

CHAPTER-4

RESULT

AND

DISCUSSION

4.1 Introduction

It is well known that the favorable dose saturation properties of thermally transferred optically stimulated luminescence signals (TT-OSL) offer the opportunity to create broad luminescence chronologies that go beyond the traditional upper limits of quartz OSL dating.¹⁻³ The thermal transfer is transfer of charges from shallow light insensitive traps to deeper light-sensitive traps, as a result of preheating the sample.⁴

R Bailey et al reported that thermal transfer results in electrons from deep traps, which may not have been bleached during their last deposition, being freed to move to shallow traps and give a signal during OSL measurement, this may lead to an apparent high age. Thermal transfer is mainly dependent on the temperature (preheat and cut heat) used during measurement and maybe a significant problem for young sediments.⁵⁻⁶ Thus, the eliminating of unwanted traps by thermal transfer process in the phosphors gives advantage of OSL as a TT-OSL. It is necessary to understand the history of traps which are developed in the irradiated sample which is possible by the thermoluminescence (TL) experiment.

The thermoluminescence (TL) study on quartz material is being carried out for last many decades to the date. It has shown overlapped glow peaks pattern which is associated with complexity in the internal structure of the material. The changes in the internal structure of the material happen by physical treatment to the sample, origin of the sample, and experimental condition. The literature survey⁷ reports that material has optical and thermal sensitive nature of the defects as electron traps and hence all optically sensitive traps participate in TL and OSL process but the traps that have thermal sensitivity in nature participate in TL process only. However, few of them may show the dual nature (optical and thermal sensitive) of the traps and hence it may support establishing the correlation between TL and OSL phenomenon. Given these, the researchers have carried out systematic work on TL emission of quartz, they have reported that natural quartz material shows broad usual TL glow peaks around 110°C, 165°C, 210°C, 245°C, 280°C, 325°C, 365°C and 375°C with their different emission over TL measurement temperature.⁸

These traps are classified as a usual shallow trap (110°C-160°C TL glow peaks), stable dosimetry traps (210°C -220°C TL glow peaks), intermediate traps (240°C -260°C TL peaks), rapidly bleachable traps (325 °C TL glow peak) TL glow peak (RBP) and slowly

bleachable traps (around 375 °C TL glow peak) according to their contribution in TL applications.

In a usual process of TL emission, the thermally stimulated electrons from the traps are recombined with the hole at the luminescence center via conduction band. According to Nambi K V S⁹, the possibilities in which the thermally released electrons do not follow the usual path and hence it may re-trap into shallow traps, same traps, different localities, and deep TL traps which arises the loss of TL counts and it could not give sufficient TL efficiency. Therefore, before proceeding for present OSL study, systematic investigations on TL properties of synthetic quartz material are required. It may give them information about the nature of traps that are responsible for re-tapping of electrons, act as an unstable trap, stable traps, etc. by suggested possibilities. Also, it may become easy to implement for the study of thermal transfer effect on OSL of synthetic quartz samples.

4.2 Thermoluminescence (TL) study of synthetic quartz.

According to the literature survey, the changes in TL glow curve patterns are influenced by the magnitude of physical treatments like ionizing radiation, thermal treatment (fired temperature and annealing temperature), duration of thermal treatment, illumination of light, grain sizes, TL heating rate and storage time. However, the TL outcomes under influence of these physical treatments have been explained by various luminescence models, structural changes due to defects, TL-related phenomena, and computational glow curve deconvolution (CGCD) technique.³⁸⁻³⁹ It has been also revealed that TL of quartz followed by above physical treatments, help to establish the connection between typical TL trap associated with OSL mechanism. In present investigations, the thermoluminescence glow curves have been recorded for synthetic quartz material under novel protocols which include the effect of annealing temperature, beta dose/test dose, optical bleaching, and thermal bleaching. The TL outcomes have been discussed for the glow peak position, shape of TL glow peak, and TL intensity. The TL dose responses curves (TL-DRC) are obtained from the observed TL records for predominantly contributed TL glow peaks. Subsequently, their dose-response nature is segregated as sub-linear, linear, and super-linear have been discussed.

4.2.1 Effect of beta dose on TL glow curve of unannealed sample.

As mentioned earlier, the TL glow curves are influenced by various physical treatments to the sample, and hence for the present work, the influence of beta doses on TL glow peak is studied under the following suggested protocol stepwise and the changes in glow peak pattern and TL intensity are discussed.

