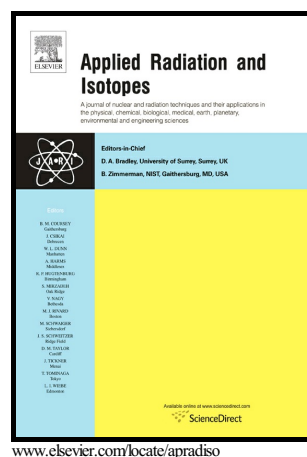# Author's Accepted Manuscript

New algorithm for analysing phosphorescence curves: its application to the K_2YF_5:Pr compound

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New algorithm for analysing phosphorescence curves:

its application to the K_2YF_5:Pr compound.

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Abstract

Analysis of phosphorescence curves is an alternative procedure to glow curve analysis in order to find the parameters characterizing trap and recombination centers of thermoluminescence materials. In this article a new algorithm to analyze phosphorescence curves is reported, which is derived from the set of coupled differential equation describing the carrier traffic without resorting to approximations. The new algorithm has been employed to analyze the phosphorescence curves of the K_2YF_5:Pr compound.

Keywords: phosphorescence; trap parameters; curve analysis

Introduction

The analysis of phosphorescence curves consists of three steps: 1) a model consisting of trap and recombination centers is devised based on the experimental findings at hand, 2) a theoretical expression $I_{th}(t,\alpha)$ is derived for the phosphorescence light emitted at a given temperature. $I_{th}(t,\alpha)$ contains the parameters to be found, which are indicated with α, and t stands for the time, and 3) the theoretical curve is then fitted to the experimental one by adjusting the parameters. The fitting is acceptable if the factor of merit (*FOM*) given by

$$FOM = \frac{\sum_{j=1}^{L}\left|I_{th}(t_j,\alpha) - I_{exp}(t_j)\right|}{\sum_{j=1}^{L}\left|I_{exp}(t_j)\right|}.100\% \tag{1}$$

is less than 5% (Horowitz and Yossian, 1995). In Eq. (1) $I_{exp}(t)$ stands for the experimental phosphorescence curve, and L for the number of sampling points of the glow curve.

The simplest model for describing thermoluminescence is that shown in figure 1. It consists of a trap center and a recombination center in the band gap of a crystal. Irradiation of a sample raises valence

electrons into the conduction band (CB). Some of the electrons in the CB will be capture by the trap. Later, upon heating electrons in the trap are freed into the conduction band, from where they recombine in a recombination center (RC) or are trapped again. This model is known as one trap-one recombination model (OTOR).

For the OTOR model the most employed kinetics for analyzingthermoluminescence and phosphorescence curves is the general order kinetics (GO). This kinetics is a heuristic expression, which has been put forward by May and Partridge (May and Partridge, 1964). The starting equation is:

$$I(t) = n^b \cdot s \cdot exp\left(-\frac{E}{kT}\right) \tag{2}$$

E is the activation energy, also known as trap depth, s is called the frequency factor, and the parameter b is dubbed kinetics order. It is loosely related to the retrapping of electrons (or holes). k is the Boltzmann constant. n is the concentration of trapped electrons.

In Eq. 2 the expression $s \cdot exp\left(-\frac{E}{kT}\right)$ is the escape probability of an electron (or hole) from a trap. It is

usually indicated with p, namely, $p = s \cdot exp(-\frac{E}{kT})$.

Eq. 2 is a heuristic interpolation between first and second order kinetics, namely:

Between $I(t) = n \cdot s \cdot exp\left(-\frac{E}{kT}\right)$ (first order kinetics)

and $I(t) = n^2 \cdot s \cdot exp\left(-\frac{E}{kT}\right)$ (second order kinetics)

Both kinetics are derived by assuming that the concentration of electrons in the conduction band is negligible (qusi-equilibrium approximation).

For $b \to 1$ Eq. 2 converges to the first order kinetics (FO), and for $b = 2$, to the second order kinetics (SO). A problem with Eq. (2) is that the units of the parameter s changes with the kinetics order b. To overcome this problem Rasheedy (Rasheedy, 1993) proposed the following expression

$$I(t) = \frac{n^b}{N^{b-1}} \cdot s \cdot exp\left(-\frac{E}{kT}\right) \tag{3}$$

N is the concentration of traps.

Phosphorescence curves are obtained by heating suddenly a sample to a given temperature, say $T0$, and then keeping the sample at that temperature until the signal becomes negligible.

From Eq. 3 it results for the phosphorescence light:

$$I(t) = \frac{n_0^b}{N^{b-1}} \cdot p \cdot \left[1 + (b-1) \cdot p \cdot n_0^{b-1} \cdot t\right]^{\frac{-b}{b-1}} \tag{4}$$

If three or more phosphorescence curves are recorded at different temperatures in the range of a glow peak, by fitting expression (4) to each of the recorded phosphorescence curve the value of p for each temperature can be found. Later, the slope of the expression $ln(p)$ against $(-1/kT)$ is the activation energy. From the intercept of the straight line with the $ln(p)$ axis the frequency factor s can be found. In this way the activation energy and frequency factor of a trap can be determined (Chen, 1997). Usually the fitting is performed by resorting to algorithms such as the Levenberg-Marquardt method (Horowitz, 1995).

