosimetry.

Recommended that might be explained the superlinear behavior of some materials might be explained by competition , Rodine and Land [68] , instead of a contest during the excitation stage . Kristiapoller et al [69] , the reliance dose of the TL reply may be quadratic in nature . Was developed as a mathematical formulation of TL models of this type , on frame of such models .

That when competition especially the main assumption occurs during the irradiation stage, Chen and Fogel [70] , discussed some of the disadvantages of these two separate approaches , no competition takes place during the heating phase, and versa conversely. Competition during heating and competition during excitation modes a model that combines the characteristics of the two models was these authors developed .

Based on both competition approaches is given in Exercise 3.3. Chen and Fogel [70] and A typical example of the TL model .

3.3 The Filling of Traps in Crystals During Irradiation

Write rate equations relevant to the TL model shown in Figure 3.1 [71], one recombination center while the model consists of one trapping state . The allowed electron and hole transitions from the conduction and the valence band is arrows indicate , As shown in Figure 3.1.

The trap is distinguished by whole concentration (N) and by immediate electron occupancy $n(t)$ in the crystal. The recombination center has instantaneous hole occupancy $n_h(t)$ and total concentration N_h in the crystal. The functions $n_c(t)$ and $n_v(t)$ representing the instantaneous concentration of free electrons in the conduction band and free holes in the valence band correspondingly . During the irradiation process are [71] , the equations describing the rate of change of the functions $n(t)$, $n_h(t)$, $n_c(t)$ and $n_v(t)$

$$\frac{dn}{dt} = nc(N - n)A \tag{3.4}$$

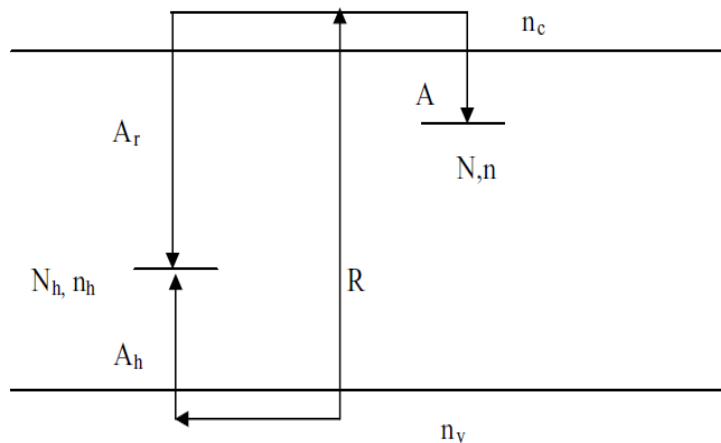Overview of Nonlinear Dose Response of TL Materials and Terminology

Figure 3.1 Kinetic model for the filling of traps during crystal irradiation.

$$\frac{dn_v}{dt} = R - n_v\,(N_h\, -\, n_h\,)A_h \tag{3.5}$$

$$\frac{dn_h}{dt} = n_v\,(N_h\, -\, n_h\,)A_h\, -\, n_c\,n_h\, -\, A_r \tag{3.6}$$

$$\frac{dn_c}{dt} + \frac{dn}{dt} = \frac{dn_h}{dt} + \frac{dn_v}{dt} \tag{3.7}$$

That electrons in the conduction band can be trapped into the electron trap the first equation expresses mathematically the reality . A about the explanation of which are formed free holes in the valence band during the excitation at a constant rate(R) , is about the second equation , and these holes can also be trapped from the valence band into the recombination center as signified by the term$-\, n_v\,(N_h\, -\, n_h)\,A_h$. The reality that the concentration of holes in the recombination center is altered by either trapping electrons from the conduction band (term$-\,n_c\,n_h\,A_r$), or by trapping holes from the valence band (term $n_v\,(N_h\, -\, n_h)\,A_h$) is expressed in the third equation. While the last equation (3.7) the total charge deals with the preservation in the crystal, being equal to the total instant concentration of electrons with the left-hand side , and total concentration of holes in the crystal at any time(t) the right hand side . The parameters in the above expressions are as follows:

A = transfer probability coefficient of electrons into the trap ($cm^3\,s^{-1}$), R = fixed rate of production of electron–hole pairs per cm^3 per second ($cm^{-3}\,s^{-1}$), n = instant focus of electrons in the electron trap at time t (cm^{-3}) , A_r = recombination probability coefficient of electrons from the (CB) into the recombination center ($cm^3\,s^{-1}$), N = total focus of electron traps in the crystal (cm^{-3}) , A_h = trapping probability coefficient of holes from the

(VB)into the recombination center ($cm^3\,s^{-1}$) , n_c = instant focus of electrons in the conduction band (cm^{-3}) , (N − n) = instant focus of empty main traps available at time t , n_h = instant focus of holes in the recombination center (cm^{-3}) , N_h = sum focus of holes in the crystal (cm^{-3}) ,n_v = instant focus of holes in the valence band (cm^{-3}) .

3.4 Competition During Excitation Model

Kinetic rate equations related to the TL model shown in Figure 3.2 [72]. The model consists of two electrons trapping states distinct by entire concentrations N_1 and N_2, and by instant occupancies $n_1(t)$ and $n_2(t)$, respectively. The is considered first trap to be the one responsible for TL , and as the competitor the second trap is denoted . Instant hole occupancy p(t) with the model also has a recombination center .

Into the conduction band During the irradiation method the electrons are raised from the valence band , and can be trapped into either N_1 or N_2, with the two traps contending for the electrons as shown in Figure 3.2 . The kinetic equations for this model are [72]

$$\frac{dn_1}{dt} = A_1(N_1 - n_1)n_c \tag{3.8}$$

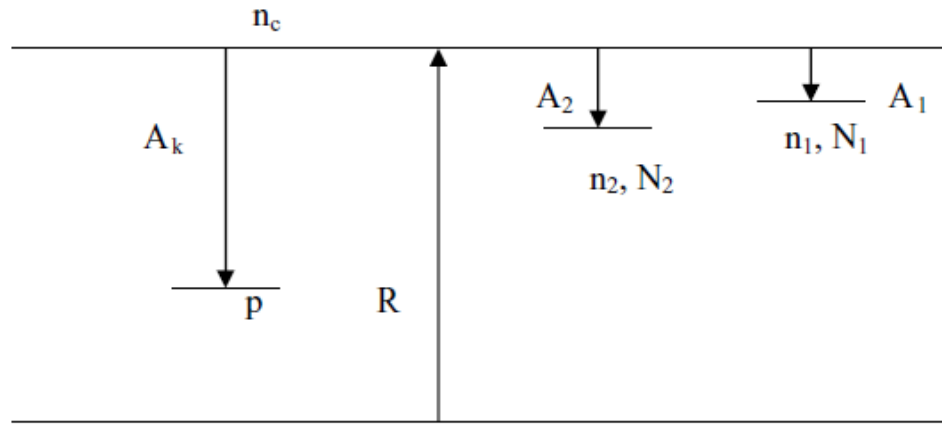$$\frac{dn_2}{dt} = 2(N_2 - n_2)n_c \tag{3.9}$$

Figure 3.2 The competition during excitation model.

$$\frac{dn_c}{dt} = R - \frac{dn_1}{dt} - \frac{dn_2}{dt} - A_k \, n_c \, p \tag{3.10}$$

$$p = n_1 + n_2 + n_c \tag{3.11}$$

The first two equations tackled mathematically can be the fact that electrons in the conduction band trapped into either the major or into the trap competitor . about the fact that the electrons in the conduction band are created by the constant excitation rate R , and can also be trapped into either of the two traps (terms $-dn1dt$ and $-dn2 dt$), or into the recombination center (term $-Aknc$ p) is the third equation . As discussed in the book by Chen and McKeever [30], dealt with the preservation of the entire charge in the crystal the last equation (3.11) , being equal to the concentration of holes trapped in the recombination center with the left-hand side , and representing the total concentration of electrons in the crystal at any moment t , the right-hand side . The last is based equation on the hypothesis that the concentration of free holes in the valence band can be ignored as compared with the amassed concentration of holes p(t).

The parameters in the above expressions are as follows:

A_1 = coefficient probability the major trap transference (m^3 s^{-1}), n_c = the focus instantaneous (CB) electrons (m^{-3}) , n_2 = focus in the emulator of electrons immediate trap (m^{-3}),A_2 = the emulator trap transference probability coefficient of electrons (m^3 s^{-1}) ,n_1 = immediate focus of electrons in the major trap at time t (m^{-3}) , N_1 = sum focus of major traps in the crystal (m−3), ($N_1 - n_1$) =instantaneous concentration of empty major traps available at time t , R = fixed rate of production of electron–hole pairs per m^3 per second (m^3 s^{-1}) , p = instantaneous focus of holes in the recombination center (m^{-3}) , N_2 = total concentration of competitor traps in the crystal (m−3) , $A_{k=}$ transference probability coefficient of electrons in the conduction band into recombination center (m^3 s^{-1}).

