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Applicability of the Zimmerman predose model in the thermoluminescence of predosed and annealed synthetic quartz samples

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Abstract

The "110°C" thermoluminescence (TL) peak of unfired synthetic quartz is known to exhibit a highly superlinear growth with absorbed dose. In this paper, it is shown that the well-known Zimmerman predose model can explain recent experimental results on the superlinearity of annealed synthetic quartz, as well as experimental results on the superlinearity of heavily predosed samples at room temperature. In the case of the predosed samples, the simulation solves the kinetic rate equations for the various stages in the experimental TL predose process. The results of the simulation explain the behavior of the TL versus dose curves at different predoses, as well as the detailed behavior of the superlinearity coefficient k as a function of the predose amount. In the case of the annealed samples, the simulation solves the kinetic equations for different values of the initial concentration of holes in the recombination center. The results of the simulation explain the behavior of the TL versus dose curves at different annealing temperatures, as well as the detailed behavior of the superlinearity coefficient k in each of the two distinct superlinearity regions. The simulation also produces the correct order of magnitude for the large sensitivity changes of the TL intensity observed in both sets of experiments.

Keywords: Thermoluminescence; Superlinearity; Quartz; Annealing; Kinetic rate equations

1. Introduction

The thermoluminescence (TL) dose response of the "110°C" TL peak of quartz has been the subject of several experimental and theoretical studies because of its importance in TL dating (e.g. Bailey, 2001; Chen and McKeever, 1997; Sunta et al., 1994). In particular, the TL dose response of quartz and its well-known change of sensitivity upon thermal and radiation treatments have been studied in both natural and synthetic samples (McKeever, 1991; and references therein).

Chen et al. (1988) found that the "110°C" TL peak of unfired synthetic quartz exhibits a highly superlinear growth with absorbed dose. Specifically, they found that the maximum TL intensity I_{max} follows a dose dependence of the form:

$$I_{max} = aD^k. \quad (1)$$

Here, D represents the absorbed dose; a and k are constants. By plotting Eq. (1) on a log–log scale, a linear graph is obtained with slope k. The value of k is a direct measure of the degree of superlinearity (Chen and McKeever, 1997), and will be referred to as the "superlinearity slope k" in the rest of this paper. It is noted that k is assumed constant within a certain dose range. It is also noted that superlinear growth of I_{max} is known to occur only for low doses.

Chen et al. (1988) found that this superlinearity is removed by firing the samples above 300°C. The work of Chen et al. (1988) was limited to the "110°C" TL peak

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and to doses under 10 Gy. These authors explained both the superlinearity and the well-known change in the TL sensitivity of synthetic quartz by using the recombination during heating model. According to this model, firing the quartz samples at high temperatures removes the competitors and results in a reduction of the superlinearity. Chen et al. (1988) suggested that the role of the competitors may be filled by electron traps known as E_1' centers.

Charitidis et al. (1999) studied the dependence of the TL dose response on the pre-dose delivered to the sample. By carefully controlling the pre-dose conditions, these authors showed that a high pre-dose effectively removes the superlinearity effect. Recently, Charitidis et al. (2000) extended the experimental work of Chen et al. (1988) by measuring the sensitivity and TL dose response of several glow-peaks in synthetic quartz as a function of the annealing temperature. Their measurements extended the range of annealing to temperatures between 300°C and 900°C. These authors obtained complete TL dose response curves between 0.1 and 170 Gy at each annealing temperature.

Chen et al. (1988) and Charitidis et al. (2000) found that the superlinearity slope k of the "110°C" TL peak is strongly reduced as the firing temperature increases. Furthermore, it was found that each TL dose-response curve consists of two dose regions, which on a log–log scale appear linear with different slopes k, indicating two different degrees of superlinearity. For doses less than 11 Gy, the superlinearity slope k drops continuously from a value of 2 at room temperature to a value of 1 for an annealing temperature of 700°C, and then slowly increases to a value of 1.2 at a temperature of 900°C. For doses between 20 and 100 Gy, the superlinearity slope k exhibited a very different behavior, by remaining constant at a value of 2.5 from room temperature upto annealing temperatures of 500°C, and then decreases continuously to a value of 0.6 at an annealing temperature of 900°C.