Recently it has been reported that the GO kinetics is not appropriate for analyzing phosphorescence curves [Ortega et al., 2013].

In this paper we report a new algorithm, which is derived from the set of differentials equations describing the carrier traffic among the trap center, the recombination center and the bands. We employed it for analyzing phosphorescence curves of the K_2YF_5:Pr compound.

Derivation of the new kinetics

In order to derived the new algorithm we resort to a more general model. It is shown in Fig. 2. This model, known as non interactive multitrap system (NMTS), contains an additional trap named thermally disconnected trap. This trap is fully occupied, and for the temperature range a sample is heated electrons are not raised into the conduction band.

In Fig. 2 N stands for the concentration of active traps, n for the concentration of trapped electrons in active traps, M for the concentration of thermally disconnected traps, H stands for the concentration of recombination centers, and h is the concentration of holes in the recombination centers. A_n stand for the retrapping probability, p is the released probability from the active trap, and A_m is the recombination probability. CB and VB indicate the conduction and valence band respectively.

The equations describing the phosphorescence kinetics of the model shown in Fig. 2 are:

$$\frac{dn(t)}{dt} = -p \cdot n(t) + A_n \cdot [N - n(t)] \cdot n_c(t) \tag{5}$$

$$\frac{dh(t)}{dt} = -A_m \cdot h(t) \cdot [h(t) - n(t) - M] \tag{6}$$

$$n_c(t) = h(t) - n(t) - M \tag{7}$$

$$I(t,T) = -\frac{dh(t)}{dt} = A_m \cdot h(t) \cdot n_c(t) \tag{8}$$

$I(t,T)$ stands for the phosphorescence intensity recorded at temperature T.

Integrating the relationship $\frac{dh(t)}{dt} = -I(t,T)$, we obtain

$$h(t) = h(t=0) - \int_0^t I(u,T).du = h(t=0) - F(t,T) \tag{9}$$

The function $F(t,T)$ stands for $F(t,T) = \int_0^t I(u,T).du$, namely, $F(t,T)$ is the integral of the

phosphorescence intensity recorded at the temperature T between $t=0$ and t.

When the phosphorescence light becomes negligible both the concentration of trapped electrons and the concentration of holes in the recombination centers are nearly zero, namely,

$n_c = n0 \approx 0$

Then according to the Eq. (7)one has $h(\infty) = M$ .Substituting in Eq (9) we get:

$$M = h(t=0) - F(\infty) = h(t=0) - A \qquad (10)$$

where A is the area under the curve $I_{exp}(t,T)$,i. e., the area of the phosphorescence curve recorded at temperature T.

then

$$h(t=0) = M + A \qquad (11)$$

Finally we have:

$$h(t) = M + A - F(t,T) \qquad (12)$$

From Eqs. 8 we have:

$I(t,T) = A_m \cdot h(t) \cdot n_c(t)$

Then

$$n_c(t) = \frac{I(t,T)}{A_m [M + A - F(t,T)]} \qquad (13)$$

If in this equation $I(t,T)$ is replaced by $I_{exp}(t,T)$ we have:

$$n_c(t) = \frac{I_{exp}(t,T)}{A_m [M + A - F(t,T)]} \qquad (14)$$

Inserting this equation into equation 5, and making the approximation $n << N$, equation 5 becomes:

$$\frac{dn(t)}{dt} + p \cdot n(t) = A_n \cdot \frac{N \cdot I_{exp}(t,T)}{A_m \cdot [M + A - F(t,T)]} \tag{15}$$

Multiplying both sides of Eq. 15 by $e^{p \cdot t}$ we have:

$$e^{p \cdot t} \frac{dn(t)}{dt} + e^{p \cdot t} p \cdot n(t) = e^{p \cdot t} A_n \cdot \frac{N \cdot I_{exp}(t,T)}{A_m [M + A - F(t,T)]} \tag{16}$$

$$\tag{16}$$

Since

$$e^{p \cdot t} \frac{dn}{dt} + e^{p \cdot t} p \cdot n = \frac{d}{dt}\left(e^{p \cdot t} \cdot n\right) \tag{17}$$

Integration of Eq. 16 yields

$$n(t) = n_0 \cdot e^{-p \cdot t} + R \cdot N \int_0^t \frac{I_{exp}(u,T) \cdot e^{p(u-t)} du}{[M + A - F(u,T)]} \tag{18}$$

R stands for A_n/A_m.

From equations (8), (12) and (13) the following relationship for the theoretical light can be derived:

$$I(t,T) = A_m \cdot \left(M + A - F(t,T)\right) \cdot \left(A - F(t,T) - n(t)\right) \tag{19}$$

Eq. 19, along with Eq. 18, permits the calculation of the phosphorescent intensity for a given set of parameters.

