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Optically stimulated luminescence studies on CaF_2:N using CW and LM OSL techniques

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Abstract: N*atural calcium fluoride (fluorite - CaF_2:N) exhibits multiple LMOSL peaks when stimulated using blue LED's, which makes it an unique phosphor. Such a property is not observed experimentally in any other dosimetric phosphors namely Al_2O_3:C, BeO etc. This makes CaF_2:N as an effective tool not only for testing the kinetic models of luminescence but also for investigating OSL using CW and LM stimulation modes. In view of this, CW and LM OSL processes have been investigated for CaF_2:N and the effect of optical bleaching and pre-readout annealing on various components of OSL have also been studied. It has been found that CWOSL from CaF_2:N could be fitted as a sum of four first order decaying exponentials (components) and the photo-ionization cross-sections for these four components were found to be ~$2.77*10^{-17}cm^2$ (σ_1), ~$0.90*10^{-17}cm^2$ (σ_2), ~$0.16*10^{-17}cm^2$ (σ_3) and ~$0.03*10^{-17}cm^2$ (σ_4) respectively.*

Effect of optical bleaching using blue LEDs on CWOSL curves was also studied. It has been found that for stimulation power of 22.5 mW cm^{-2} using blue LEDs, the first component of OSL is bleached in ~ 5-15 s. Further for optical bleaching times of ~ 20-25 s, only two components are observed. Similar studies were carried out for LMOSL and it has been found that the first peak in LMOSL from CaF_2:N is bleached in 5-15 s using blue LEDs whereas the second peak which is a broader peak, shifts toward higher side with increase in optical bleaching time. CW and LM OSL studies were also carried out after performing pre-readout annealing at 120^{o}C and 200^{o}C for 10 s. It has been found that after optical bleaching or pre-readout thermal annealing, the photo-ionization cross-sections of the remaining components are closer to the corresponding component values obtained by fitting CWOSL curve as a sum of four order exponentials. Transformation of CW to LM OSL curves was carried out as per: i) Bulur's method, and ii) Bos & Wallinga method. These results indicate that the behavior of the transformed LM OSL curves is nearly similar to the LM OSL curves recorded experimentally.

Keywords: *TL glow curve, CW & LM OSL curves; CaF_2:N; optical bleaching; decay constants; ionization cross-section and Transformation techniques.*

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1.0 INTRODUCTION

Continuous wave (CW) and linearly modulated (LM) optically stimulated luminescence (OSL) processes are commonly used for the estimation of various kinetic parameters such as photo-ionization cross-section, retrapping and recombination cross-sections and order of kinetics. Although CW and LM OSL represent the same physical information (under different excitation/stimulation profiles), LM OSL appears peak shaped visually like a thermoluminescence (TL) glow curve. This is because in LM OSL process, peaks corresponding to each trap participating in the luminescence process may be recorded if photo-ionization cross-sections differ widely. Traps having single/closer values of photo-ionization cross-section, however, generally yields narrow/broader LM OSL curves. This could explain why multiple LM OSL peaks are not observed experimentally in many popular OSL phosphors such as Al_2O_3:C, BeO *etc*. although recorded LM OSL curve can further be decomposed into multiple LM OSL curves through curve fitting or using transformation of CW to LM OSL curves using various transformation techniques [1-5]. Also it is worth mentioning that OSL is a popular technique nowadays and various phosphors and associated reader systems are being developed and studied [6-8]. CaF_2 based detectors have been studied comprehensively in the last fifty years and the studies on various aspects of luminescence and dosimetry still continue [9-21]. A survey of literature for the last decade shows lot of research papers on CaF_2 based materials (natural as well as synthetic) and summary of few of the studies are worth mentioning. Chougaonkar and Bhatt studied blue light stimulated luminescence in natural calcium fluoride along with its characteristics and implications in radiation dosimetry [11]. Polymeris *et al.* studied correlation between TL and OSL properties of natural CaF_2. [12]. Yazici and co-workers performed extensive studies and analysis of dosimetric thermoluminescent glow peak of CaF_2:Mn after β-irradiation as well as various dosimetric studies on natural CaF_2 [13-16]. Massillon *et al.* studied thermoluminescent response and glow curve induced by γ-rays and ions in CaF_2:Tm (TLD-300) dosimeters [17]. Bakshi *et al.* studied various dosimetric & luminescence (TL & OSL) characteristics of CaF_2:Mn phosphor material [18]. Zahedifar *et al.* studied the thermoluminescence characteristics of the novel CaF_2:Dy nano-particles prepared by using the hydrothermal method [19]. More recently, Ferreira *et al.* studied the correlation of optically and thermally stimulated luminescence of natural fluorite pellets [20]. This study examined the behaviour of TL and OSL (stimulated with Blue LEDs) signals from the Brazilian natural fluorite pellets with NaCl as binding agent, as well as their correlations, in order to study and optimize the dosimetric process with this material [20]. In addition, Cemre [21] has also investigated optically stimulated luminescence studies on natural fluorites, whereas Yoshimura and Yukihara have also reported OSL studies on fluorites [22].