SQ (grain size 63-53 μ m)

Step-1 Unannealed sample

Step-2 Beta Dose

2.268 Gy, 22.68 Gy, 158.76 Gy @Dose Rate 4.45 \pm 0.06Gy/min

Step-3 TL record from 0-450 $^{\circ}$ C @heating rate 5 $^{\circ}$ C /sec

Unannealed synthetic quartz materials were irradiated for 2.268Gy, 22.68Gy, and 158.76Gy beta doses individually to each sample. TL glow curves were recorded from 0 $^{\circ}$ C to 450 $^{\circ}$ C measurement temperature with heating rate 5 $^{\circ}$ C/s. Each irradiated sample shows the usual TL glow peak around 110 $^{\circ}$ C. TL intensity was found to increase from 80.07 counts to 540.66 counts which reveals about 85% of TL sensitization takes place in the sample as a function of beta dose. Apart from this TL glow peak, several other TL glow peaks are also observed at 190 $^{\circ}$ C, 272 $^{\circ}$ C, and 362 $^{\circ}$ C with feeble intensity under higher beta irradiation of 22.68Gy and 158.76Gy. (Fig. 4.1)

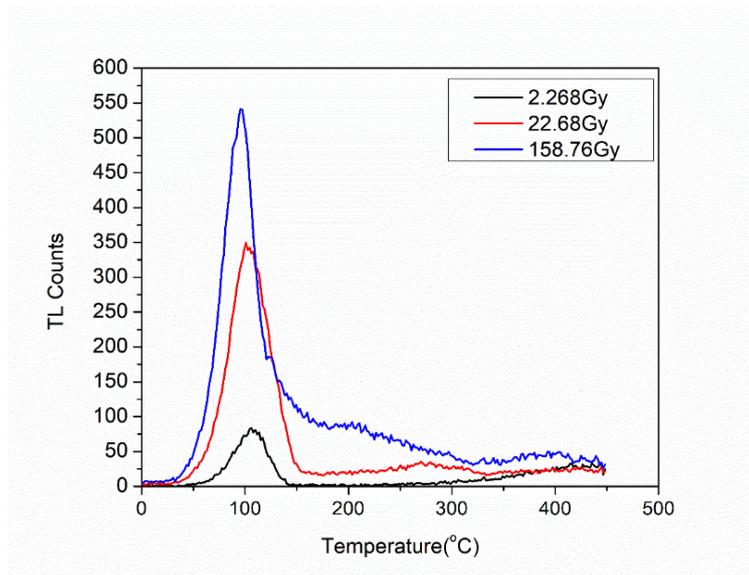
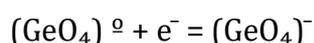


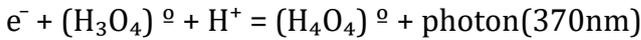
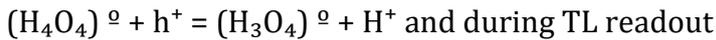
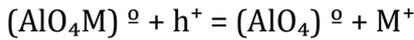
Fig. 4.1 TL recorded for different beta doses

It is well established that in the TL mechanism, the material must be previously irradiated so defects/traps are induced in the material. The lifetime of charges in the trap depends upon the depth of the TL trap. This trap depth is responsible for releasing the electrons at an adequate temperature. Pre-dose sensitization is the increase of TL sensitivity following the application of radiation dose and heat treatment. The increase in sensitivity is related to the absorbed radiation dose received by the quartz sample before thermal treatment. Zimmermann suggested a model account for the pre-dose sensitization effect. In this model, the increase in the TL output following pre-dose sensitization is believed to be due to an enhanced probability of radiative recombination at the luminescence site due to the transfer of holes from so-called reservoir centers (R) to luminescence centers (L).¹⁰

Benney P et al ¹¹ have reported that E₁' center grows on thermal treatment up to a temperature of 400°C and decays beyond this temperature; its intensity being insignificant beyond 550°C. Also, the model for E₁' center and its precursor in quartz represent that E₁' center, an oxygen vacancy containing two electrons in a singlet state (S = 0). These electrons release during post-irradiation heating which could get trapped at the competing deep traps rendering the competing trap ineffective during the TL readout. The E₁' center becomes unstable and decays in the temperature range 400°C -500°C. This decay would involve the release of the E₁' center electron and results in the bared oxygen vacancy. The released electron also has the possibility of getting trapped at the competing deep trap. As a consequence, in the sensitized sample, the probability of recombination at the recombination center will increase. This will give rise to increased TL emission.