Some remarks should be made at this point: 1) the insertion of the experimental light intensity $I_{exp}(t,T)$ into equation 13 allows getting an equation for the theoretical light intensity $I_{th}(t,T)$, 2) equation 19 is a self-consistent equation because parameters should be found so that $I_{th}(t,T)$ fits $I_{exp}(t,T)$, and $I_{exp}(t,T)$ is contained in equation 18,3) the experimental intensity is measured at sampling times. Fitting algorithms, such as the Levenberg-Marquardt, require that both $I_{th}(t,T)$ and $F(t,T)$ be continuously

differentiable functions. In the calculations below both functions were approximated with cubic splines.

Thealgorithm can be extended to a model of r active traps and several thermally disconnected traps. Then Eq. 19 reads:

$$I(t,T) = A_m \cdot \big(M + A - F(t,T)\big) \cdot \big(A - F(t,T) - n_1(t) - n_2(t) - \ldots - n_r(t)\big) \quad (20)$$

where $n_i$stands for the concentration of electrons in trap#i.

In this case M stands for the net concentration of charge in the thermally disconnected traps. A remark should be made about how Eq. 20 should be employed in order to find the set of parameters α. Equation 19 gives the intensity in counts per second. Since usually the TL readers give the light intensity in Ampers, to perform a fitting Eq. 19 should be written

$$I(t,T) = C \cdot A_m \cdot \big(M + A - F(t,T)\big) \cdot \big(A - F(t,T) - n_1(t) - n_2(t) - \ldots - n_r(t)\big)$$
$$(21)$$

The constant C takes into account the proportionality between the intensity given by counts/second and the recorded intensity given by the TL readers in Amper.

Eq. 21 can be rewritten:

$$I(t) = \lambda \cdot \big(C \cdot M + C \cdot A - C \cdot F(t,T)\big) \cdot \big(C \cdot A - C \cdot F(t,T) - C \cdot n_1(t) - C \cdot n_2(t) - \ldots - C \cdot n_r(t)\big)$$
$$(22)$$

where $\lambda = A_m/C$

C can be chosen so that the concentration of trapped electrons just before the recording of a glow curve, namely, $C \cdot n_0$, be given in units of area. Thus $C \cdot n_0$is the area of the glow curve. By the same token $C \cdot n_{0i}$ is the initial concentration of trapped electrons in trap #i also given in units of area. Because of this change of units the concentration M of deep traps is also

given in units of area, and $C \cdot F(t,T) = \int_0^t I_{exp}(u,T)du$ is the area of the recorded glow curve

$I_{exp}(t,T)$ between the $t=0$ and t. This metric for giving the concentration of carriers in units

of area is not new. It has been put forward by Rashhedy (Rasheedy 1996). The advantage of

this metric is that the initial concentration of trapped electrons n_0 is known, namely, it is

given by sum of both the area of the glow curve and the residual glow curve, as explained

below.

Analysis of the phosphorescence curves of the high temperature peak of $K_2YF_5:Pr^{3+}$ compounds

The samples were irradiated with a Sr-90 source. At the position of the samples the dose

rate was 22mGy/min. The samples were irradiated during 10 minutes. Thus the dose was

220 mGy. Glow and phosphorescence curves were recorded with a Harshaw 3500 reader.

Figure 3 shows a glow curve of the $K_2YF_5:Pr^{3+}$ compound recorded with a heating rate of 1

K/s.

To determine the number of traps Tm-Tstop measurements were performed [McKeever].

Figure 4 shows that there are two traps.

In order to record phosphorescence curves related to the high temperature peak the

following procedure was follows: 1) the samples were first heated up to 648Kand

maintained at that temperature for 1 min, and allowed to cool down to room temperature.

This was made for erasing any residual signal. 2) the samples were irradiated, 3) since the

maximum of the high temperature peak lies at 496 K the samples were heated up to 425 K,

followed by an annealing at this temperature for 10 minute, and let it cool down to room

temperature in order to empty the trap related to the low temperature peak, and 2) after the

annealing a phosphorescence curve was recorded at 463 K, 3) similar procedures were

carried out to record phosphorescence curves at 483 and 503 K.

The trap related to the low temperature peak, named trap 1, does not affect the phosphorescence curves at 463, 483 and 503 K because of the following reasons: 1) in Eq. 22 $n_1(t) \approx 0$ after the thermal cleaning, and 2) according to figure 7 of the article by Marcazzó et al. 2004the position of the peaks do not change with dose, which indicates that retrapping is negligible against recombination for both traps, i.e., $R_i \approx 0$ for i=1, 2. Thus the electrons released by the trap related to the high temperature peak will mainly recombine instead of being trapped by the low temperature trap, and therefore the latter will remain nearly empty during the recording of the phosphorescence curves. As a consequence, equation 19 can be used for analyzing the phosphorescence curves related to the highest temperature peak.

Figure 5 shows the recorded curves. A sample was heated with a heating rate of 50 K/s up to the temperature selected for recording the phosphorescence. The phosphorescence curves have been obtained from the curves shown in figure 5 by considering the curves two seconds after the instant at which the intensity is maximal.