In view of above, it may be seen that limited studies have been performed on the CW and LM OSL properties of CaF_2: natural [11-12, 20-22]. It is also important to note that CaF_2: natural (CaF_2:N) exhibits two (visible) LM OSL peaks when stimulated using blue LED's [11]. This property of natural calcium fluoride (fluorite) makes it an unique phosphor in the sense that such a property is not commonly observed in popular dosimetric phosphors namely Al_2O_3:C, BeO *etc*. This makes as an effective tool not only for testing the kinetic models of luminescence but also for investigating OSL using CW and LM stimulation modes. In view of this, CW and LM OSL processes have been investigated comprehensively for CaF_2:N and the effect of optical bleaching and pre-readout annealing on various components of OSL have also been investigated. Preliminary studies on transformation techniques popularly used to convert CW to LM OSL curves have also been investigated in this study.

As mentioned earlier, OSL process in CaF_2:N provides an effective tool for testing the kinetic models of OSL and associated features such as transformation of CW to LM OSL techniques. In view of this, kinetic analysis of CW and LM OSL curves has been carried out for CaF_2:N. The photo-ionization cross-section of the traps participating in the luminescence process is also measured. The effect of optical bleaching as well as pre-readout annealing on CW and LM OSL curves is studied and correlation between CW and LM OSL curves is studied using transformation techniques proposed by Bulur and Bos & Wallinga [1-5]. The respective LMOSL curves obtained from the transformation of CW OSL curves are also compared with the experimentally obtained LM OSL curves.

2. 0 TRANSFORMATION TECHNIQUES

Transformation of CW to LM OSL curves is necessary to equate CW and LM stimulation processes and may be helpful in evaluating various kinetic parameters. Agreement between these techniques may indicate the validity of simple trap model however disagreement may result from large number of factors. Also, the transformation from CW to LM OSL is required because the CW OSL always appear to be a featureless decaying curve. There are two techniques which are used to convert CW to LM OSL curves and were suggested by Bulur and Bos & Wallinga [3-5]. The transformation techniques are described below:

i). Bulur's transformation technique

The luminescence output in a CW OSL measurement for first order kinetics can be written as [1-4]

$$I(t) = n_0 b \exp(-bt) \qquad (1)$$

Where n_0 is initial number of trapped electrons, b is a constant describing the decay of luminescence curve and is proportional to detrapping probability α and the stimulation light intensity I_0 ($b = \alpha I_0$). In order to convert the CW OSL curve to a LM OSL curve, one may introduce a new independent variable u, which is defined as [3-4]

$$u = \sqrt{2tP} \quad \text{or} \quad t = \frac{u^2}{2P} \qquad (2)$$

where u has the units of second like time t and the measurement period P of the LM OSL experiment. Substituting Eq. (2) to Eq. (1) and further multiplying by u/P one obtains [4]