Chen and Leung (1999) developed a mathematical model based on two electron and two hole trapping states. The model is a modification of the Zimmerman theory that explained first the predose effect (Zimmerman, 1971a, b). Chen and Leung (1999) simulated the sequence of experimental actions taken during the pre-dose dating technique, by solving the differential equations for each stage in the experiment.

The purpose of the present paper is to show that the model of Chen and Leung (1999) can also be used to describe the behavior of the superlinearity of the "110°C" TL peak as a function of the annealing temperature, as well as a function of the predose amount. It is found that the model accurately reproduces several features observed experimentally by Charitidis et al. (2000). Specifically, in the case of annealed quartz samples, the model agrees with the following experimental observations:

1. There are two superlinearity regions, which exhibit a distinct behavior as a function of the annealing temperature.
2. In the low dose region, the dose dependence exhibits initially a nearly quadratic behavior with a superlinearity constant, k, approximately equal to 2. As the initial concentration of recombination centers is increased, the behavior changes to linear with a superlinearity coefficient of 1. As discussed later in the paper, the low dose region corresponds to doses $D < 6 \times 10^{10}$ cm^{-3}.
3. In the high dose region, the superlinearity coefficient gradually decreases from a value of about 2.3 to a value near 1.0, as the initial concentration of recombination centers is increased. The high dose region corresponds to doses $D > 6 \times 10^{10}$ cm^{-3}.
4. The TL vs. dose graphs at different annealing temperatures do not saturate at the same level, but the saturation levels differed by many orders of magnitude.

Furthermore, it is found that the same modified Zimmerman model can be used to explain the superlinearity changes observed as a function of the predose amount, as measured by Charitidis et al. (1999). In the case of predosed quartz samples, the model agrees with the following experimental observations:

(1) The TL vs. dose graphs at different predoses were found to saturate at the same level of TL intensity.
(2) As the amount of the predose was increased within two orders of magnitude, the superlinearity coefficient k changed gradually from a superlinear behavior with slope near $k = 2$ to a linear behavior with $k = 1$.

In addition, the simulation also produces the correct order of magnitude changes for the large sensitivity changes taking place in the TL intensity, in both sets of experiments.

The work presented in this paper provides strong supporting evidence for the applicability of the modified Zimmerman model in explaining the superlinearity and sensitivity changes in synthetic quartz.

2. Description of the modified Zimmerman predose model

Zimmerman (1971a, b) first proposed a predose model consisting of one electron trapping state T, a luminescence center L and a hole reservoir R. Chen (1979) proposed a modification of the Zimmerman model by adding an extra electron level S which competes for electrons during the heating stage. By using physical arguments concerning the observed experimental behavior of quartz samples, Chen and Leung (1999) were able to arrive at a "good" set of parameters for the modified Zimmerman model, which explains successfully the following experimental results:

1. Linear dependence of the TL signal on the test dose.
2. Exponential approach of the sensitivity to saturation with repeated additive doses.

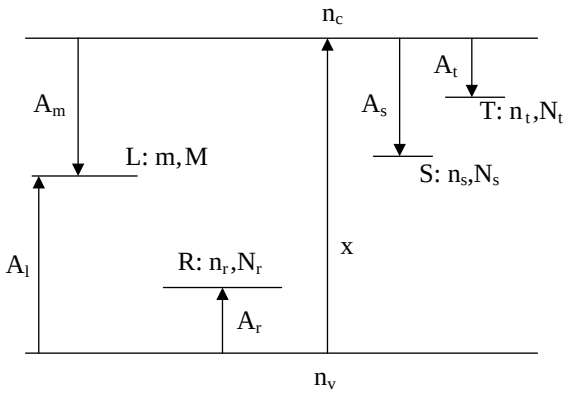

Fig. 1. The energy scheme used in the modified Zimmerman predose model.

3. Quenching by high dose exposure, UV reversal and the distinction between reservoir and center saturations. Further work using the present model is necessary in order to obtain a quantitative description of these phenomena in agreement with experimental data available in the literature. Such work is currently in progress.