Mckeever S W S¹² has reported that the emission at 100°C is characterized by a single TL peak emitting at 380nm, with a less intense, broad emission peak near 470nm. The Electron Spin Resonance (ESR) signal clearly indicates that the trap responsible for the 100°C peak is a (GeO₄)⁻ center, formed by the trapping of an electron at a (GeO₄)^o site. Also, the correlation between Electron Spin Resonance (ESR) signals and the 100°C TL glow peak have revealed that the hole traps involved in the process are (AlO₄)^o and (H₃O₄)^o centers. Further, the sequence of charge trapping and TL production is suggested by the following equations during irradiations





The noticeable growth in TL sensitivity of 110°C glow peak of synthetic quartz is attributed to an increase in the strength of ionizing radiation. Also, higher temperature TL glow peaks during TL measurement is attributed to the released of electrons at this temperature from these traps may recombine with hole center which is associated with 470nm TL emission of light.

4.2.2 TL-Dose Response Curve (TL-DRC) of unannealed sample.

The Thermoluminescence technique is widely used for dosimetry purpose to measure the ionizing radiation. The important property of TL material shows good linear relation between TL intensity and absorbed dose(D). The expression for measured TL dose response curves(TL-DRC) may be written as $TL = a(D)^k$ where, a is constant, D is applied dose and k value is related to slope of the DRC and it determines the nature of the graph by super-linear if $k > 1$, linear if $k = 1$ and sublinear if $k < 1$ over $\log TL$ against $\log D$ scale which follows equation $\log TL = k \log D + \log a$ which is received by applying logarithm to the equation $TL = a(D)^k$.¹³

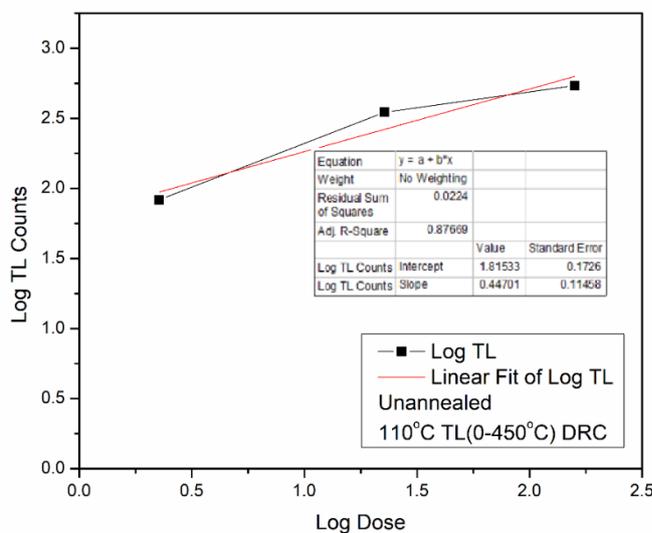


Fig.4.2 TL dose response curve of 110°C TL glow peak

The unannealed sample shows contribution of 110°C TL glow peak by significant growth in TL intensity under influence beta doses of 2.268Gy, 22.68Gy and 158.76Gy. However, the equation is fitted to TL-DRC of 110°C TL glow peak and it shows sublinear TL-DRC nature by having value of k is 0.44. (Fig. 4.2)

Lawless J L et al¹⁴ have suggested that the nature of the dose dependence of the luminescence signal is of great importance for the use of thermoluminescence (TL) and optically stimulated luminescence (OSL) as a dosimetry and dating application. The sub linearity, which had been found in several materials, was usually attributed to saturation effects during excitation of either the relevant traps or recombination centres. The competition effects during the excitation amongst traps may result in super linearity of some TL peaks. Also, sublinear dose-dependence may take place in the simplest possible case of one trap–one recombination centre (OTOR), and even when the traps and centres are far from saturation. Under the influence of different beta doses, the traps corresponding to the 110°C TL glow peak may fill sufficiently and may follow the usual path to recombine with the hole which may give a noticeable TL signal. The development of several new higher temperature TL traps may indicate that shallow traps begin to saturate under beta doses. This may be responsible for the sublinear nature of TL-DRC of an unannealed sample.