After having recorded a phosphorescence curve, the sample was allowed to cool down to room temperature. Immediately a glow curve was recorded. This means that after the measurement of the phosphorescence curve some electrons remained in the trap. Thus the value of $C·A$ in Eq. 21 is the sum of the area of the phosphorescence curve plus the area of the residual glow curve.

The phosphorescence curves were fitted employing the new algorithm.   The three phosphorescence curves, namely, $I_{exp}(t,463\ K)$, $I_{exp}(t,483\ K)$ and $I_{exp}(t,503\ K)$ were approximated with cubic splines. From the three $I_{exp}(t,T)$ the three $F(t,T)$ were computed, and approximated also with cubic splines. The fitting was accomplished using the Levenberg-Marquardt algorithm.

Table I show the resulting parameters, and figures 6-8 the phosphorescence curves and those resulting from the fitting.

From the data in table I the activation energy is 1.32 eV, and the frequency factor to 1.4 10^{12} s^{-1}. Furetta et al., 2007, reported E= 1.25 eV, and s = 2.2 10^{10} s^{-1}. The last values were obtained with the GO kinetics.

Fig. 9 depicts the concentration n of trapped electrons, and the concentration n_c of electrons in the conduction band for T= 483 K. n was computed with Eq. 18, and n_c is given by:

$$n_c(t) = A - F(t,T) - n(t)$$

As can be seen, n_c is larger than n. It should be remembered that $t = 0$in Fig. 9 corresponds to the time t in Fig. 4 two seconds after the instant at which the intensity is maximal. Because of the sudden temperature rise (see figure 5) a large number of electrons are injected into the conduction band. If the recombination rate is not high enough electrons accumulate in the conduction band.

Because the concentration of electrons in the conduction band is not negligible, the GO kinetics yields wrong values (Ortega et al., 2013).

Summary

An expression for the light intensity has been derived from the set of coupled differential equations describing the carrier traffic for the NMTS model. This expression makes feasible the use of algorithms, such as the Levenberg-Marquart, for fitting the theoretical phosphorescence curves to the experimental ones. Eq. 19 was derived for the case $n << N$. In fact, as shown in the appendix, an expression for the phosphorescence can be derived without approximations. The reason for employing Eq. 19 is twofold: first, as reported in many articles, the dose given to samples in order to analyze phosphorescence curves is low, and second, the computational times are lower if Eq. 19 is employed instead of the expression derived in the appendix.

The new kinetics gives for the high temperature trap an energy of 1.32 eV and a frequencyfactor of 1.4 10^{12} 1/s. Both values differ from the values obtained with the GO kinetics, namely,1.25 eV and 2.2 10^{10} 1/s.

Finally it is worth mentioning that the algorithm put forward in this article takes into account, if necessary, the interaction among traps.

Appendix

Equation 5 can be rearranged as follows:

$$\frac{dn(t)}{dt} + \left(p + A_n \cdot n_c(t) \right) \cdot n = A_n \cdot N \cdot n_c(t) \qquad \text{(A1)}$$

Since

$$n_c(t) = \frac{I_{exp}(t,T)}{A_m \left[M + A - F(t,T) \right]} \qquad \text{(A2)}$$

then

$$\frac{dn(t)}{dt} + \left(p + A_n \frac{I(t,T)}{A_m \left(M + A - F(t,T) \right)} \right) \cdot n(t) = A_n \cdot N \frac{I(t,T)}{A_m \left(M + A - F(t,T) \right)};$$

$$R = \frac{A_n}{A_m} \Rightarrow$$

$$\frac{dn(t)}{dt} + \left(p + R \frac{I(t,T)}{\left(M + A - F(t,T) \right)} \right) \cdot n(t) = R \cdot N \frac{I(t,T)}{\left(M + A - F(t,T) \right)}$$
$$\text{(A3)}$$

Defining the integration factor

$$U(t) = exp \left[\int_0^t du \left(p + R \cdot \frac{I(u,T)}{\left(M + A - F(u,T) \right)} \right) \right] \qquad \text{(A4)}$$

Note that

$$\frac{dU(t)}{dt} = U(t) \cdot \left(p + R \cdot \frac{I(t,T)}{(M + A - F(t,T))} \right)$$
(A5)

substituting in (A3) it results

$$\frac{dn(t)}{dt} + \frac{1}{U(t)} \cdot \frac{dU(t)}{dt} \cdot n = R \cdot N \frac{I(t,T)}{(M + A - F(t,T))}$$
(A6)

We have

$$U(t) \cdot \frac{dn(t)}{dt} + \frac{dU(t)}{dt} \cdot n(t) = R \cdot N \cdot U(t) \cdot \frac{I(t,T)}{(M + A - F(t,T))}$$

then

$$\frac{d}{dt}(U \cdot n) = R \cdot N \cdot U \cdot \frac{I}{(M + A - F)}$$

By integration

$$U(t) \cdot n(t) - n_0 = R \cdot N \cdot \int_0^t du \cdot U(u) \frac{I(u,T)}{(M + A - F(u,T))} \Rightarrow$$

$$n(t) = \frac{n_0}{U(t)} + \frac{R \cdot N \cdot \int_0^t du \cdot U(u) \frac{I(u,T)}{(M + A - F(u,T))}}{U(t)}$$
(A7)