$$I(u) = n_0 \frac{b}{P} u \exp(-\frac{b}{2P} u^2) \qquad (3)$$

This transformation suggested first by Bulur may be helpful for obtaining the LM OSL curve in cases where linear modulation of OSL is not easily achievable. It can also be used to compare transformed LMOSL curves with actual experimental curves. Bulur also suggested that in order to use all the available data in the CW OSL curve, it is a good choice to have P = 2t where t is the total measurement time of the CW OSL data which can be deduced from equating the total light intensity impinging on the sample as well. Also the stimulation power and acquisition time for CW and LM OSL measurements should be such that total energy delivered to the sample is equal for two modes of optical stimulation [23].

ii). Bos and Wallinga's transformation technique

According to Bos and Wallinga, the intensity of transformed LM OSL ($I_{tr}(t)$) corresponding to CW OSL curve defined by Eq. (1) is given by [5]

$$I_{tr}(t) = \frac{t}{P} I(t^{'}) \quad (4)$$

where

$$t^{'} = \frac{t^2}{2P} \quad (5)$$

and $I(t^{'})$ can be approximated using $I(t)$ defined by equation (1). According to Bos and Wallinga, their transformation uses interpolation whereas Bulur's transformation depends on extrapolation. Since Bulur's transformation expands the t domain to u domain, there is considerable loss of resolution [5]. Also there are more number of points in the rising portion of the LM OSL curve obtained using Bos and Wallinga's transformation.

3. 0 EXPERIMENTAL STUDY

CaF_2:N phosphor used in this study was in powder (< 75 µm) form. The CW and LM OSL studies were carried out using RISO TL/OSL system TL/OSL-DA-15 in which a cluster of 42 blue light emitting diodes (λ = 470 ± 30 nm) were used for stimulation. A green long pass GG-420 filter minimizes the directly scattered blue light from reaching the photomultiplier tube (EMI 9235QA). The blue light stimulated signal was detected using a 7.5 mm thick x 35 mm diameter HOYA U-340 (λ_p~340 nm, FWHM~80nm) filter. The LED cluster delivers ~25 mW cm^{-2} (maximum) power to the sample and the stimulation power can be linearly ramped to record LMOSL [11, 24]. Irradiation of the sample was carried out using a $^{90}Sr/^{90}Y$ source (dose rate: 1.22 Gy min^{-1}) housed in the system.

The CWOSL curves were recorded by holding the sample at room temperature and using blue LEDs. Each curve was recorded for a run time of 500 s unless mentioned otherwise. The LMOSL was recorded by holding the sample at room temperature and by varying the LED intensity between 0 and 90 % powder for 1000 s. In addition to OSL, influence of optical bleaching and thermal annealing on thermoluminescence (TL) glow curves was also studied. Typical TL, CW and LM OSL curves for CaF_2:N recorded immediately as well as after 24 hours of delay are shown in Figs. 1a, 1b and 1c respectively. The effect of optical bleaching was also studied and the results are shown in Figs. 2a, 2b and 2c respectively for TL, CW and LM OSL curves. Further the effect of pre-readout annealing on TL, CW and LM OSL curves is shown in Figs. 3a, 3b and 3c respectively.

To study the transformation techniques for converting CW to LM OSL curves, the LED power during CWOSL recording was kept at 15 mWcm^{-2} (60% of the maximum value) for 355 s. The experimental LMOSL curves were recorded for 502 s (~21.22 mWcm^{-2}) and 710 s (~15 mW cm^{-2}). The stimulation power and acquisition times during experiment were such that total energy incident on sample is constant (~5325 mJ cm^{-2}). The experimental CW and LM OSL curves were recorded as per procedure described above and are shown in Figs. 4a and 4b respectively.