Fig. 1 shows the energy scheme used by Chen and Leung (1999). It consists of two trapping states T and S and the hole reservoir R, with total concentrations N_t, N_s and N_r (in cm^{-3}) and with instantaneous occupancies denoted by n_t, n_s and n_r (in cm^{-3}) correspondingly. The activation energy for the main traps T is E_t (in eV) and the frequency factor is s_t (s^{-1}), while the competitor traps S are considered to be thermally disconnected. The activation energy for the hole reservoir R is E_r (in eV) and the frequency factor is s_r (s^{-1}). The retrapping probability coefficients for R, T and S are denoted by A_r, A_t and A_s (in $cm^3\ s^{-1}$), and n_c and n_v (cm^{-3}) represent the concentrations of electrons and holes in the conduction and valence band, respectively. The rate of production of electron–hole pairs x (in $cm^{-3}\ s^{-1}$) is proportional to the dose rate. The quantity $D = xt$ represents the total concentration of pairs produced, and is proportional to the dose (where t is the irradiation time in sec and D is in cm^{-3}). For simplicity, the quantity $D = xt$ will be referred to as "the dose $D = xt$" in the rest of this paper. M and m denote, respectively, the concentration and occupancy of the hole centers (cm^{-3}) and A_m, A_l ($cm^3\ s^{-1}$) are, respectively, the recombination probability coefficients of electrons and holes into the recombination center L.

The differential equations for the excitation and heating stages are given by Chen and Leung (1999) and will not be repeated here. After the excitation stage, the simulation uses a relaxation period in which the temperature of the sample is kept constant at room temperature for a certain period of time after the excitation has stopped ($x = 0$), and the concentrations of n_c and n_v decay to negligible values. A value of 30 s for the relaxation time is found to be sufficient for n_c and n_v to reach negligible values.

A linear heating rate β is assumed in the simulation, so that $T = T_o + \beta t$ and, of course, $x = 0$ during the readout stage. The emitted light is taken to be proportional to the rate of recombination so that:

$$I = -\mathrm{d}m/\mathrm{d}t = A_m m n_c. \quad (2)$$

The systems of simultaneous differential rate equations are solved using conventional Runge–Kutta algorithms with an adaptable time interval in the commercial package Mathematica. Table 1 shows the parameters used in solving the rate equations, along with the original parameters used by Chen and Leung (1999). In order to be able to demonstrate the behavior seen in the experiments, it was found necessary to make three minor modifications to the parameters used in the model of Chen and Leung (1999) as follows:

(a) In order to explain the huge change in sensitivity caused by high temperature annealing (as much as 3–5 orders of magnitude), the capacity of competitor states N_s was changed from 10^{12} to a value of 1.01×10^{13}.
(b) The capacity of reservoir states was changed from $N_r = 10^{13}$ to 10^{14} cm^{-3}.
(c) In order to match the observed behavior of the superlinearity slope, the probability A_s was changed from a value of 10^{-11} to 0.5×10^{-11} ($cm^3\ s^{-1}$).

The rest of the parameters in the model were left unchanged, and are listed in Table 1.

Table 1
The parameters used by Chen and Leung (1999), and in this paper.

Chen and Leung (1999)	Modified values used here
E_t 1.0 eV	
s_t 10^{13} (s^{-1})	
E_r 1.4 eV	
s_r 10^{13} (s^{-1})	
xt 5×10^{10} cm^{-3}	
A_r 10^{-10} $cm^3\ s^{-1}$	
N_r 10^{13} cm^{-3}	10^{14}
M 10^{14} cm^{-3}	
A_s 10^{-11} $cm^3\ s^{-1}$	0.5×10^{-11}
A_m 10^{-12} $cm^3\ s^{-1}$	
A_t 10^{-12} $cm^3\ s^{-1}$	
N_t 10^{13} cm^{-3}	
N_s 10^{12} cm^{-3}	1.01×10^{13}
A_l 10^{-12} $cm^3\ s^{-1}$	

The numbers in the third column show the modified values used in this paper, when they are different from those of Chen and Leung (1999).