Nambi K V S¹⁵ has reported that TL properties exhibited by a phosphor depends too much upon the kind of “thermal annealing” experienced by it before the irradiation/excitation. (Obviously, any post-excitation thermal treatment erases the TL signal if any present up to the treatment temperature on the TL glow curve). Annealing is a process that reduces internal strain and surface energy. It is also generally true that more defects are produced at higher temperature of annealing. The number of defects retained by the crystal lattice also depends on the cooling rate employed to cool the crystal to the ambient temperature from the annealing temperature. As the defects are often directly involved in the TL process, it can be easily explained that TL should be closely related to the thermal annealing history of the crystal. Hence, the TL properties of synthetic quartz material are also studied under influence of annealing-quenching (AQ) treatment followed by beta doses in present work.

4.2.3 Effect of annealing temperature on TL glow curve of sample.

Under the influence of annealing treatment before irradiation, the glow peak position, TL sensitivity, and nature of the TL dose-response curve have been considered as a TL outcome from synthetic quartz material. The changes in TL outcomes have been studied for different annealed samples to understand the efficient annealing treatment for synthetic quartz and have been compared with that of the TL outcomes of an unannealed sample. For these investigations, the following experimental protocol has been implemented.

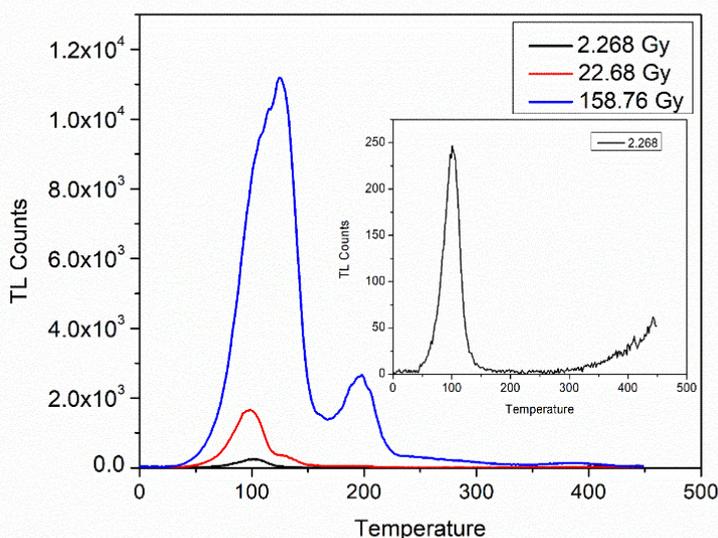
SQ (grain size 63-53 μ m)

Step-1 Annealing; 400 $^{\circ}$ C, 600 $^{\circ}$ C, 800 $^{\circ}$ C, 1000 $^{\circ}$ C @1hr and quenched at RT

Step-2 Beta Dose 2.268 Gy, 22.68 Gy, 158.76 Gy @Dose Rate 4.45 \pm 0.06Gy/min

Step-3 TL records from 0-450 $^{\circ}$ C @heating rate 5 $^{\circ}$ C /sec

Each annealed sample of 1 hour duration irradiated by 2.268Gy, 22.68Gy and 158.76Gy beta doses and the TL glow curves were recorded for 0 $^{\circ}$ C to 450 $^{\circ}$ C TL measurement temperature. The lower annealed sample (say 400 $^{\circ}$ C; 1hr) shows usual 110 $^{\circ}$ C TL glow peak, which varies between 99 $^{\circ}$ C -126 $^{\circ}$ C with different beta doses. As beta dose was increased, the TL intensity of this glow peak increases from 246.63 counts to 10286.33 counts which shows \sim 97 % of TL sensitization in sample. (Fig. 4.3) Apart from this glow peak, samples also exhibit the several new TL glow peaks around



190 $^{\circ}$ C, 196 $^{\circ}$ C and 386 $^{\circ}$ C with lower TL intensity.

Fig. 4.3 TL recorded for 400 $^{\circ}$ C annealed sample for different beta doses

With change in annealing treatments such as 600°C, 800°C and 1000°C followed by identical doses, the position of 110°C TL glow peak found to be varying between 66°C to 133°C. This broad range of 110°C TL glow peak represents that annealing treatment sensitizes number of traps which are overlapped. Therefore, it is reasonable to consider it as a shallow TL trap.