3.5 Superlinearity Model with Competition During Both Excitation and Heating

Figure 3.3 shows [70] , write kinetic rate equations related to the TL mode . The model has two trapping state features concentration of N_1 and N_2, and by moment occupancies n_1(t) and n_2(t) respectively. Regarded the first trap to be the one responsible for TL , as the competitor denoted second trap. Consists also the model of a recombination center , At the moment occupancy m(t) and total focus of hole

traps presented by M. Should have the simulation three stages: an intermediate relaxed stage, the heating (measurement of TL) stage, and the irradiation process.

The electrons are raised during the irradiation process from the valence band into the conduction band and can be trapped into either N_1 or N_2, with the two traps competing for the electrons. These electrons in the conduction band can
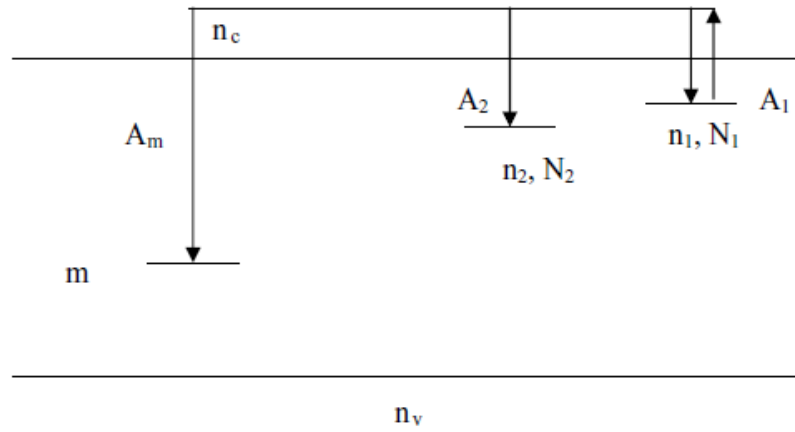
Figure 3.3 Model with competition during both excitation and heating: heating stage.

Too recombine with holes in the in the recombination center. At one time , an egalitarian number of holes are generated in the valence band by the irradiation execution . These holes can be trapped directly into the recombination center high the hole possess m(t). The kinetic equations for the agitation phase in this model are [70]:

$$\frac{dn_1}{dt} = A_1(N_1 - n_1)n_c \tag{2.12}$$

$$\frac{dn_2}{dt} = A_2(N_2 - n_2)n_c \tag{3.13}$$

$$\frac{dm}{dt} = A_n\, n_v\, (M - m) - A_m\, m\, n_c \tag{3.13}$$

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$$\frac{dm}{dt} = R - A_n\, n_v\, (M - m) \tag{3.14}$$

$$\frac{dn_c}{dt} + \frac{dn_1}{dt} + \frac{dn_2}{dt} = \frac{dm}{dt} + \frac{dn_v}{dt} \tag{3.15}$$

The fact that electrons in the conveyance band can be trapped to each the major or the emulator trap was an edifice mathematically in the first two equations state . Is tackled in the third equation . The fact that the number of holes in the recombination center is change by each trapping additional holes from the valence band (term Annv(M − m)), or by trapping electrons from the conduction band (term−Ammnc) .

The fourth equation about the fact that holes are formed permanently in the valence band by the agitation rate R, but they are too constriction in the recombination center (Term −Annv(M − m)). End , the keeping of the sum charge in the crystal, a left-hand part presence equivalent to the sum rate of change of the focus of holes , and the right-hand part presence equivalent to the sum rate of change of the focus of electrons in the crystal about in the last equation. The parameters in the above expressions are as follows:

A_1 = transmission probability degree of electrons into the major trap ($m^3\ s^{-1}$) , A_2 = transmission probability degree of electrons into the emulator trap ($m^3\ s^{-1}$) , A_m = transmission probability coefficient of electrons from the (CB) into the recombination center ($m^3\ s^{-1}$) , A_n = capture probability coefficient of holes from the (VB) into the

recombination center ($m^3\ s^{-1}$) , n_1 = instant focus of electrons in the major trap at time t (m^{-3}) , N_1 = total focus of major traps in the crystal (m^{-3}) , $(N_1 - n_1)$ = instant focus of empty major traps available at time t , n_2 = instant focus of electrons in the emulator trap (m^{-3}) , N_2 = total concentration of competitor traps in the crystal (m^{-3}) , n_c = instant focus of electrons in the(CB) (m−3) , n_v = instant focus of holes in the (VB) (m^{-3}) , R = fixed rate of product of electron–hole pairs per m^3 per second ($m^{-3}\ s^{-1}$) , m = Moment focus of holes in the center recombination (m^{-3}) , M = gross focus holes in the crystal (m^{-3}) .

3.6 The f (D) Function

Figure 3.4 , offers the conspiracy y(D) against D obtained of the data. The empirical data be was Installed by the equation (3.17):

$$y(D) = A(1 - e^{-BD}) - CD\, e^{-BD} \qquad\qquad (3.17)$$

Terms D is the provides dosage , A   is the TL reply at the fullness level (4844 a.u.),

$C = 12.93\ \mathrm{Gy}^{-1.}$ Or $B = 0.291 \times 10^{-2}\ \mathrm{Gy}^{-1}$ The objective of this practice is to research the functions f (D) and decide for which doses the TL reply is linear, superlinear, supralinear, or sublinear. The supralinearity f (D) in case where the used chief interest is in the amount of deviation from linearity, i.e. If indicator the TL a below or above the area linear extrapolated ,and procedure reforms if necessary
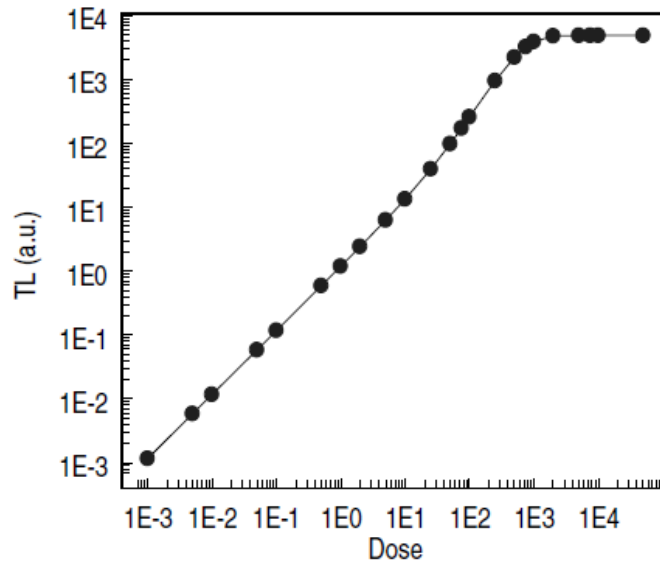

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Figure 3.4 Conspiracy of the TL against response - dose.

When know indicator the supralinearity f (D) :

$$f (D) = \frac{\left[\dfrac{y(D)}{D}\right]}{\left[\dfrac{y(D_1)}{D}\right]} \qquad\qquad (3.18)$$

f (D) < 1 way a sublinear area , f (D) ~ 0 ways the area fullness and f (D) > 1 indicates the area supralinearity.

It can be outlining the general advantages about the TL versus dosage demeanor as the

next :

- if $y``(D) > 0$, $y`(D)$ however $y(D)$ addition in D   however $y(D)$ is superlinear,
- if $y``(D) < 0$, $y`(D)$ however $y(D)$ decrease in D   however $y(D)$ is sublinear,
- if $y``(D) = 0$, $y`(D)$ is fixed in D however $y(D)$ is linear.

somewhat of the analysis data in Figure 3.4 demonstrates to next results

$D = 50$ Gy: $y` > 0$ ways y is growing ; $y`` > 0$ while y growing , upwards is the graph is concave . $g > 1$ etc y is Superlinear ; $f > 1$ ways the curve as supralinear.