3. Numerical results and discussion

3.1. Simulation of the Charitidis et al. (1999) predose measurements

The initial conditions of the electron and hole states for the predose simulation were taken as $n_s(0)=n_t(0)=m(0)=n_r(0)=0$. The model simulates the predose measurements of Charitidis et al. (1999) by solving the differential equations for the following stages:

(1) Predose irradiation stage: the sample is given a predose irradiation. The electrons go to the trapping states T and S, while most of the holes go preferentially to the reservoir R. Some of the holes go to the luminescence center L.
(2) A relaxation time of 30 s is incorporated in the program, at the end of which the concentrations n_c and n_v go to zero. The concentrations of n_s, n_t, m and n_r at the end of the relaxation period are used as the initial values for the next step (heating to 500°C) which is described below.
(3) The sample is heated to 500°C in order to empty the shallow traps. This heating stage causes some of the holes in the reservoir R to be released and to be captured in the luminescent center L. The concentrations of n_s, n_t, m and n_r at the end of this heating period are used as the initial values for the next steps (irradiation and measurement of TL) which are described below.
(4) The curve of TL vs. dose is obtained in the following manner. The sample is irradiated, then allowed to relax for 30 s, and its TL glow peak is measured by heating to 150°C. The maximum of the TL peak (TL_{max}) is recorded. Typical values of the temperature of maximum intensity T_{max} are in the range $T_{max}=85$–100°C, as measured with a heating rate of 1°C s^{-1}. This step (of irradiation, relaxation and TL measurement) is repeated several times using the same initial conditions for n_s, n_t, m and n_r and for different irradiation times (doses), and the variation of the TL_{max} with the doses is obtained.

Finally, the series of steps (1)–(4) above is repeated for different amounts of predose irradiation. The result is the series of TL vs. dose graphs shown in Fig. 2a, and it is to be compared with Fig. 1 shown in Charitidis et al. (1999). In order to make the comparison of theory and experiment easier, the data of Charitidis et al. (1999) have been redrawn in Fig. 4.

It is seen from Fig. 2a that the TL vs. dose graphs saturate at the same level, independent of the different predose amounts. The data in Fig. 2a show two superlinearity regions, a low-dose region with dose $D < 8\times10^{11}$ (cm^{-3}), and a high one with $D > 8\times10^{11}$ (cm^{-3}). Experimentally, Charitidis et al. (1999) observed only the high dose region, while the low dose region most likely is located

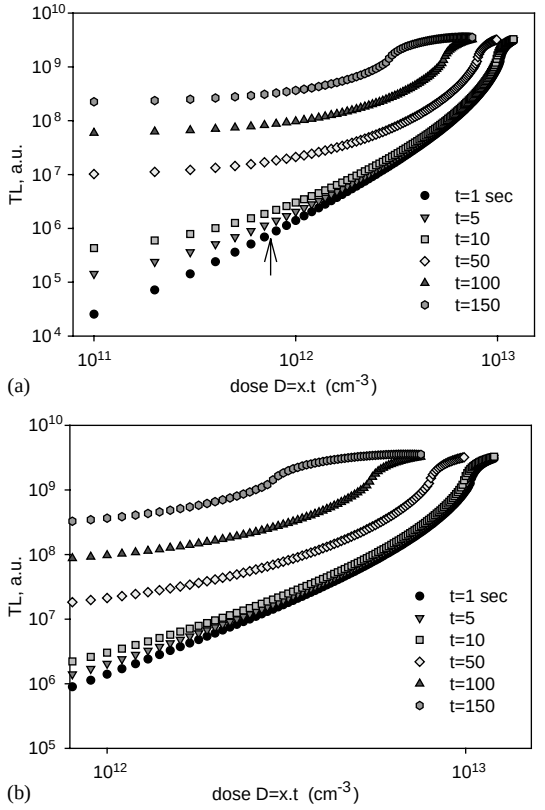

Fig. 2. (a) The TL vs. dose curves calculated from the predose model, for the different predose irradiation times t is shown. The arrow indicates the approximate location of the transition area between the two superlinearity regions; and (b) the same data is shown only in the high-dose region, which is observed experimentally.

below the sensitivity of the TL equipment. Chen and Fogel (1993), while discussing the results of their superlinearity model for synthetic quartz, also made the same observation.