4.0 RESULTS AND DISCUSSION

A typical TL glow curve for CaF_2:N recorded immediately after exposure is shown in Fig. 1a. In the same Fig., the TL glow curve recorded after 64 hours of exposure is also shown. The CW OSL curve for CaF_2:N shown in Fig. 1b could be fitted as a sum of four first order decaying exponentials. The thermal fading influences the OSL intensity substantially but the decay trend is almost unaffected up to 100 hours between exposure and readout. From Fig. 1c, it can be seen that the peak height (integral counts) of the first peak decreases substantially, when TL is recorded after 64 hours of exposure. Small reduction in peak height as well as integral counts for the second peak is also observed. Further from the LMOSL curves shown in Fig. 1c, the two peaks are clearly visible and the influence of thermal fading on these peaks can also be seen. The decrease in luminescence intensity for CW and LM OSL processes is due to the thermal fading of low temperature peak which contribute towards OSL. It is also worth mentioning that in the LMOSL curve, the second peak is broader as compared to the first peak and this may be due to the fact that the traps corresponding to second and third TL peak contribute and possibly have closer values of photo-ionization cross-sections [11].

Studies on the effect of optical bleaching carried out using blue LED's (22.5 mW cm^{-2}) on CWOSL curves show that the nature of decay (decay constants) is influenced. The CWOSL curves recorded after optical bleaching could be fitted as a sum of three first order decaying exponentials for various bleaching times ranging from >5s - ≤ 20s whereas for higher bleaching times (>20 s), the CWOSL curves could be fitted as a sum of two first order decaying exponentials. The influence of optical bleaching using blue LED's on LMOSL curves was also studied and it has also been found that after optical bleaching with blue LED's for 5s (22.5 mW cm^{-2}), the first peak in the LM OSL curve is substantially erased (Fig. 2b) although higher bleaching times (up to ~ 20s) are required for complete erasure of the OSL signal from traps responsible for first peak in LMOSL in CaF_2:N. In the bleaching study, it has also been found that higher bleaching times (>5s) affect the height as well as position of the second peak as the traps contributing towards second LMOSL peak are also erased. Further the peak position t_m of the second peak shifts toward higher side in time with increase of optical bleaching time (Fig. 2c) which is typical characteristics of LMOSL curves -obeying non-first order kinetics or arising from mixture of more than one LMOSL curves [25-26].

Pre-readout annealing studies were carried out to check its influence on the nature of decay (decay constants). It was found that with pre-readout annealing at 120ºC for 10s, the CWOSL curve could be fitted as a sum of three first order decaying exponentials whereas with pre-readout annealing at 200ºC for 10s, the CWOSL curve could be fitted as a sum of two first order decaying exponentials. Also the pre-readout annealing at 120ºC for 10s removes the first peak in the LMOSL curve and a broader LMOSL curve is recorded whereas pre-readout annealing at 200ºC for 10s further decreases the intensity of the LMOSL curve and peak position is also affected. The change in nature of decay of CW OSL curves with thermal annealing is due to the fact that after pre-readout thermal annealing, the low temperature peaks contributing towards OSL are erased.

As described earlier that the CWOSL for CaF_2:N could be fitted as a sum of four first order decaying exponentials and the photo-ionization cross-sections (σ) were found to be ~$2.77*10^{-17}cm^2$ (σ_1), ~$0.90*10^{-17}cm^2$ (σ_2), ~$0.16*10^{-17}cm^2$ (σ_3) and ~$0.03*10^{-17}cm^2$ (σ_4). With pre-readout annealing (>200ºC) and optical bleaching (>20-25 s), the first two components are removed and the fitting of CWOSL curve as a sum of two first order decaying exponentials lead to photo-ionization cross-sections (σ) values ~$0.15*10^{-17}cm^2$ (σ_3) and ~$0.032*10^{-17}cm^2$ (σ_4). This confirms that the photo-ionization cross-section of deep traps is not influenced by the low temperature traps and with pre-readout annealing or optical bleaching; and the remaining OSL components can be correlated to the two of the original four components of OSL.