$D = 500$ Gy: $y` > 0$, after that y is growing ; $y`` < 0$, y graph is concave lessening and downwards ; $g < 1$ y is sublinear ways ; $f > 1$ y is supralinear ways.
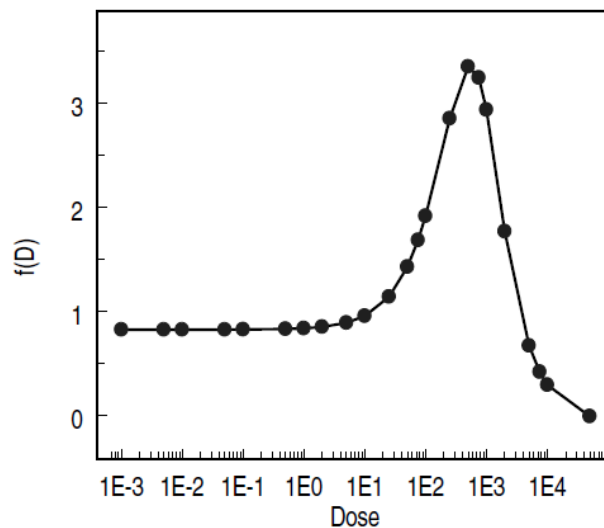

Figure 3.5   The function f (D).

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CHAPTER 4

EXPERIMENTAL PROCEDURE

The materials, equipments and experimental procedures were used in this work were described below.

4.1 Lithium borates

The material that performed in this study was $Li_2B_4O_7$: Mn (TLD-800) from Thermo Electron Corporation .The dimensions of this sample are (3.2 x 3.2 x 0.89 mm^3), with a mean mass of (0.220 g). Thermoluminescence dosimeters (TLD) like manganese activated lithium borate have attracted a significant attention due to their low atomic number, which is an approach to tissue equivalence, simple glow curve and low cost. Lithium borate has been utilized as a practical radiation dosimeter [57]. Most of the earlier publications pertain to the use of lithium borate with (0.1 wt%) of manganese impurity in medical applications, while in this work, lithium borate with (0.3 wt%) impurity of manganese has been prepared for applications of high-dose (1-30 kGy), [15 ,59,60]. Thermoluminescence dosimetry with borates has been of interest for some 15 years and has been stimulated by a number of factors. The most obvious is that materials such as $Li_2B_4O_7$ or MgB_4O_7 have a near tissue – equivalence and therefore, it is worth considering for the properties of their TL. In fact that the dosimeters of the lithium borate are override to LiF in tissue equivalence terms. The borates are relatively stable chemical compound and reply without serious problems for trying for doping them withthermoluminescence sensitisers like the rare earths , ions of copper or manganese . Now explain the resultant materials some of the desirable features for thermoluminescence in high sensitivity terms , linearity, storage and many of the earlier

fading problems, sensitivity of light and behavior of poor humidity have been avoided. We will first report some of the methods used for sample preparation [61].

4.2 Experimental Procedure and Equipments

Before next irradiation, the samples were first annealed to wipe any remaining information and then cool rapidly in the air at (75 ^{o}C/min) to the temperature of the room. For this reason annealed sample of the $Li_2B_4O_7$: Mn (TLD-800) at (300 ± 1 ^{o}C) for thirty minutes , Implemented all the treatments of annealing with specially designed oven of microprocessor- controlled electrical which is able to control the temperature within (± 1.0 ^{o}C). The samples were irradiated at room temperature with beta rays from a calibrated ^{90}Sr-^{90}Y source. The β-source activity is about (100 mCi). It is calibrated by the manufacturer on March, 10, 1994. The recommended working lifetime is about fifteen years. Stronium–90 emits high - energy beta particles from their daughter products (^{90}Sr β-0.546 MeV together with ^{90}Y β-2.27 MeV). Beta radiation is absorbed by air, so its intensity reduced with distance much More quickly than inverse square law calculations would indicate.

The maximum range of Y–90 beta particles in air is approximately 9 meters. The typical strength of a 100 mCi Sr–90 β-source installed in a 9010 Optical Dating System is 2.64 Gy/minute=0.0438 Gy/Sec for fine grains of aluminium, or 3,3 Gy/min=0.055 Gy/sec for 100 m quartz on stainless steel. The equipment of irradiation is an additional part of the 9010 Optical Dating System which is purchased from Little More Scientific Engineering, UK [72] . The irradiated samples were read out by a reader of Harshaw QS 3500 manual type that is connected to a PC where studied and analyzed the signals of thermoluminescence. It economically provides high reliability. The basic block diagram of reader is illustrated obviously in the figure 3.2. It always has been installed the standard filter of clear glass in the reader between the planchet and photomultiplier tube to eliminate the emitted infrared lights from the reader plus samples. All functions are divided between the reader and the specialized software Shell of the TLD that are running on the computer. All data storage, instrument control, and operator inputs are performed on the PC. Signal acquisition and conditioning are performed in the reader. As such, and can be analyzed each glow curve

by using a best-fit computer program depends on a procedure of a Marquardt algorithm minimization, related to expressions of the first-order and general-order kinetics. The individual peaks present in the curve are resolved by the program , giving the best values for the parameters of different peak . The instrument includes a sample change drawer for inserting and removing the TLD elements.The reader uses contact heating with a closed loop feedback system which produces adjustable linearly lifted temperatures from 1 ^{o}C to 50 ^{o}C per second accurate to within ± 1 ^{o}C to 600 ^{o}C in the standard reader. The Time Temperature Profile (TTP) is user defined in three segments: Preheat, Acquire, and Anneal, each with independent times (Pre-read anneal: adjustable 0 to 1000 sec, Linear ramp: adjustable from 1 ^{o}C to 50 ^{o}C per second, Post-read anneal: 0 to 1000 sec) and temperature (Pre-read anneal:room temperature to 200 ^{o}C, Post-read anneal: up to 400 ^{o}C). For improving the low-exposure reading accuracy and to extend planchet life, the 3500 provides for nitrogen to flow around the planchet . Through the elimination of oxygen in the salinity, flow of nitrogen eliminates unwanted TL signal caused by the oxygen. Nitrogen is also directed through the photo-multiplier tube (PMT) chamber to eliminate moisture caused by condensation.
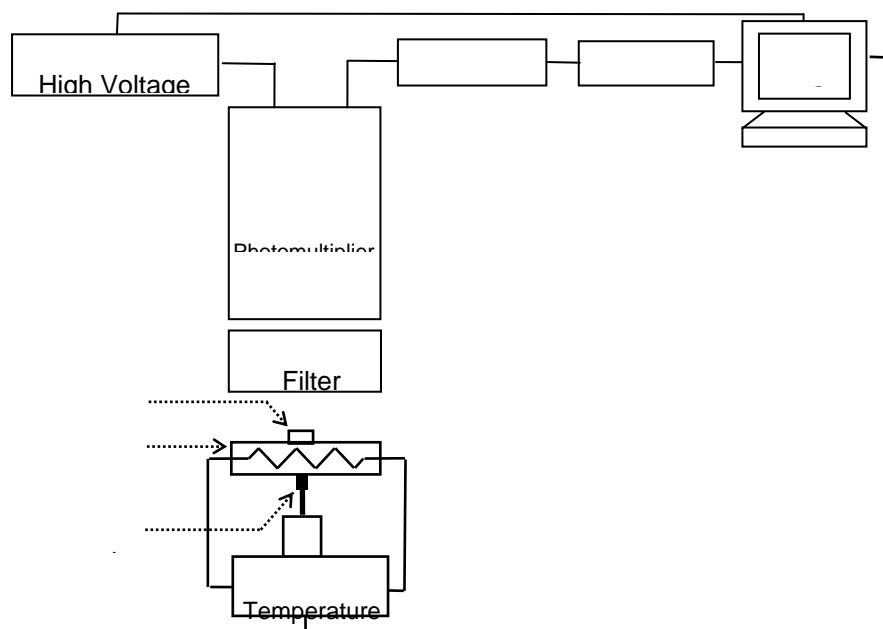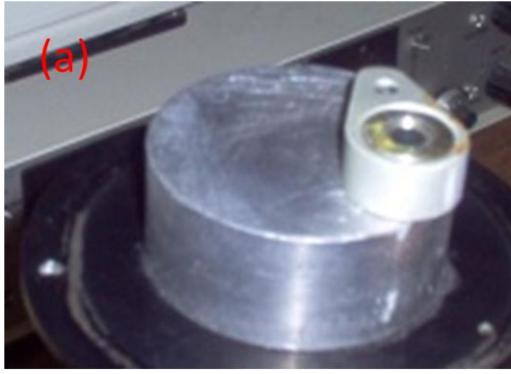

Figure 4.1 Basic block diagram of TL reader [72].