Fig. 2b shows the same data as Fig. 2a, but restricted to the high dose region. Fig. 3 shows the superlinearity slope k calculated at the linear regions of the curves in Fig. 2b. The region of superlinearity being analyzed is much easier to visualize by referring to the inset of Fig. 3, which shows two of the calculated graphs from Fig. 2(a). The inset shows clearly the two regions of superlinearity, and the range of doses $1.5\times10^{12} < D < 3.5\times10^{12}$ (cm^{-3}), where the superlinearity slope k is being calculated. It is seen from Fig. 3 that the value of the superlinearity slope k changes gradually from a value of k near 2.0, to a value of approximately $k=1.0$ when the predose irradiation time is changed by two orders of magnitude (from predose time $t=1$ to 100 s. This is in good agreement with the data shown in Fig. 4(b), where the same behavior of the slope k is found when the experimental predose amount was varied from 1 to 100 Gy.

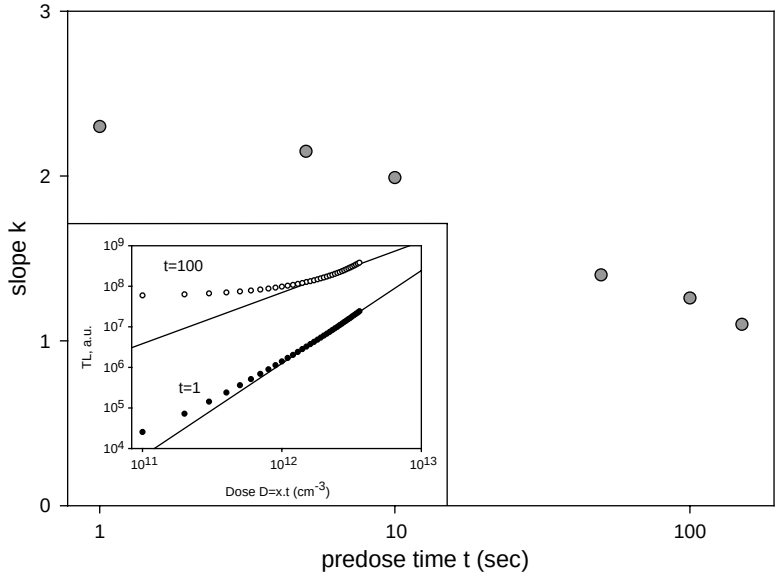

Fig. 3. The superlinearity slope k calculated from the initial part of Fig. 2(b). This is to be compared with Fig. 4 in this paper, which shows the data of Charitidis et al. (1999). The inset shows two of the graphs of Fig. 2(a), indicating clearly the two regions of superlinearity, and the range of doses where the superlinearity slope k is being calculated.

The model also predicts the correct order of magnitude of the change in sensitization caused by the predose effect. Fig. 2b shows that at the lowest doses, the sensitivity is changed by about 2.5 orders of magnitude, in close agreement with the data shown in Fig. 4(a).

It must be noted that the TL vs. dose curves in Fig. 2b have a slightly different shape than the experimental curves in Fig. 4(a), especially near the saturation point. This may be due to the fact that the experimental results shown in Fig. 4(a) contain only six data points, which may be obscuring the exact shape of the TL vs. dose curves near saturation. Also, it is not claimed that the exact values of the parameters are known, but rather that the model results in curves which agree at least qualitatively with the experimental results.

3.2. Simulation of the Charitidis et al. (2000) annealing experiments

In order to explain the results of the annealing experiments, the initial conditions of the electron and hole states must be varied according to the modified Zimmerman predose mechanism. The sensitization mechanism here is assumed to be the transfer of holes from the reservoir R to the luminescence center L by high temperature annealing (Chen and McKeever, 1997, p. 206). Conservation of charge requires that