As CWOSL could be transformed to LMOSL curve using techniques described earlier, the transformed LMOSL could be compared with the experimental LMOSL curves. In view of this, the transformed LMOSL curves were obtained using Bulur's and Bos and Wallinga's methodologies. The transformed LMOSL curves as well as comparison of experimental LMOSL curves with transformed LMOSL curves are shown in Figs. 4a and 4b. The transformed LMOSL curves corresponding to Fig. 4a also exhibit two peaks indicating that approximation of CW OSL curve of CaF_2:N as a sum of four first order decaying exponentials hold. The four components of the transformed LMOSL curve are also shown in Fig. 4c. Further transformation studies on the CWOSL curves obtained after optical bleaching (>5 s) or pre-readout annealing at 120ºC for 10 s were also carried out and it has been found that transformed LMOSL curves exhibit only one peak as was recorded experimentally.

The CWOSL curve for CaF_2:N fitted as a sum of four first order decaying exponentials is shown in Fig. 5a. Further CWOSL curves recorded after optical bleaching of 5s and pre-readout thermal annealing at 120ºC for 10 s are shown in Figs. 5b and 5c respectively. It can be seen that the CWOSL curves recorded after pre-readout annealing at 120ºC or optical bleaching for ~10s-20s, could be fitted as a sum of three first order decaying exponentials whereas CWOSL curves recorded after pre-readout annealing at 200ºC or optical bleaching for >20s, could be fitted as a sum of two first order decaying exponentials.

It is worth mentioning that the second peak in LMOSL of CaF_2:N may be composed of three first order peaks. This is also supported by the fact that the shape factor μ_g for second LMOSL peak was found to be ~0.80 which is much higher than the values defined for first as well as second order kinetics (0.65-0.68) [26-27]. This indicates that the second LMOSL peak may be a mixture of more than one LMOSL curves possibly obeying first order kinetics. However the LMOSL curve having more than two peaks could not be recorded experimentally for CaF_2:N even using very weak stimulation (1% of the maximum power *i.e.* 0.25 mW cm^{-2}) for 24, 000 s. Further efforts are in progress to record OSL of CaF_2:N using newly proposed technique namely non-linearly modulated optically stimulated luminescence (NLOSL) [27].

5. CONCLUSIONS

1. The CWOSL from CaF_2:N could be fitted as a sum of four first order decaying exponentials (components) and the photo-ionization cross-sections (σ) were found to

be ~$2.77*10^{-17}cm^2$ (σ_1), ~$0.90*10^{-17}cm^2$ (σ_2), ~$0.16*10^{-17}cm^2$ (σ_3) and ~$0.03*10^{-17}cm^2$ (σ_4).

2. Optical bleaching using blue LED's for 5-15 s is sufficient to erase the first component whereas for bleaching times around 20-25 s, only two components are observed. Similar results are observed after pre-readout annealing at 120ºC and 200ºC for 10 s. The photo-ionization cross-sections of the remaining components match with the photo-ionization cross-sections of the two of the four components of OSL.
3. The first peak in LMOSL from CaF_2:N is bleached in 5-15s using blue LEDs whereas the second peak shifts towards higher side with increase in bleaching time. The shape factor μ_g for second LMOSL peak was found to be ~0.80 which is much higher than the values encountered for first (0.55-0.58) as well as second order kinetics (0.65-0.68). This indicates that the second LMOSL peak may be mixture of more than one LMOSL curves possibly obeying first order kinetics.
4. Transformation of CWOSL to LMOSL curves indicate that the behavior of the transformed LMOSL curves is similar to the LMOSL curves recorded experimentally.