Now we calculate $U(t)$. First its exponent

$$\left[\int_0^t du \left(p + R \cdot \frac{I(u,T)}{(M + A - F(u,T))} \right) \right] = p \cdot t + \int_0^t du \left(R \cdot \frac{I(u,T)}{(M + A - F(u,T))} \right)$$
(A8)

Since $F(t,T) = \int_0^t I_{exp}(u,T) du$, we have:

$$\int_{0}^{t} du \left(R \cdot \frac{I(u,T)}{(M+A-F(u,T))} \right) = \int_{F(0)}^{F(t)} dF \left(\frac{R}{(M+A-F)} \right) \tag{A9}$$

$$\int_{F(0)}^{F(t)} dF \left(R \cdot \frac{1}{(M+A-F)} \right) = R \cdot ln \left(\frac{M+A}{M+A-F} \right) = ln \left(\frac{M+A}{M+A-F} \right)^{R} \Rightarrow$$

$$U(t) = \left(\frac{M+A}{M+A-F(t,T)} \right)^{R} \cdot exp(p \cdot t)$$
(A10)

Finally, replacing (A10) in (A7) is obtained:

$$n(t) = \left[\frac{n_{0}}{(M+A)^{R}} + N \cdot R \cdot \int_{0}^{t} du \cdot \frac{I(u,T) \cdot exp(p \cdot u)}{(M+A-F(u,T))^{R+1}} \right] \cdot (M+A-F(t,T)^{R} \cdot exp(-p \cdot t)$$
(A11)

This expression is valid whatever the dose.

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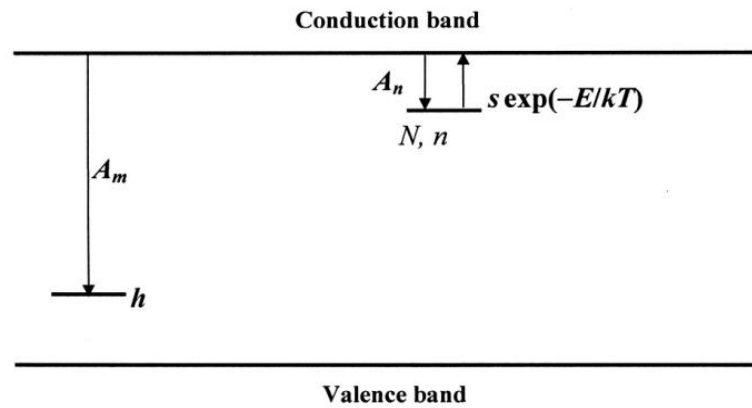
Figure 1: OTOR model. *N* stands for the concentration of traps, *n* for the concentration of trapped electrons, *h* for the concentration of holes in the recombination center, A_n and A_m for the retrapping and recombination probabilities respectively.