$$n_s(0) + n_t(0) = m(0) + n_r(0). \quad (3)$$

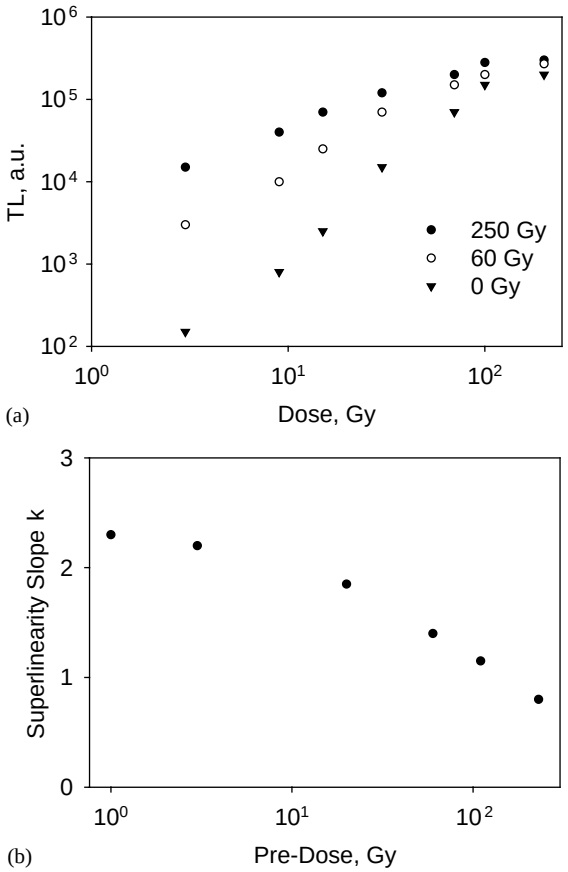

Fig. 4. The data of Charitidis et al. (1999) are partially redrawn for comparison purposes.

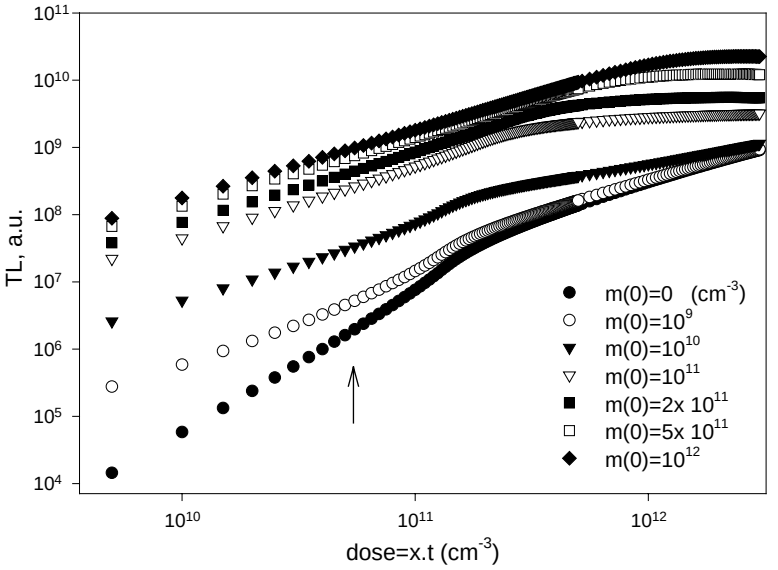

Fig. 5. The TL vs. dose curves calculated for different initial concentrations of luminescence centers. The arrow indicates the approximate location of the transition area between the two superlinearity regions. This is to be compared with Fig. 6(a) in this paper, which shows the experimental data of Charitidis et al. (2000).

As the annealing temperature is increased, it is expected that more holes are thermally released from the reservoir R and are trapped in the luminescence center L. The value of $n_r(0)$ will be decreased and the value of $m(0)$ will be increased by the same amount, so that the balance of charge is maintained according to Eq. (3) above.

It is assumed that the initial concentration of thermally disconnected competitors $n_s(0)$ remains the same at different annealing temperatures, while the main traps have been emptied by the annealing so that $n_t(0) = 0$. Finally, it is assumed that a constant small initial number of electrons is trapped in the thermally disconnected competitor states $n_s(0) = 0.1 \times 10^{14}$ cm^{-3}, so that Eq. (3) is satisfied.

In summary, the model assumes that as the annealing temperate is increased:

(a) $n_r(0)$ is decreased;
(b) $m(0)$ is increased by the same amount;
(c) $n_t(0) = 0$;
(d) Eq. (3) is satisfied by using an arbitrary constant value of $n_s(0) = 0.1 \times 10^{14}$ cm^{-3}.