Figure 4.2 Experimental equipments (a) ^{90}Sr-^{90}Y β-source (b) 9010 Optical Dating System (c) Harshaw TLD System 3500.

CHAPTER 5

EXPERIMENTAL RESULTS

In this study, the researcher has investigated the effect of heating rate on the intensities and the linearities of thermoluminescence (TL) glow peaks of Lithium tetraborate ($Li_2B_4O_7$) single crystals at different linear heating rates between 1 $^oC/s$ and 20 $^oC/s$. Our major concern is a try to understand the effect of linear heating rate on the linearity and dose reaction features of $Li_2B_4O_7$ by using the supralinearity index $f(D)$.

A promising material for nonlinear optics and piezoelectric devices is the lithium tetraborate. These applications mean extensive studies of $Li2B4O7$ single crystals [1]. And, also this material is an older significant application: by inserting the impurities in these materials like (Cu, Mn, or Eu), it is acknowledged to be suitable for creation detectors of thermoluminescent (TLD) of ionizing radiation. ($Li2B4O7$) ceramics has a well tissue-equivalence to absorb the dose of ionizing radiation. Perfectly, it should be approximated the atomic number (Z) of the dosimetric substances to the biological tissue ($Z_{eff} = 7.42$) to be irradiated and thus becomes independent measurement of the energy of the photon. When the energy of the incident radiation like (γ- or X) radiation is in the range between (20 and 100 keV). By these energies, the photoelectric interaction is prevalent and depends on the (3) power of the atomic number (Z) ,therefore, in that region there is an over-response in the case of substances having a higher (Z). An effective atomic number of the ($Li_2B_4O_7$:Mn) ceramics are (7.40), and it is closer to the biological tissue.

When co-doped a small amount of SiO_2 with the ($Li_2B_4O_7$:Mn), can be fulfiled the precise value of ($Z_{eff} = 7.42$) of the biological tissue. The excellent tissue equivalence lead these materials to be more benifit as thermoluminescence dosimeter (TLD), in specific medical applications like the radiological examinations and therapeutic

treatments. Now the Li$_2$B$_4$O$_7$:Mn,Si is commercially available - for example, from Bicron on behalf of (TLD-800) [3].

And thus, there are more problems unresolved that restrict broader applications of this (TLD) substances. It is not clear yet, the (TL) mechanism of (Li$_2$B$_4$O$_7$:Mn)and this main problem resulting from issues of the several technical, like false dose problem that is result from sensitivity of the daylight or by some of the other causes. TLD could not measure an integral of the daylight dose, but must measure the integral of the ionizing radiation dose The daylight impact reduces the accuracy of measurements and decreases the lower threshold of the TLD sensitivity.

In this study the dose reply behavior of Li$_2$B$_4$O$_7$ (TLD-800) has been investigated at different linear heating rates between 1 ^{o}C and 20 ^{o}C by using the dose response function $f(D)$. TLD-800 crystal chips was firstly annealed at 400 ^{o}C for 30 minutes before the following irradiation and then cooled in air to room temperature. The sample was irradiated immediately after the standard annealing at room temperature with β rays from a ^{90}Sr- ^{90}Y source (≈0.04 Gy/s). Figure 5.1 shows one of the CGCD analyzed glow curve and results of fitting on the assumption of three peaks. Typical glow curves of TLD-800 sample, using a linear heating rate between 1 ^{o}C and 20 ^{o}C are shown in Figure 5.2a, 5.2b and 5.2c.
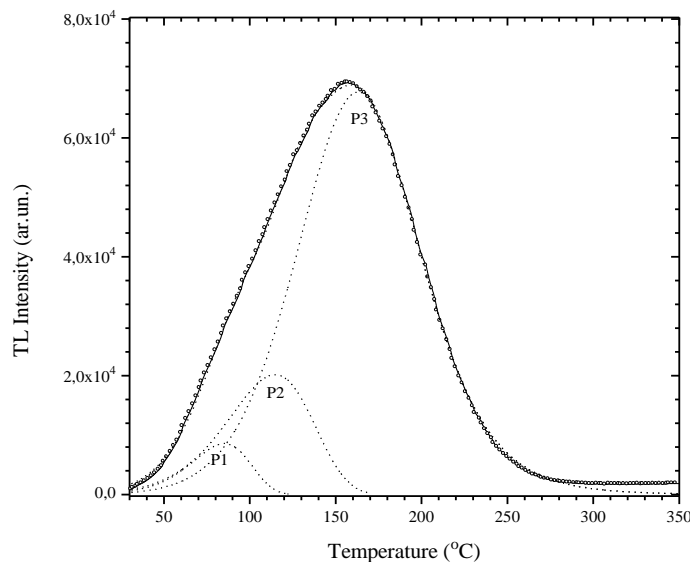

Figure 5.1 The CGCD analyzed glow curves of Mn-doped Li$_2$B$_4$O$_7$ measured after 72 Gy irradiation by beta ray at room temperature.

Figure 5.2a Typical glow curves of TLD-800 exposed to beta rays from 2,4 Gy up to 3.45 kGy and readout at linear heating rates of (a) $\beta = 1$ ^{o}C/s, (b) $\beta = 2$ ^{o}C/s, (c) $\beta = 3$ ^{o}C/s.