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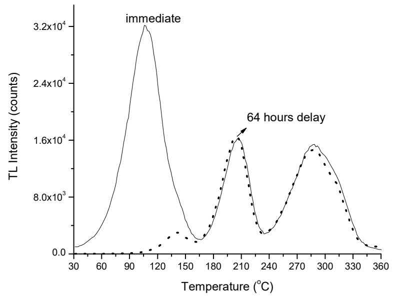

Fig. 1a: TL glow curve for CaF_2:N recorded immediately as well as after 64 hours. The TL glow curves were recorded at a heating rate of 5ºC s^{-1} and a β dose of 1.22 Gy was delivered to the sample.

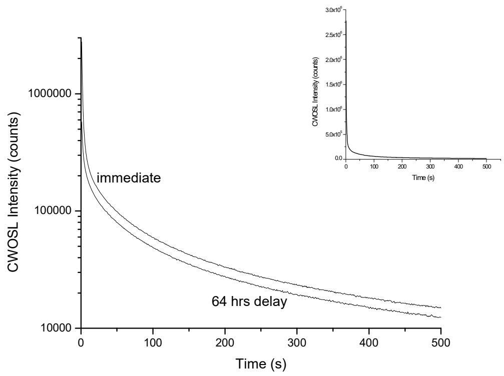

Fig. 1b: CW OSL curve for CaF_2:N recorded immediately as well as after 64 hours. The β dose of 1.22 Gy was delivered to the sample. The LED power was kept at 90% of the maximum power. The Fig. in the inset shows CWOSL curve plotted in linear scale and recorded immediately after exposure.

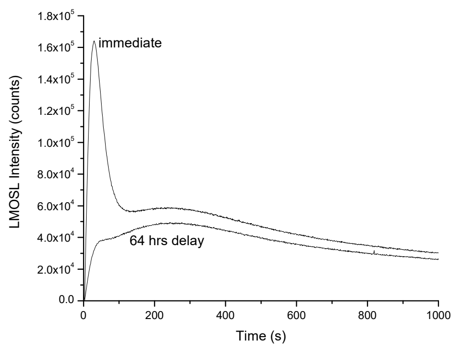

Fig. 1c: LM OSL curve for CaF_2:N recorded immediately as well as after 64 hours of β dose (1.22 Gy). Two peaks in LMOSL curve are clearly visible. The LED power was varied from 0-90% of the maximum power.

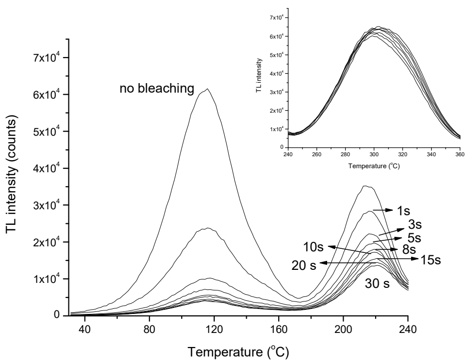

Fig. 2a: Effect of optical bleaching on TL glow curves. The TL glow curves were recorded at a heating rate of 5ºC s^{-1} and the dose delivered to the sample was 1.22 Gy. Optical bleaching was carried using blue LEDs at 90% power of the maximum power. High temperature peak is unaffected by optical bleaching (inset).

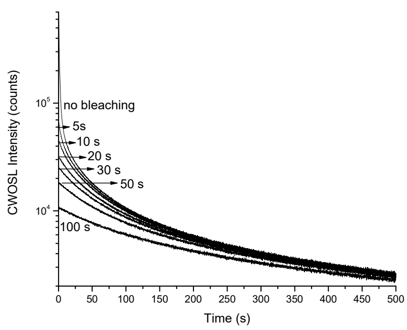

Fig. 2b: Effect of optical bleaching on CW OSL curves. The β dose delivered to sample was 1.22 Gy. The LED power was kept at 90%. The optical bleaching was carried out using blue LEDs at 90% of the maximum power.