The modified Zimmerman model simulates the annealing measurements of Charitidis et al. (2000) in the following stages:

(1) The sample is irradiated with a certain dose, and the differential equations given by Chen and Leung (1999) are solved using the initial conditions described above. The concentrations of n_s, n_t, m and n_r at the end of the irradiation period are used as the initial values for the next step (relaxation).
(2) A relaxation time of 30 s is incorporated in the program, which results in the concentrations n_c and n_v going to zero at the end of the 30 s interval. The concentrations of n_s, n_t, m and n_r at the end of the relaxation period are used as the initial values for the next step (measurement of the TL glow curve).
(3) The sample is heated to 300°C in order to measure the TL glow curve. The maximum intensity TL_{max} is recorded.
(4) The steps (1)–(3) above are repeated for different doses and by using the same initial conditions, and the curve of TL_{max} vs. dose is obtained in this fashion.

Finally, the series of steps (1)–(4) above is repeated for a larger initial value of $m(0)$, with the corresponding values of $n_r(0)$ calculated from Eq. (3). The result of the whole process is the series of TL vs. dose graphs shown in Fig. 5, and it is to be compared with the data in Charitidis et al. (2000), which is redrawn in Fig. 6.

It is seen from Fig. 5 that the TL vs. dose graphs saturate at different levels that range within many orders of magnitude. The data in Fig. 5 also show two superlinearity regions, a low-dose region with dose $D < 6 \times 10^{10}$ (cm^{-3}), and a high-dose region with dose $D > 6 \times 10^{10}$ (cm^{-3}). Experimentally Charitidis et al. (2000) also observed the two superlinearity regions, for doses $D < 11$ Gy and doses $D > 11$ Gy, as shown in Fig. 6(a). The two regions of superlinearity being analyzed are much

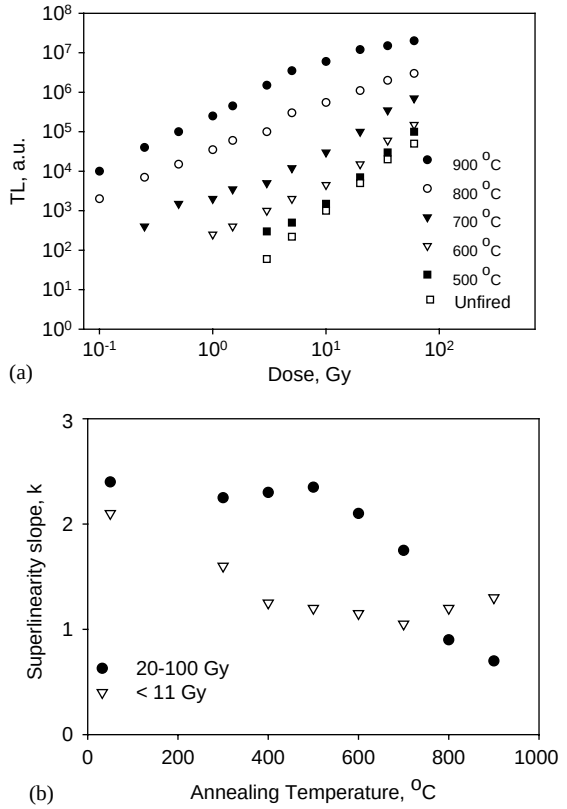

Fig. 6. The data of Charitidis et al. (2000) are partially redrawn for comparison purposes.

easier to visualize by referring to the inset of Fig. 7, which shows two of the calculated graphs from Fig. 5. The inset of Fig. 7 shows clearly the two regions of superlinearity, and the range of doses $D < 6 \times 10^{10}$ and $6.5 \times 10^{10} < D < 1.5 \times 10^{11}(\mathrm{cm}^{-3})$, where the two superlinearity slopes k are being calculated.

Fig. 7 shows the superlinearity slope k calculated at the two superlinear regions of the curves in Fig. 5. It is seen from Fig. 7 that the value of the superlinearity slope k changes in the low-dose region from a value of approximately $k = 2$, to a value of approximately $k = 1$. At the same time, the value of k changes in the high-dose region from a value of approximately $k = 2.3$, to a value of approximately $k = 1$. This is in very good agreement with the behavior shown in Fig. 6(b), where the same behavior of the slope k is found when the annealing temperature is varied from 0°C to 900°C.