Figure 5.2b Typical glow curves of TLD-800 exposed to beta rays from 2,4 Gy up to 3.45 kGy and readout at linear heating rates of (d) $\beta = 4\ ^{\circ}C/s$, (e) $\beta = 5\ ^{\circ}C/s$, (f) $\beta = 8\ ^{\circ}C/s$.
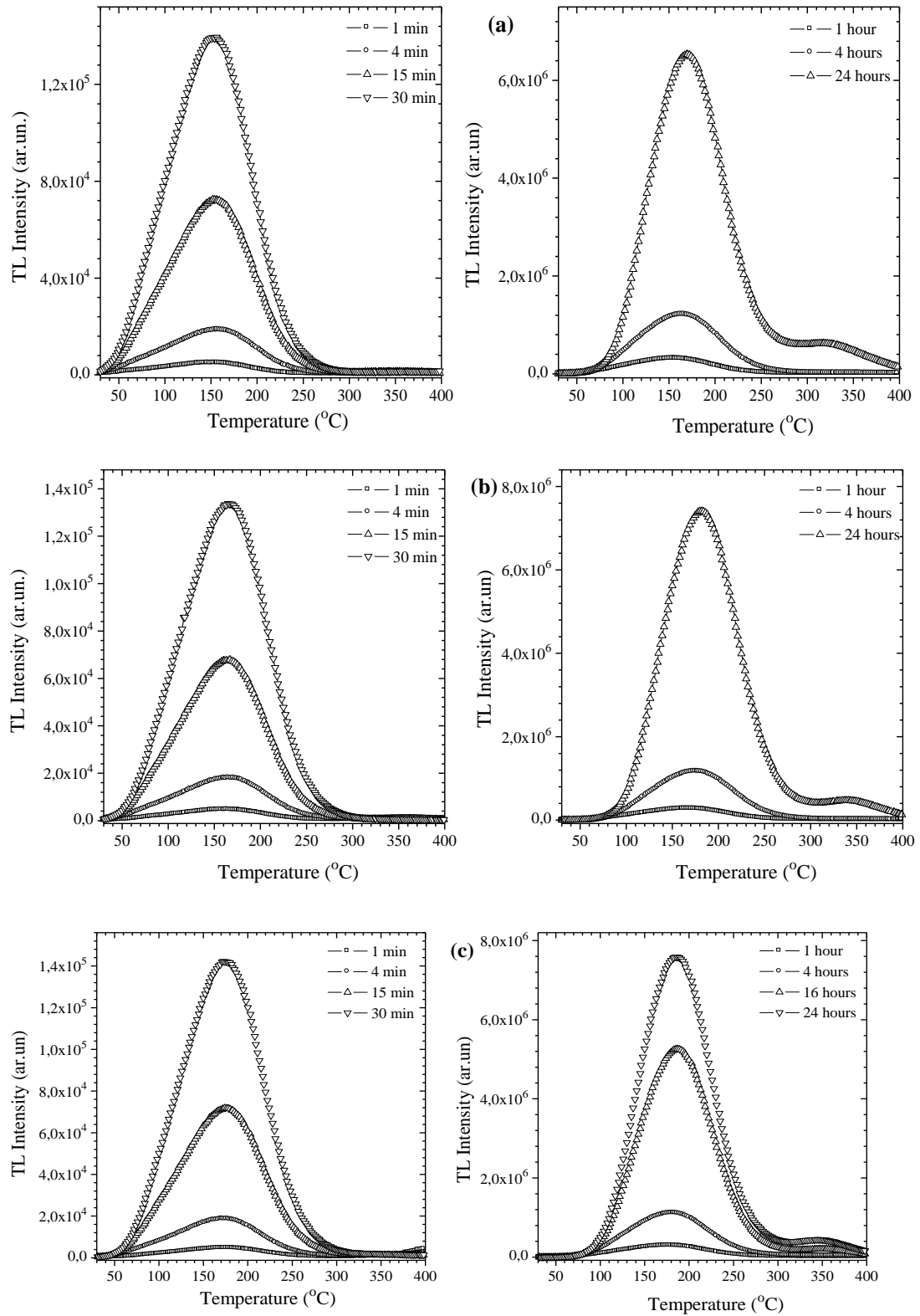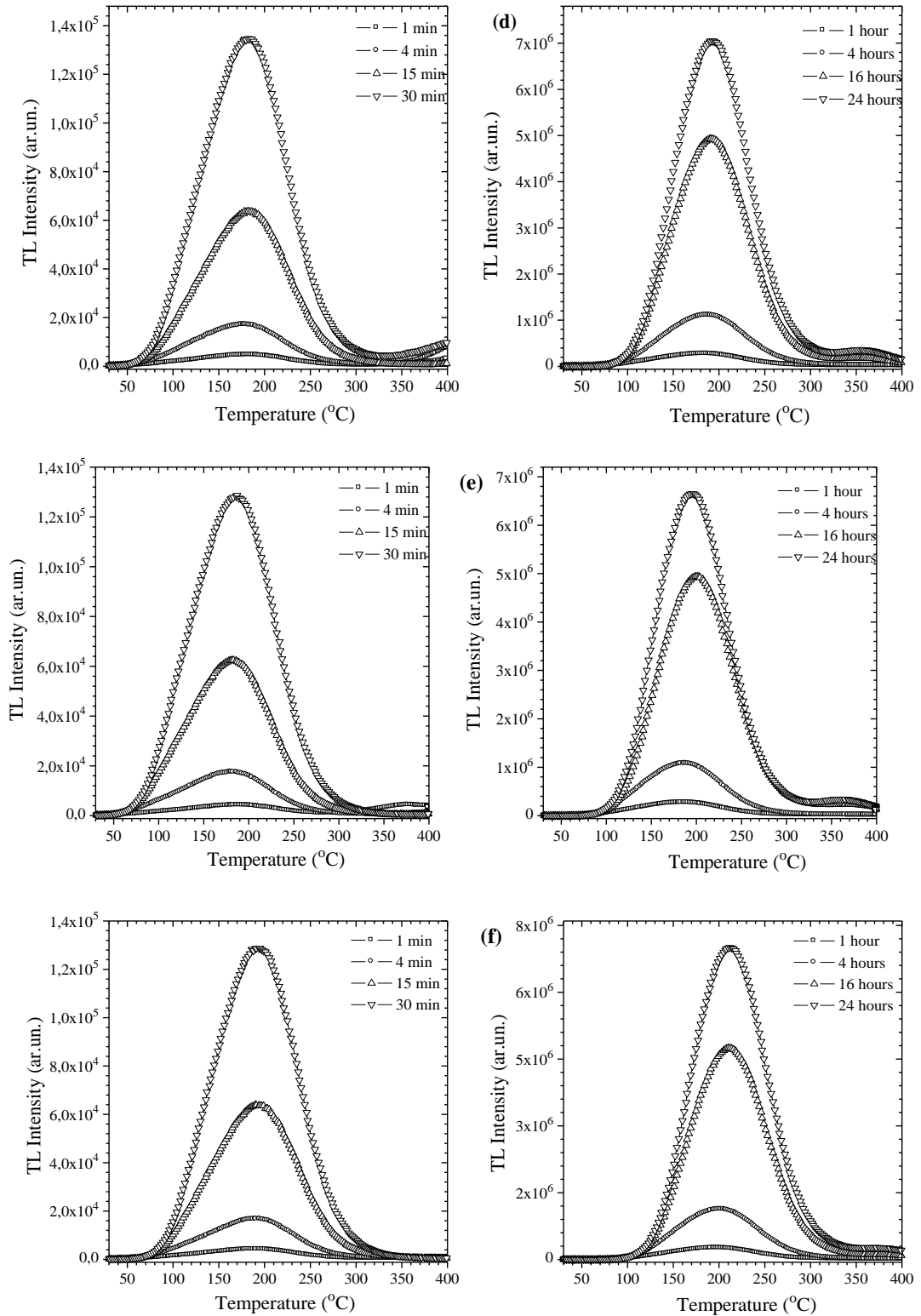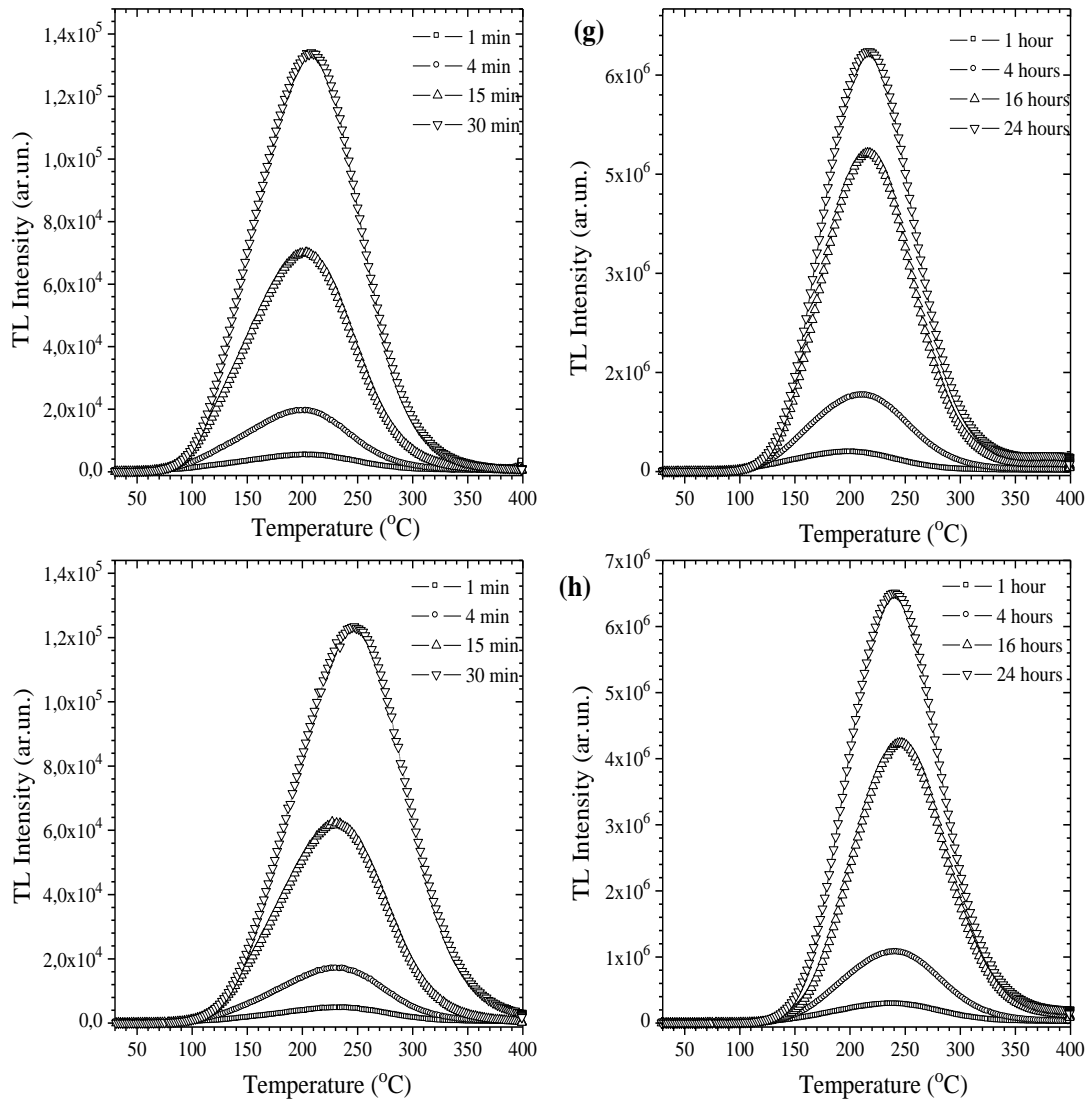
Figure 5.2c Typical glow curves of TLD-800 exposed to beta rays from 2,4 Gy up to 3.45 kGy and readout at linear heating rates of (g) $\beta = 10$ ^{o}C/s, (h) $\beta = 20$ ^{o}C/s.