The pattern of overlapped TL glow peaks is also observed for higher temperature glow peaks. It is observed as range of 190°C -198°C, 202°C -212°C, 290°C -295°C, 300°C, 330°C, 365°C and 386°C. Due to wide range of overlapped TL glow peaks, the average TL glow peak is considered for further discussion. However, for the 600°C annealed sample, the TL sensitivity of average 293°C glow peak increases from 30 counts to 4895 counts which is ~97 % of TL growth in the sample as a function of beta dose. Such TL growth pattern remains identical for average 300°C TL glow of 800°C annealed sample. (Fig. 4.4,4.5)

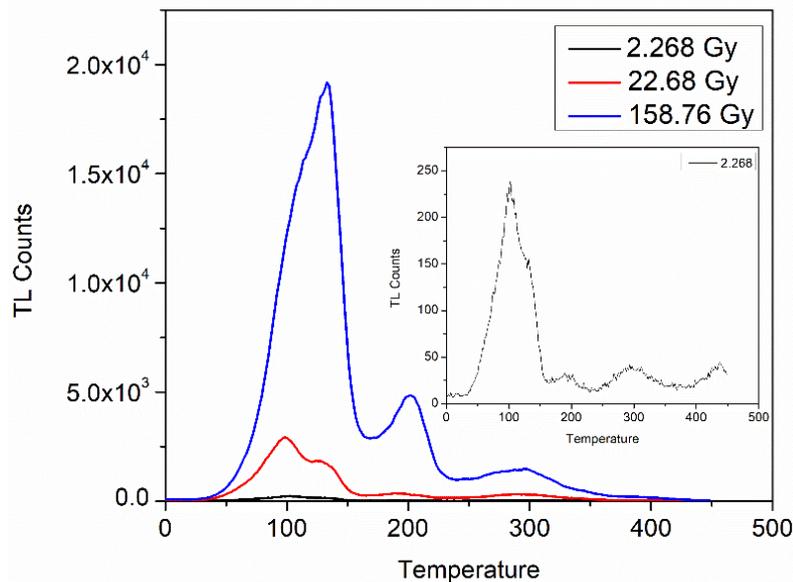


Fig. 4.4 TL recorded for 600°C annealed sample for different beta doses

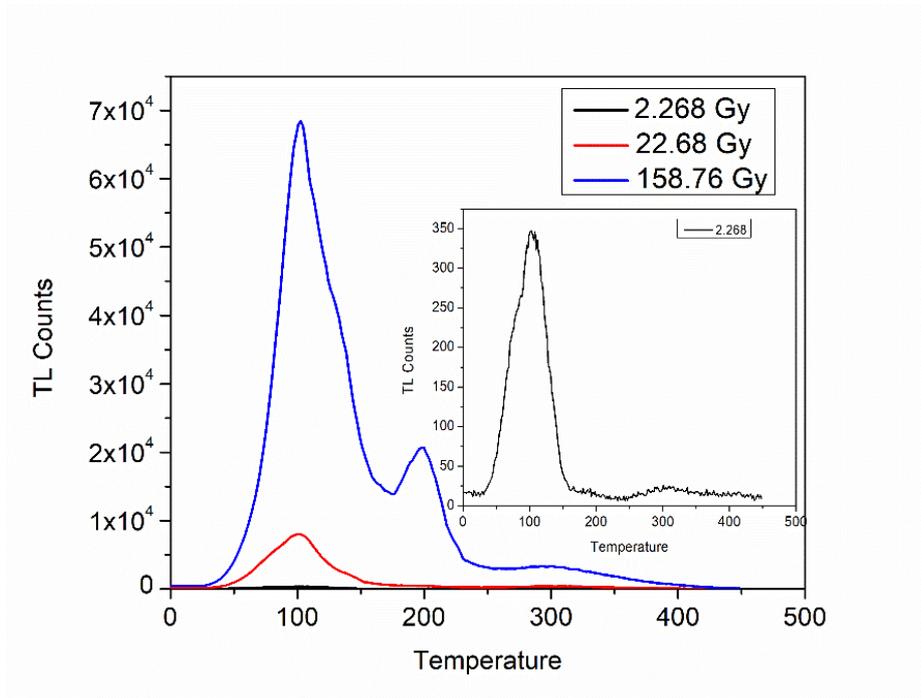


Fig. 4.5 TL recorded for 800°C annealed sample for different beta doses

The average contribution of 293°C and 300°C TL glow peaks are eliminated by the appearing average 207°C and 365°C TL glow peaks with growth in TL sensitivity in 1000°C annealed sample. (Fig. 4.6)