CB

A_n p

N,n

A_m M

H, h

VB
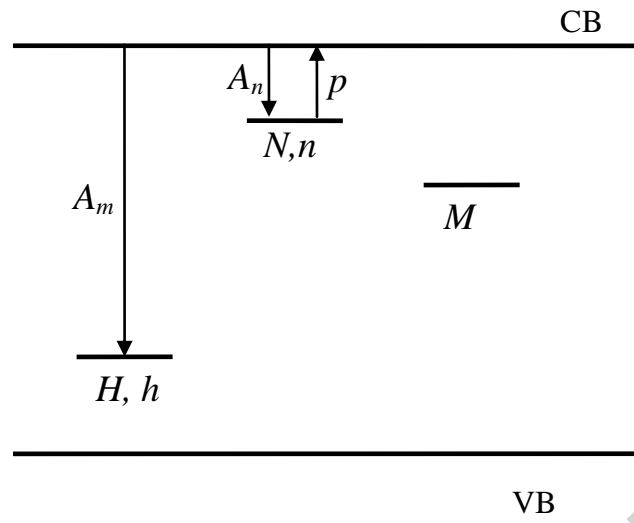
Figure 2: NMTS model.
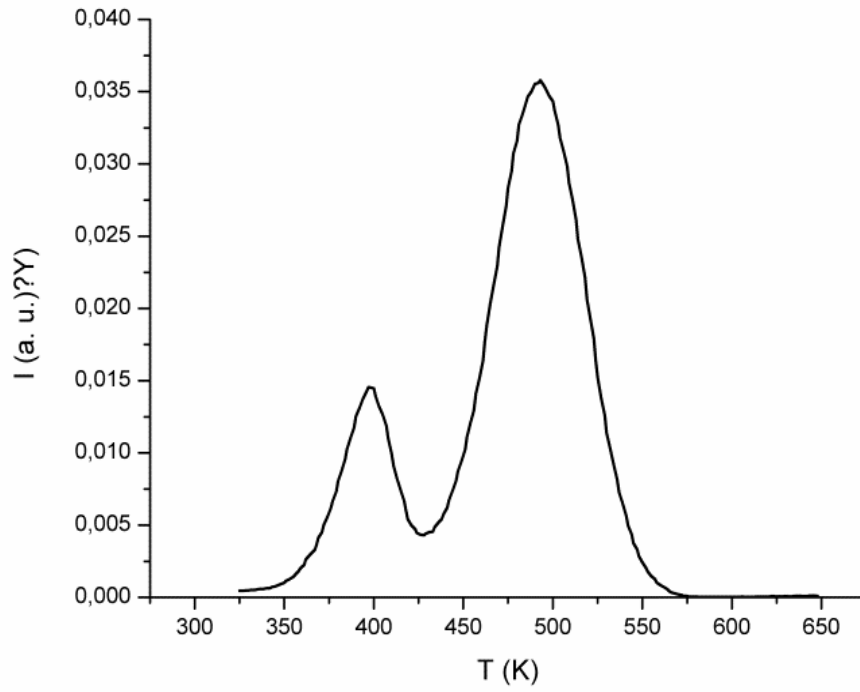
Figure 3: glow curve recorded with a heating rate of 1 K/s. The dose was 220 mGy.
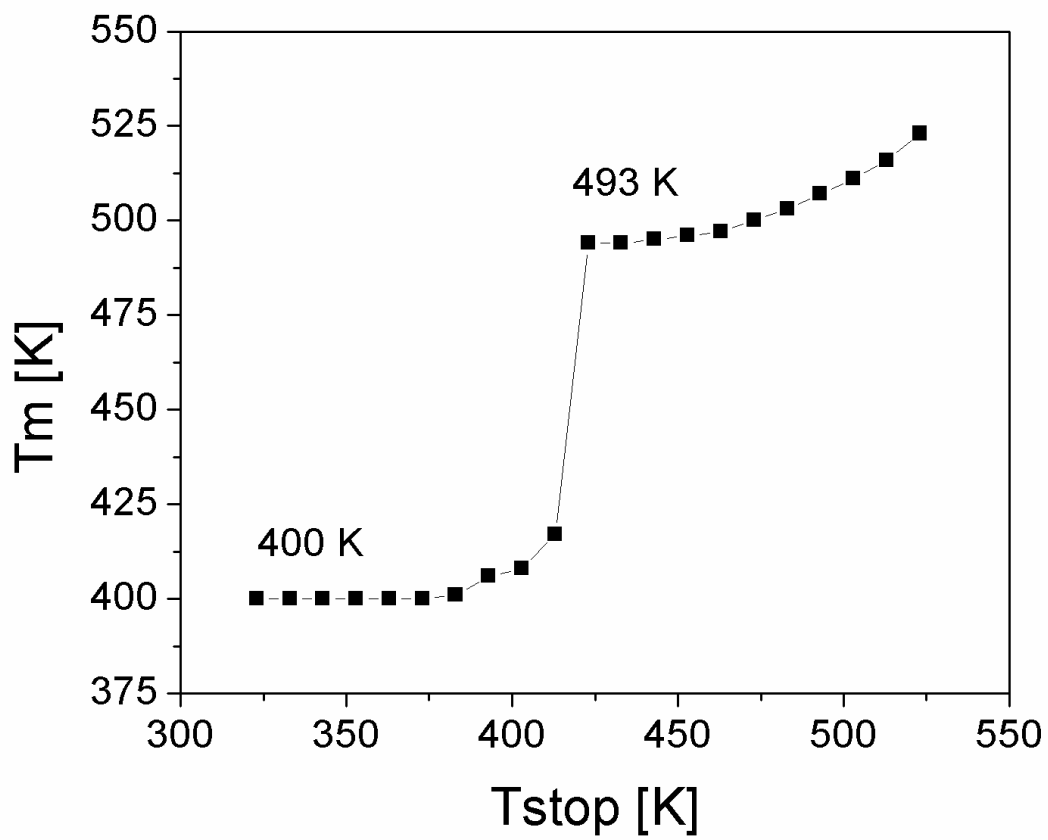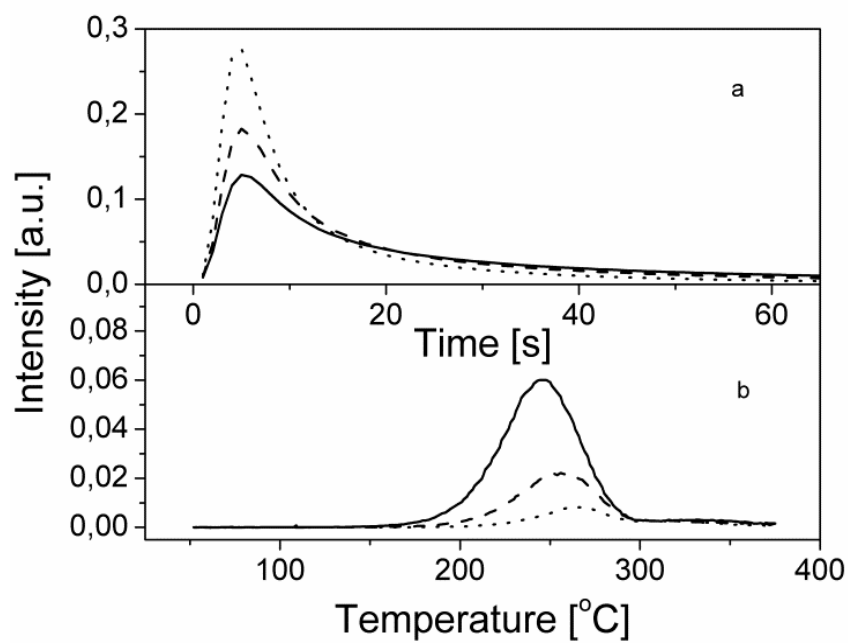
Figure 4: Tm-Tstop experiment.