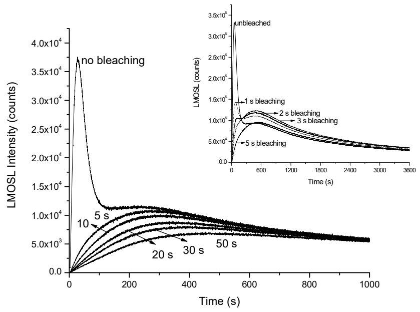

Fig. 2c: Effect of optical bleaching carried out using blue LEDs (90% power) on LM OSL curves. The β dose delivered to sample was 1.22 Gy. During LMOSL recording, the LED power was varied from 0-90% of the maximum power. For optical bleaching times from (1-5) s, the LMOSL curves are shown in the inset and the LED power was varied from 0-100% for 3600s.

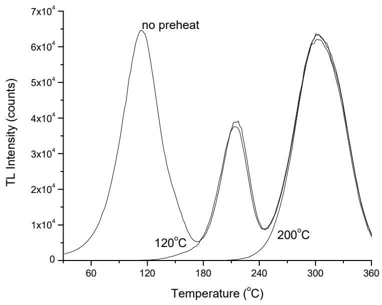

Fig. 3a: Effect of pre-readout thermal annealing on TL glow curves. The TL glow curves were recorded at a heating rate of 5ºC s^{-1} and the dose delivered to the sample was 1.22 Gy.

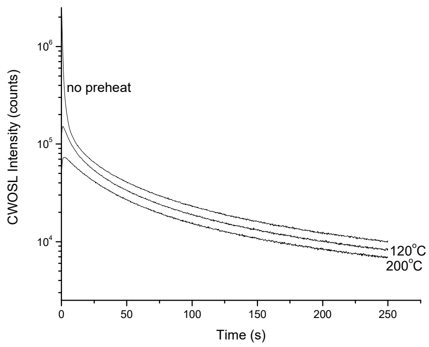

Fig. 3b: Effect of pre-readout annealing on CW OSL curves. The β dose delivered to sample was 1.22 Gy. The LED power during CWOSL recording was kept at 90% of the maximum power.

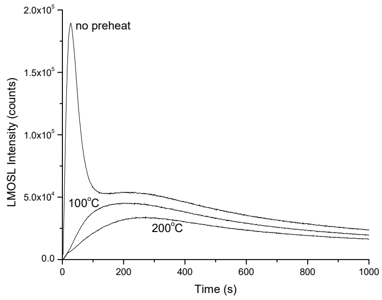

Fig. 3c: Effect of pre-readout annealing on LM OSL curves. The β dose delivered to sample was 1.22 Gy. During LMOSL recording, the LED power was varied from 0-90% of the maximum power.

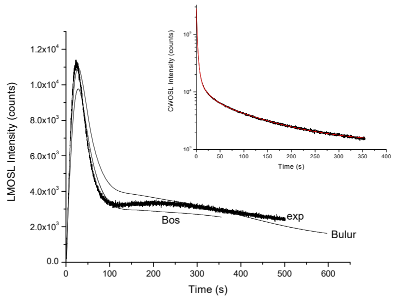

Fig. 4a: Transformation of CW OSL curve corresponding to Fig. 5a into LM OSL curve using Bulur and Bos techniques. The β dose delivered to sample was 1.22 Gy. The LED power during CWOSL recording was kept at 60% of the maximum power. The transformed LMOSL curves are also compared with the experimental curve which was recorded by varying LED power from 0-85% of the maximum power. The stimulation power and acquisition times are such that total energy incident on sample is constant.

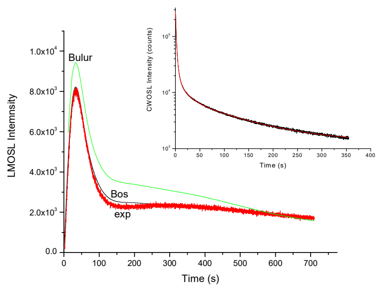

Fig. 4b: Transformation of CW OSL curve corresponding to Fig. 5a into LM OSL curve using Bulur and Bos techniques. The β dose delivered to sample was 1.22 Gy. The LED power during CWOSL recording was kept at 60% of the maximum power. The transformed LMOSL curves are also compared with the experimental curve which was recorded by varying LED power from 0-60% of the maximum power. The stimulation power and acquisition times are such that total energy incident on sample is constant.