It must also be noted that the model predicts the correct order of magnitude of the change in sensitization caused by the annealing effect. Fig. 5 shows that the TL sensitivity is changed within a range of 6 orders of magnitude (10^4–10^{10} arb. units) when the dose is changed within 2.5 orders of magnitude, in close agreement with Fig. 6(a).

Finally, the model predicts that the location of the maximum in the glow curve $\mathrm{TL}_{\max}$ shifts to lower temperatures as the value of $m(0)$ is increased. This is in agreement with the experimental results of Charitidis et al. (2000) who observed a similar shift in the TL glow curve maximum when the annealing temperature was increased. In all cases, the shape of the calculated TL glow curves is close to a first-order kinetics peak, in agreement with experiment.

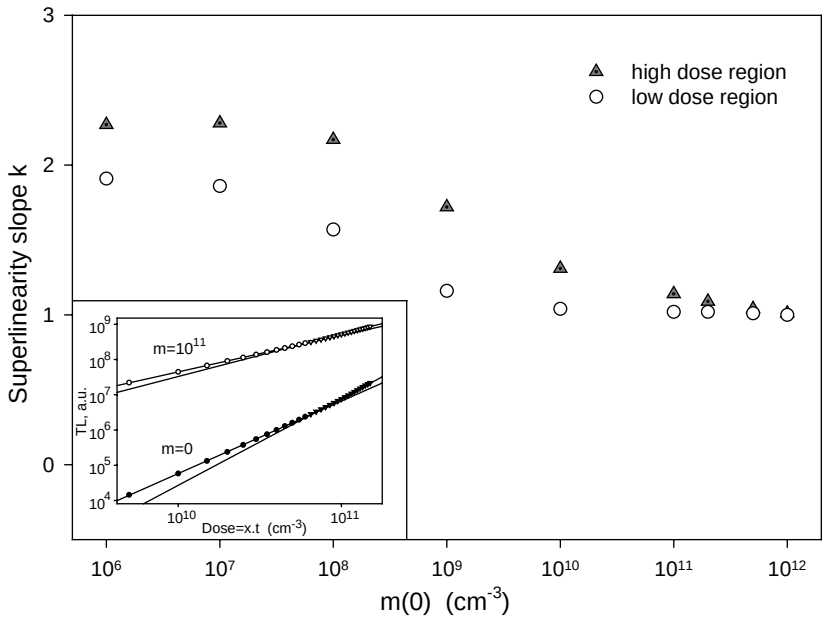

Fig. 7. The superlinearity slope k calculated from the two superlinearity regions of Fig. 5. This is to be compared with Fig. 6(b) in this paper, which shows the data of Charitidis et al. (2000). The inset shows two of the graphs of Fig. 5, indicating clearly the two regions of superlinearity, and the range of doses where the two superlinearity slopes k are being calculated.

4. Conclusions

The model of Zimmerman (1971a) with the amendment by Chen (1979) as further discussed by Chen and Leung (1999) has now been used to describe the observed behavior of the superlinearity of the "110°C" TL peak as a function of the annealing temperature, as measured experimentally by Charitidis et al. (2000). It is found that by varying the initial concentration of holes in the recombination center $m(0)$, the model reproduces most of the features observed experimentally, namely the existence of two superlinearity regions which exhibit a distinct behavior as a function of the annealing temperature. The model also predicts the correct quantitative behavior of the superlinearity slopes k in each superlinearity region, and the correct order of magnitude for the sensitivity changes occurring by high temperature annealing.

Furthermore, the same modified Zimmerman model provides for a natural explanation of the pre-dose results of Charitidis et al. (1999). The same electronic mechanism explains the fact that the superlinearity effects can also be removed by delivering a high pre-dose to the samples. By varying the predose irradiation time, the model predicts the correct quantitative behavior for the superlinearity slope k, as well as the correct order of magnitude for the sensitivity changes occurring by predose irradiation.

The parameters of the model used in this paper are certainly not unique, and the value of the model lies in the fact that it correctly describes the complex shape and behavior of the TL growth curves at various annealing temperatures and predoses. It also provides further supporting evidence for the validity of the modified Zimmerman model, on explaining the superlinearity and sensitivity changes occurring in synthetic quartz.

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