Figure 5.3a and 5.3b show the development of the height of the peaks as a function of dose. As seen from these figures, the chief glow peak of TLD-800 shows linear behavior up to high dose levels especially at low heating rates. But when the heating rate is increased the peak shows a linear region at low doses. When the dose level is increased the peak goes into the supralinear region after 10^2 Gy then the peak saturates and goes into sublinear region after 10^3 Gy at high heating rates.

(a)

Figure 5.3a The growth of the height of the peaks from TLD-200 as a function of dose at a linear heating rate of $\beta = 1$ $^{\circ}$C/s, 2 $^{\circ}$C/s, 3 $^{\circ}$C/s, 4 $^{\circ}$C/s.

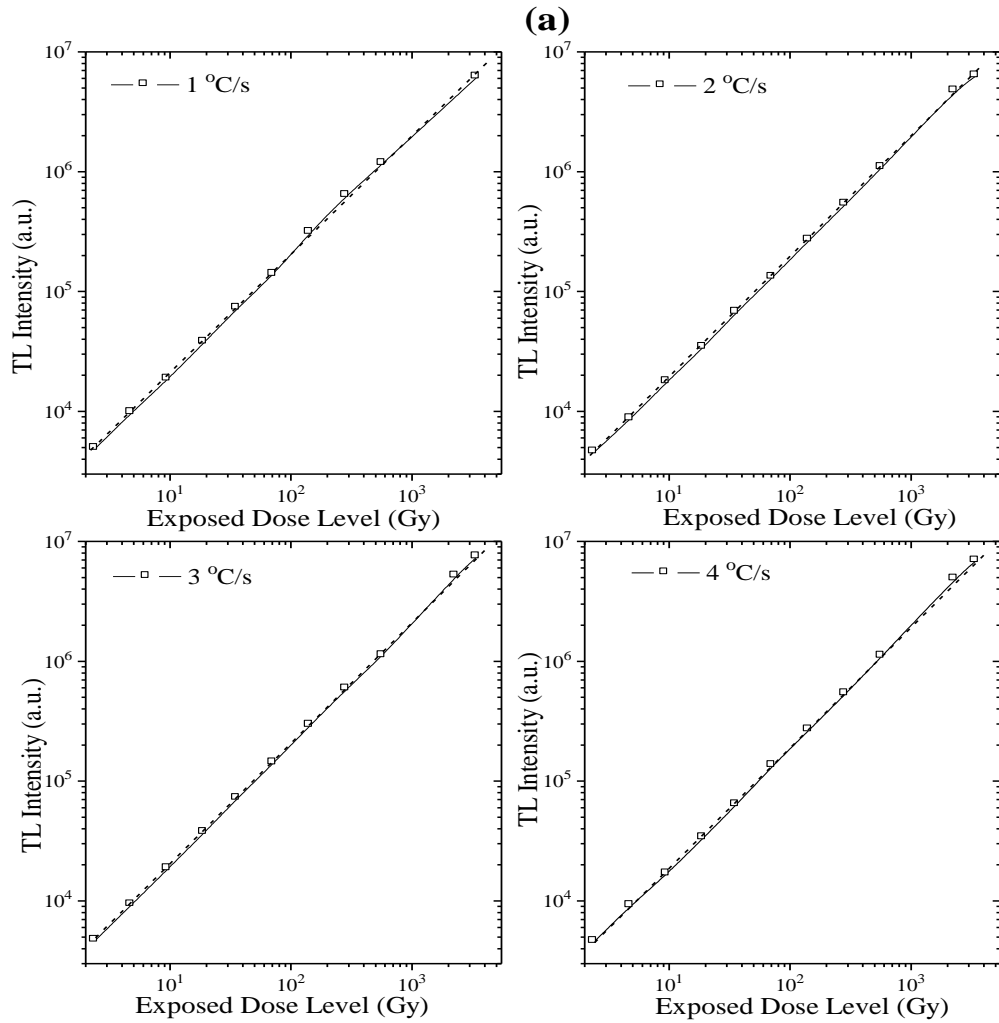Even figure 5.3a and 5.3b clarify the growth of the height of the peaks as a function of dose, to best understand the dose rate behavior of the peaks the dose response function, *f(D)*, is evaluated by using equation(**3.3**) **.** As seen from the figure 5.3a the curves display the main features of the dose reply. For the chief glow peak   *f(D)* of TLD-800, namely a linear *(f(D)=1)* region up to 10^1 Gy, followed by a region of supralinear growth *(f(D)>1)*, gradually going into a region of sublinear growth *(f(D)<1)* after 10^3 Gy for the heating rate of 1 $^{\circ}$C/s. The peak shows linear *(f(D)=1)* behavior nearly up to 10^2 Gy, followed by a region of supralinear growth *(f(D)>1)*then it saturates after that dose and shows a sublinear *(f(D)<1)* behaviour after 10^3 Gy for the heating rate of 2 $^{\circ}$C/s. When the heating rate is increased to 3 $^{\circ}$C/s, the linearity of the peak continues up to 10^2 Gy, the peak displays supralinear