Figure 5: recorded phosphorescence (a) and residual thermoluminescence glow curves (b).
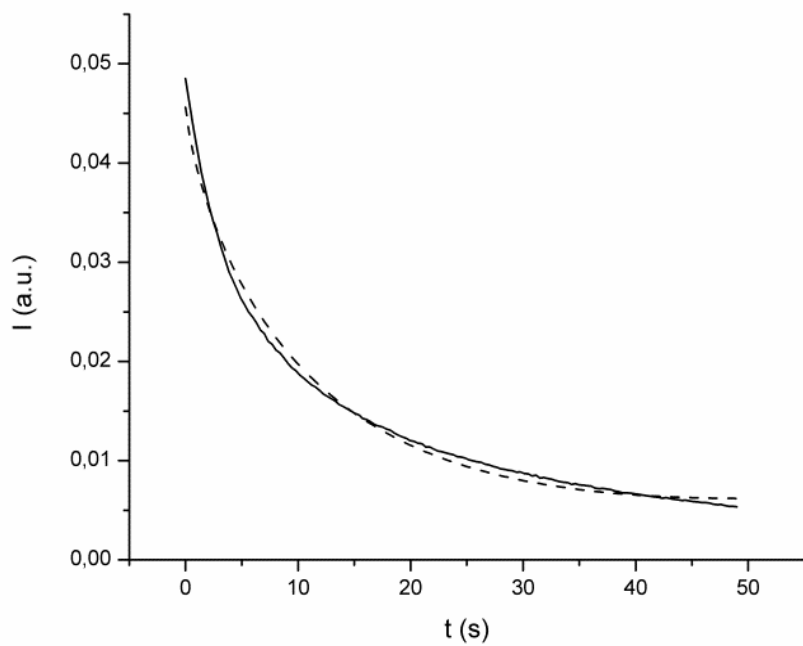T= 463 K (solid line), T= 483 K (dash line), and T= 503 K (dot line).

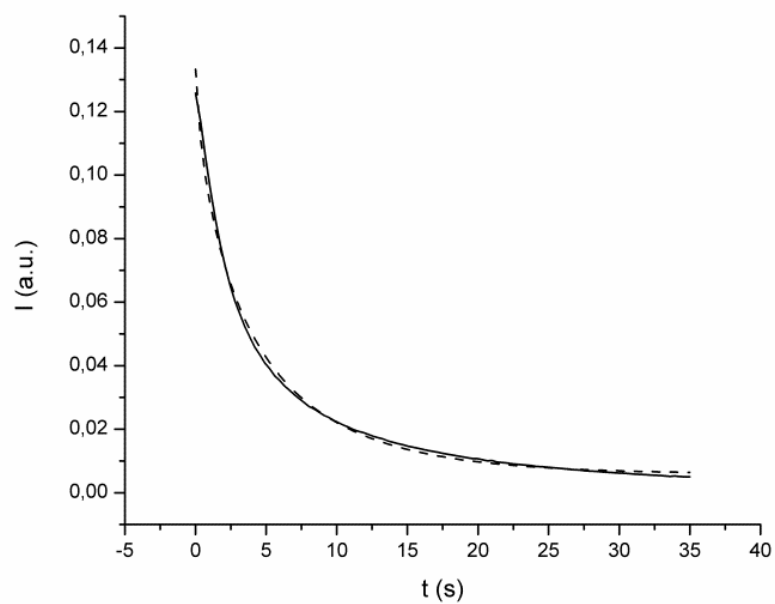Figure 6: Phosphorescence curve for T=463 K. Experimental curve (solid line), fitted curve (dash line)