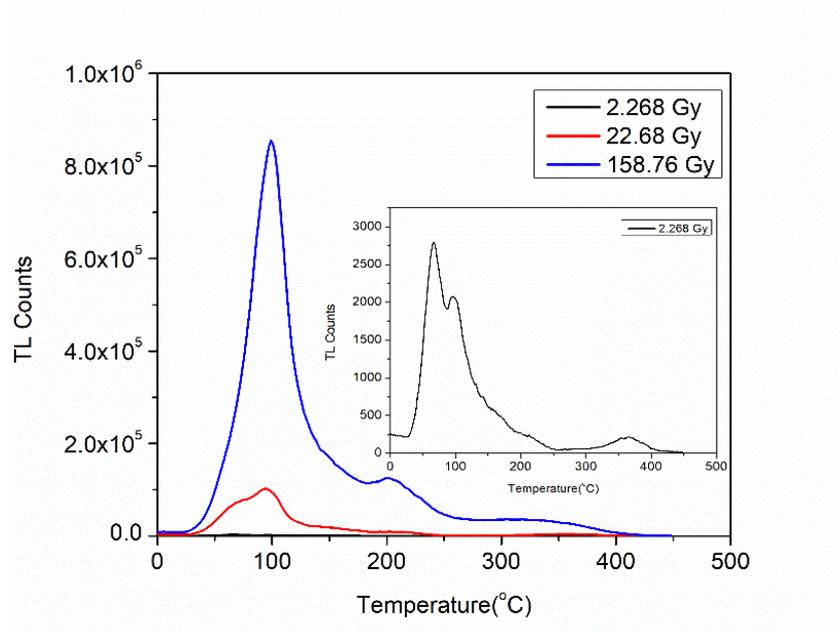


Fig. 4.6 TL recorded for 1000°C annealed sample for different beta doses

It is reported that, each annealing temperature has its own strength to sensitize the sample which helps to develop varieties of new TL traps with significant intensity at identical doses. Further, as compared to lower annealed (say 400°C) sample and unannealed sample, the 110°C TL glow peak shows ~ 97% of TL sensitization in higher annealed samples and hence the annealing treatment is given all samples for further works. (Fig. 4.7,4.8)