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characteristic up to 10^4 Gy dose level. After this dose level the peak saturates and shows sublinear behavior. The dose response characteristic of the chief glow peak of TLD-800 states almost the same behavior at linear heating rates of 4 ^{o}C/s, 5 ^{o}C/s and 8 ^{o}C/s as in 3 ^{o}C/s. But the heating rate is further increased to 10 ^{o}C/s and 20 ^{o}C/s the peak shows linear behavior *(f(D)=1)* only at low doses, then the peak saturates *(f(D)<1)* immediately without a supralinear region.
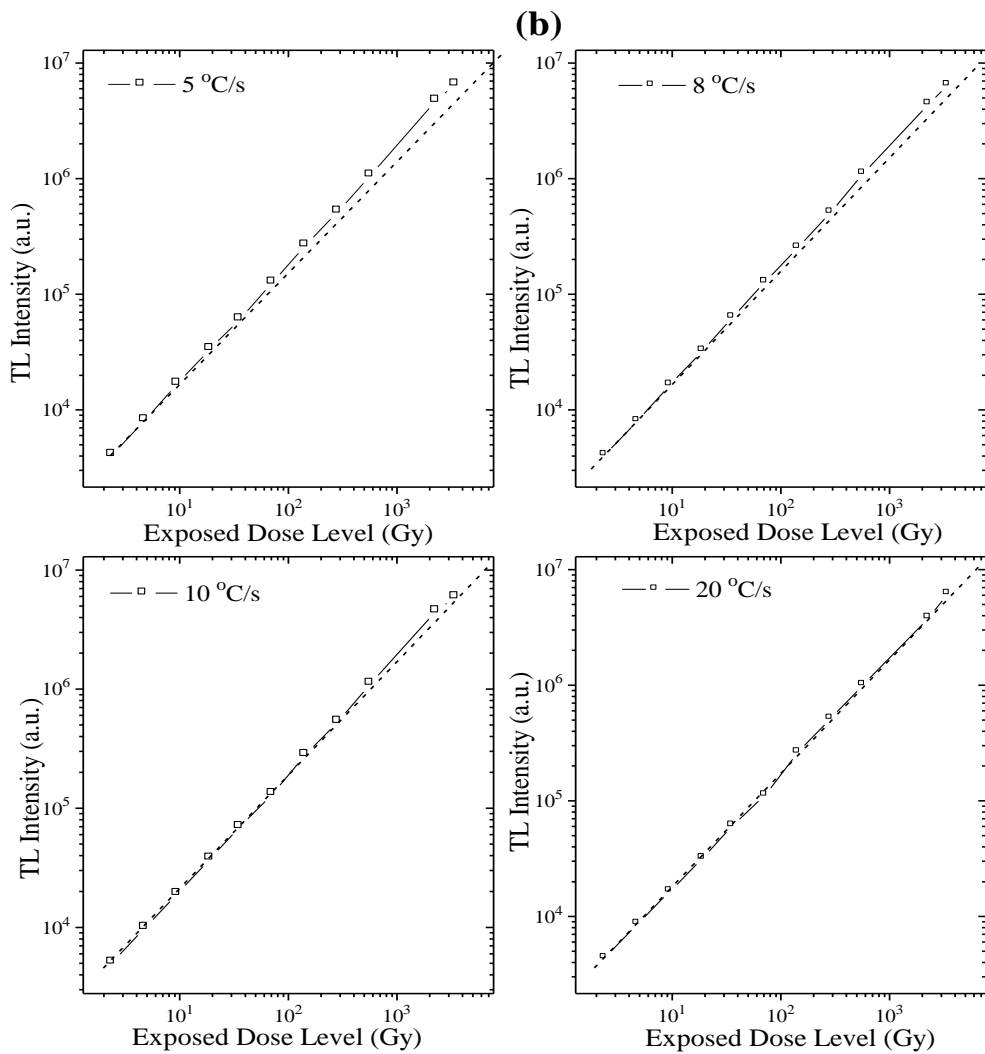
Figure 5.3b The growth of the height of the peaks from TLD-200 as a function of dose at a linear heating rate of β = 5 ^{o}C/s, 8 ^{o}C/s, 10 ^{o}C/s, 20 ^{o}C/s.
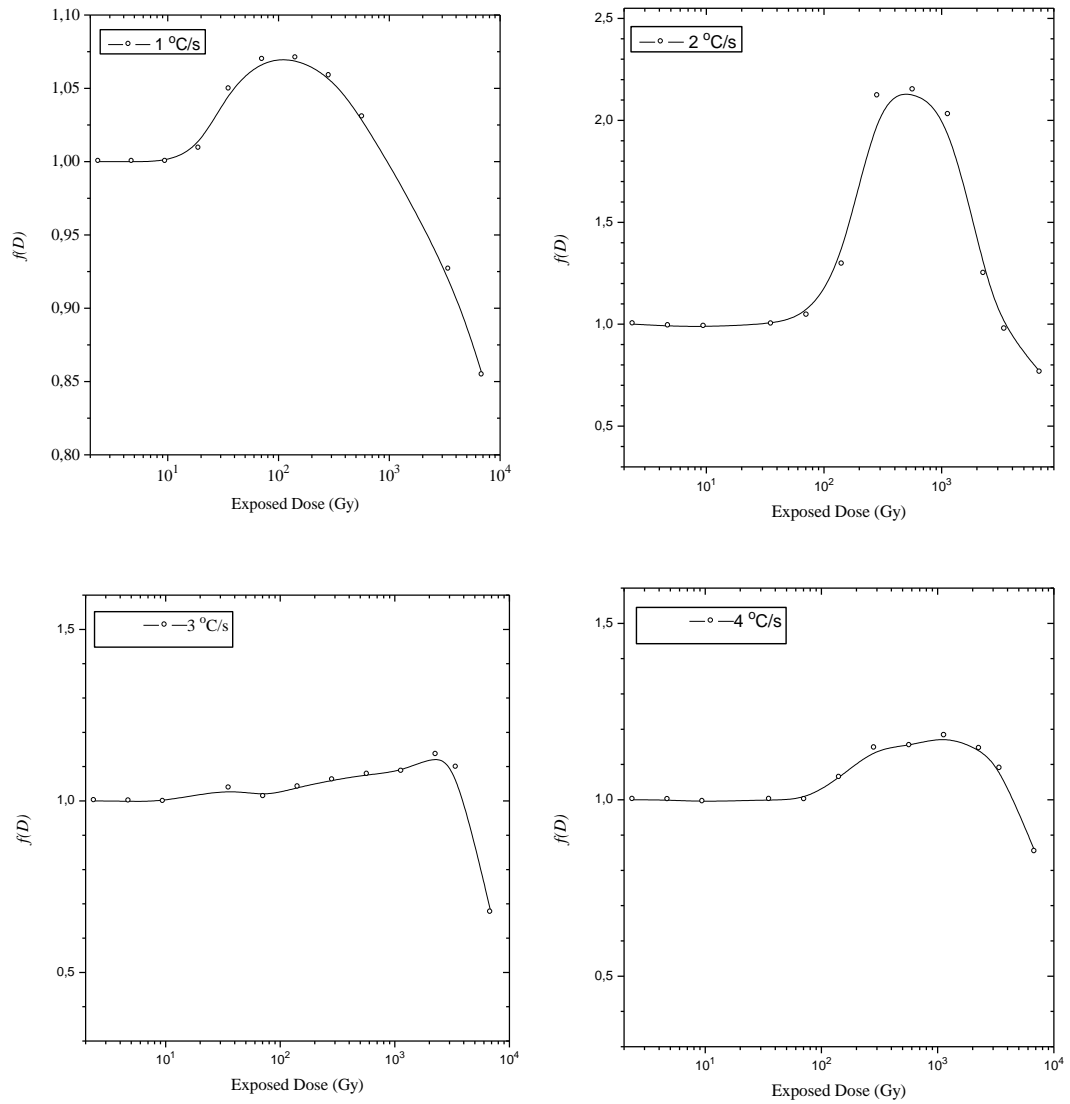
Figure 5.4a The dose response function *f(D)* vs *D* of TLD-800 ($\beta = 1$ °C/s, 2 °C/s, 3 °C/s, 4 °C/s)

The behavior of the glow curves and the effect of thermal quenching because of the heating rate on the entire peak area of glow curves were also investigated at different linear heating rates between 1 °C/s and 20 °C/s. Figure 5.5 shows some of the selected glow curves of $Li_2B_4O_7$ (TLD–800) at linear heating rates between 1 °C and 20 °C. As seen from this figure the peak temperatures of all peaks are shifted higher temperatures when the heating rate increases as expected in theory. It is seen from figure 4.4, the total peak area of the glow curves lessens as the heating rate increases.