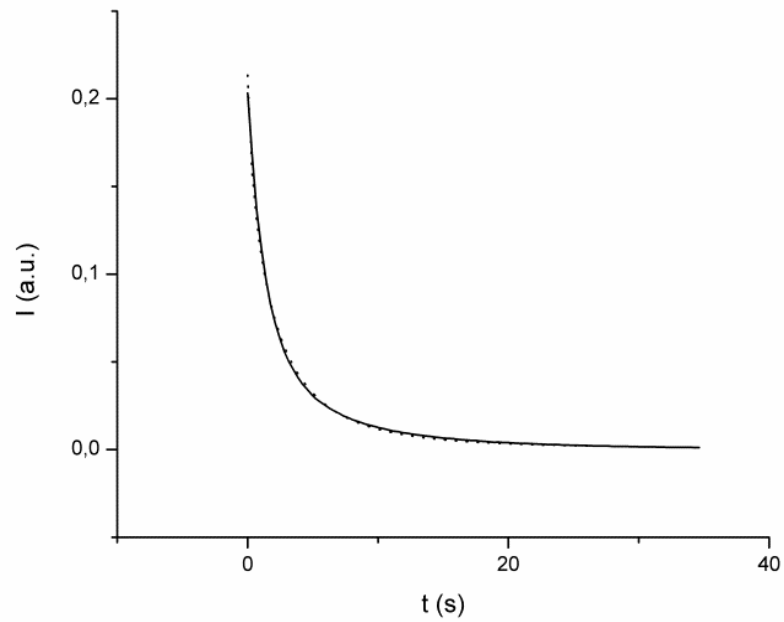Figure 7: Phosphorescence curve for T=483 K. Experimental curve (solid line), fitted curve (dash line)

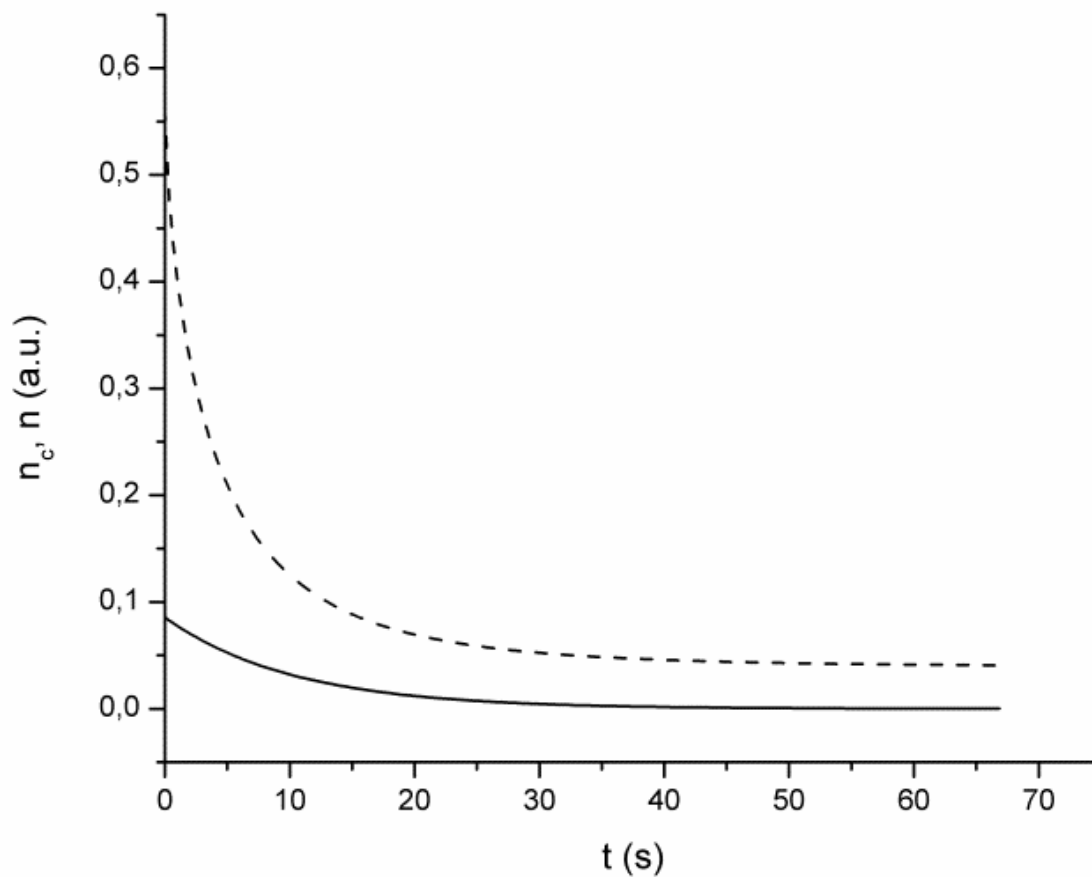Figure 8: Phosphorescence curve for T=503 K. Experimental curve (solid line), fitted curve (dash line)

Figure 9: concentration of trapped electrons (solid line), and concentration of electrons in the conduction band (dash line) as function of time for the phosphorescence curve recorded at T= 503 K.

Table I: parameters resulting from the fitting of Eq. 19 to the three phosphorescence curves recorded at 463, 483, and 503 K. In particular, $C.A$ is the sum of the area of a phosphorescence curve plus the area of the residual thermoluminescence curve.

$T(K)$	$C{\cdot}A$	n_0	$p \ (s^{-1})$	R	M	$FOM \ (\%)$
463	1.585	1.153	$7.1 \ 10^{-3}$	0	0	5.0
483	1.028	0.530	$26.0 \ 10^{-3}$	0	0	4.9
503	0.646	0.085	$98.0 \ 10^{-3}$	0	0	5.0

Highlights
Analysis of phosphorescence curves
Novel algorithm derived without approximations
Interaction among traps taken into account