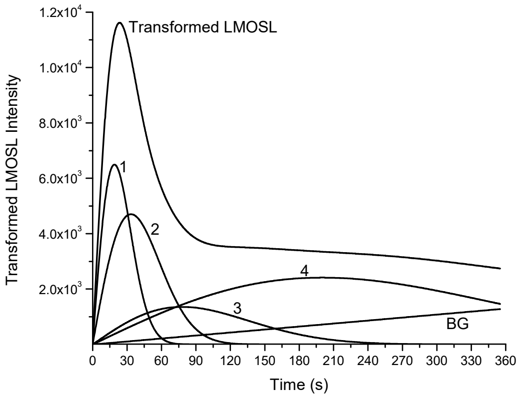

Fig. 4c: LMOSL components of transformed CW OSL curve corresponding to Fig. 5a. Other parameters are same as in Fig. 4a.

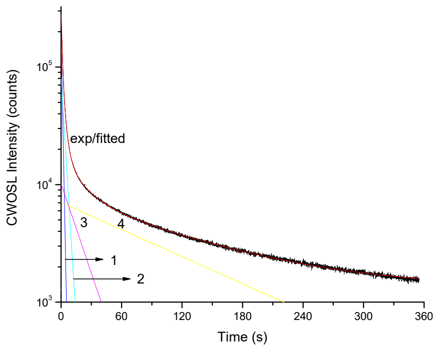

Fig. 5a: Fitting of experimental CW OSL curve for CaF_2:N as a sum of four first order decaying exponentials. The β dose of 1.22 Gy was delivered to the sample. The LED power during CWOSL recording was kept at 60% of the maximum power.

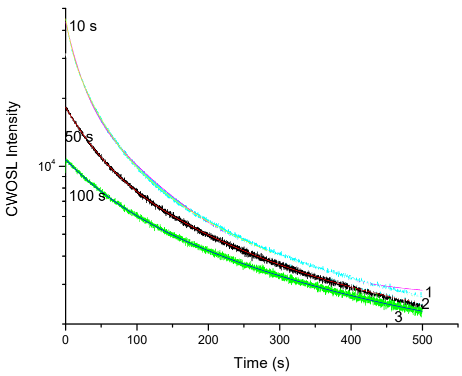

Fig. 5b: Fitting of experimental CW OSL curve recorded after bleaching times for 10s, 50s and 100s. The β dose of 1.22 Gy was delivered to the sample. The LED power during CWOSL recording was kept at 90% of the maximum power. Curve no. 1 could be fitted as a sum of three first order decaying exponentials whereas the curves no. 2 and 3 could be fitted as a sum of two first order decaying exponentials.

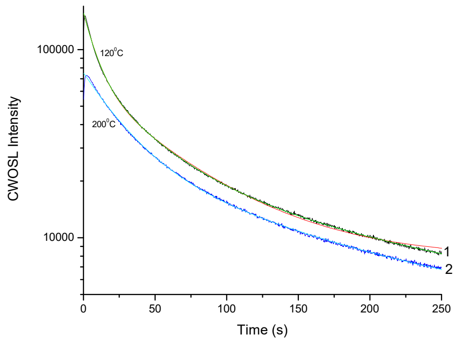

Fig. 5c: Fitting of experimental CW OSL curve recorded after pre-readout annealing. The β dose of 1.22 Gy was delivered to the sample. The LED power during CWOSL recording was kept at 90% of the maximum power. Curve no. 1 could be fitted as a sum of three first order decaying exponentials whereas the curve no. 2 could be fitted as a sum of two first order decaying exponentials.