The total area of the glow curves was normalized at the lower heating rate (1 °C s^{-1}) and it can be seen that the reduce in the integrated area of the peaks by about 65 %.
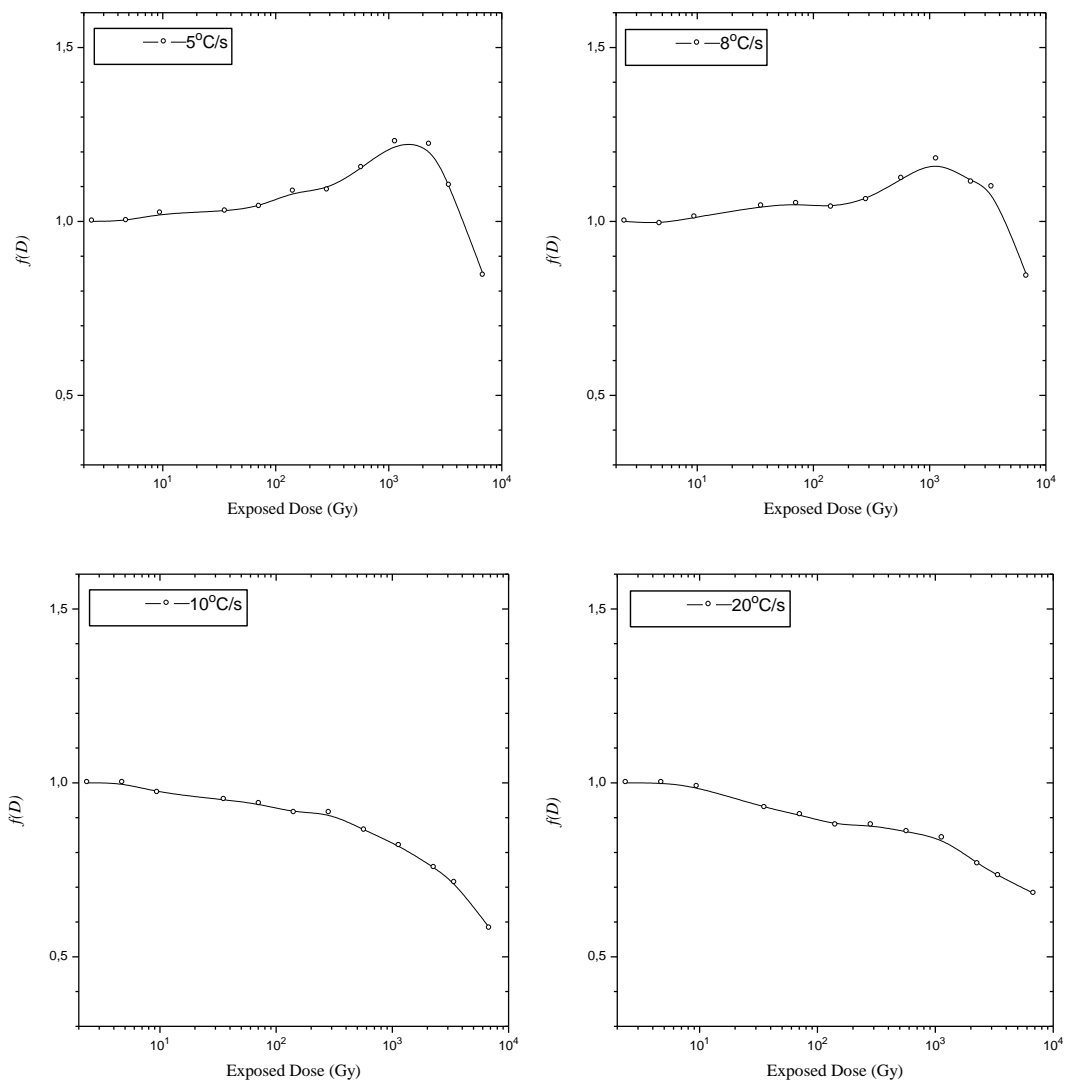
Figure 5.4b The dose response function $f(D)$ vs D of TLD-800 ($\beta = 5$ ^{o}C/s, 8 ^{o}C/s, 10 ^{o}C/s, 20 ^{o}C/s)
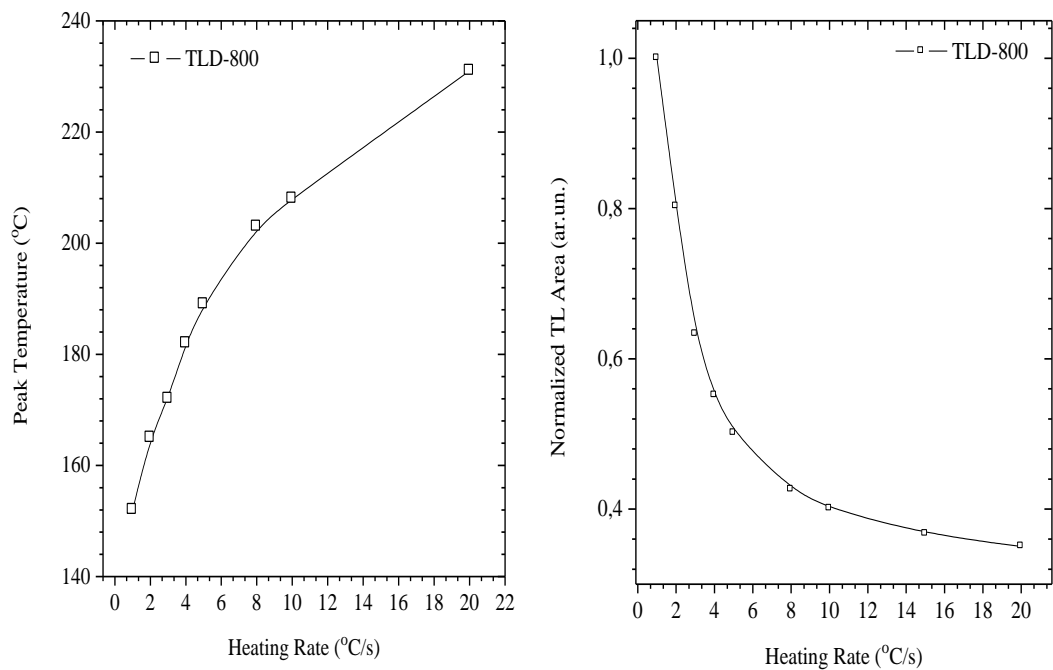
Figure 5.5 The change of the peak temperature and normalized total TL peak area of TLD-800 crystals irradiated with ^{90}Sr-^{90}Y beta source at linear heating rates between 1 ^{o}C/s and 20 ^{o}C/s (D=72 Gy)

CHAPTER 6

CONCLUSION

In this thesis, we have investigated the dose response characteristics of $Li_2B_4O_7:Mn$ (TLD-800) crystal from 2.4 Gy up to 3.45 kGy after annealing the sample at 400 ± 1 ^oC for 30 minutes. One can be seen from figure 5.1 the glow curve of this sample can be described by linear combination of three peaks between room tempearture and $300\ ^oC$ and also a best fit was always obtained by assuming that all the peaks are of general-order kinetic. In order to understand the nonlinearities in the dose dependence of this sample, the supralinearity function, *f(D)*, was evaluated by using equation 3.1. As seen from figures 5.a and 5.2b the curves exhibit the main features characteristic of the dose response. Genarally all the peaks shows linear behaviour at low doses, *fD)=1*, followed by a region of supralinear growth *(f(D)>1)*, than gradualy going into a region of sublinear growth *(f(D)<1)* at high doses.

In this study we have investigated the effect of heating rate on the intensities and the linearities of thermoluminescence (TL) glow peaks of Lithium tetraborate ($Li_2B_4O_7$) single crystals at different linear heating rates between $1\ ^oC/s$ and $20\ ^oC/s$. Our main concern is an attempt to understand the effect of linear heating rate on the linearity and dose response characteristics of $Li_2B_4O_7$ by using the supralinearity index *f(D)*. To have an idea about dose response characteristics of the glow curves, first of all the growth of the height of the peaks as a function of dose has been investigated. As seen from the figure as the dose level is increased the peak goes into the supralinear region after 10^2 Gy then the peak saturates and goes into a sublinear region after 10^3 Gy at high heating rates. Even the growth of the height of the peaks as a function of dose explain the dose response behaviors, the dose response function, *f(D)*, is evaluated by using equation 3.3 to best understand the dose rate behavior of the peaks. It can be concluded from the figures 4.4a and 4.4b, at low heating rates such as $1\ ^oC/s$ and $2\ ^oC/s$ the main glow peak *f(D)* of TLD-800, namely a linear *(f(D)=1)*

only at low doses. When the dose is increased a supralinear growth *(f(D)>1)* can be observed then the peak goes into a region of sublinear growth *(f(D)<1)* after high dose levels. When the heating rate is increased to $3\,^{\circ}$C/s, the linearity of the peak continues up to 10^2 Gy, the peak shows supralinear characteristic up to 10^4 Gy dose level. After this dose level the peak saturates and shows sublinear behaviour. The dose response characteristic of the main glow peak of TLD-800 shows nearly the similar behaviour at linear heating rates of $4\,^{\circ}$C/s, $5\,^{\circ}$C/s and $8\,^{\circ}$C/s as in $3\,^{\circ}$C/s. But the heating rate is further increased to $10\,^{\circ}$C/s and $20\,^{\circ}$C/s the peak shows linear behaviour *(f(D)=1)* only at low doses, then the peak saturates *(f(D)<1)* immediately without a supralinear region. From these observations we can conclude that the peak shows high linear behaviour at linear heating rate of $3\,^{\circ}$C/s and $4\,^{\circ}$C/s.

Nonlinearities often occur in the dose dependence of thermoluminescence (TL). These include sublinearity, usually when there is an approach to saturation in the dose dependence, as well as supralinearity, also termed superlinearity in the literature. Different researchers in the field have viewed the effect of supralinearity / superlinearity from two somewhat different points of view. One point of view has to do with the rate of change with dose of the dose dependence function. The other approach is related more to the applications of TL in dosimetry and archaeoological and geological dating, and basically has to do with the correction to be made in extrapolation in cases where supra (super) linearity occurs following an initial linear dose range, or prior to such a linear range. The effect of heating rate on the glow curves can be best described by the effect of thermal quenching. It is well known that the dosimetric characteristics of many TL materials are influenced by changes in location, size and shape of the glow curves due to changes in the heating rate. Thermal quenching was understood to be due to the increased probability of non-radiative transitions competing with the radiative transitions. Since the increase in the heating rate resulted in an increase in the temperature of the TL glow peak, this shift in temperature was held responsible for the increase in the contribution of non-radiative transitions. Then, it was inferred that the glow peaks occurring at higher temperatures must exhibit high thermal quenching than those occurring at lower temperatures in any TLD material. Apart from the increased probability of non-radiative transitions at higher temperatures, the observed effects have also been assigned to the effects of heating rate on the migration of charge carriers released

during the TL readout. The results of this study showed that, the peak temperatures of all peaks are shifted higher temperatures and the integrated peak area of the curves decreases as the heating rate increases as expected in theory. As seen from 4.4 the peak temperatures of all peaks are shifted higher temperatures when the heating rate increases as expected in theory. It is also seen from figure 5.5, the total peak area of the glow curves decreases as the heating rate increases. The total area of the glow curves were normalized at the lower heating rate (1 °C s^{-1}) and it can be seen that the decrease in the integrated area of the peaks by about 65 %.

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