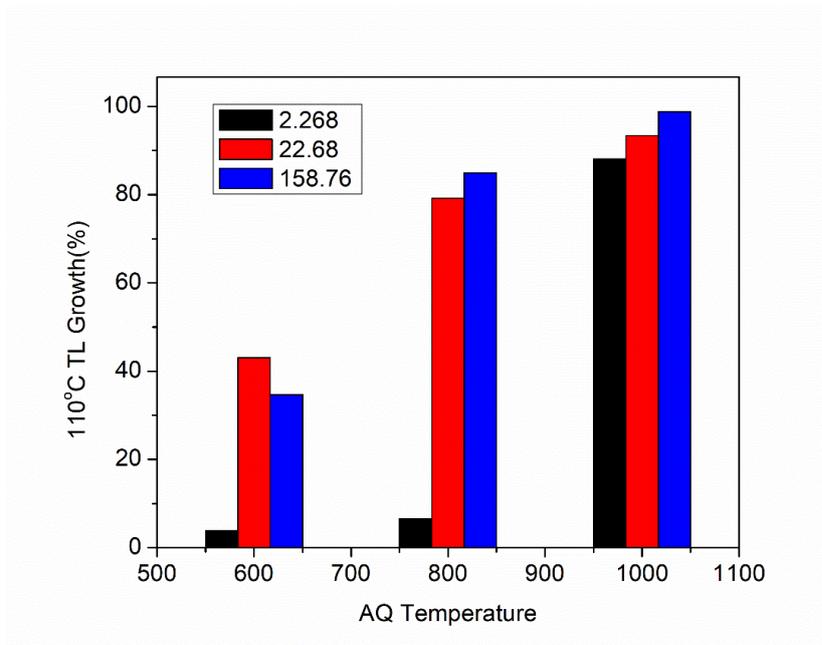


Fig. 4.7 Comparison of 110°C TL glow peak sensitivity

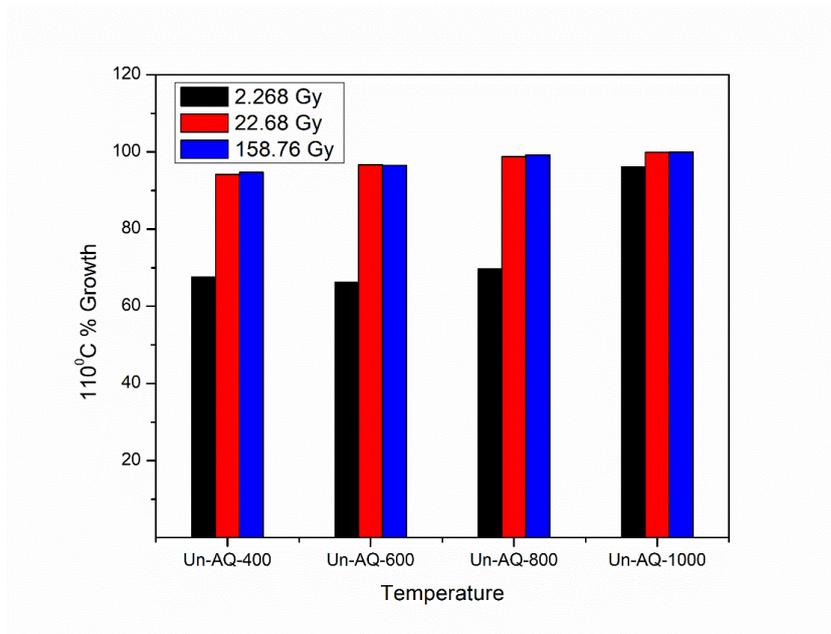


Fig. 4.8 Comparison of % growth of 110 °C TL glow peak sensitivity between Un annealed and annealed samples for different beta dose

Literature reported that in some cases, the TL sensitivity changes by the annealing treatment and it can be identified with certain crystalline phase changes in the phosphor like quartz. These temperatures of phase change also correspond to the well-known inversion temperatures of quartz at which the crystalline phase changes from alpha to beta and beta to gamma respectively. These changes are found to be more pronounced when the sample is quickly quenched to the ambient from the annealing temperature. This is perhaps due to "freezing in" some kind of mixed-phase defect.

Kale Y¹⁶ has reported that annealing-quenching treatment to the crystalline material enhances thermal sensitization and shows phase transformation at a definite temperature. According to his work, the α -quartz was stable below 573⁰C and showed Trigonal structure, the β -quartz was stable from 573⁰C to 870⁰C and showed hexagonal structure, whereas; both the α - Tridymite and β -Tridymite phase showed Orthorhombic structure.

Hüseyin Toktamış*^A, Necmeddin Yazıcı¹⁷ have reported some environmental and experimental variables such as the annealing process that alter the structure of glow curves of some thermoluminescence materials. The experimental results showed that the peak temperatures of glow peaks in annealed samples shift to higher temperature sides. The amount of shifting was discernible for higher for low-temperature peaks than high-temperature peaks. The trap depths of annealed and unannealed samples of both synthetic and natural quartz are also obtained by various heating rate methods by them. It has been noticed that the trap depth of all TL Glow peaks gets affected by annealing process. Trap depth was also found increases due to the application of annealing process.

In the present investigation, the TL sensitization of 110⁰C glow peak with given annealing treatments is also in agreement with the first transformation from alpha to the beta phase. Further, it is confirmed by ESR studies in which the annealing temperature is found to be responsible for the increment of thermal sensitization of defects level related to E1' centres. Also, in a yet another significant development, it is observed that for higher annealing treatment, the broad range of higher temperature TL glow peaks are developed, which may be associated with the sensitivity of new TL traps. However, with a further rise in annealing temperature (say 1000⁰C of 1 hour), the position of higher temperature is shifted toward the lower temperature side of

measurement temperature and stabilized at $\sim 207^{\circ}\text{C}$. The ESR work of Kale Y D reported that Ge centres are associated with a TL glow peak corresponding to $\sim 220^{\circ}\text{C}$ glow peak which is nearly correlated to present TL outcomes. Hence, it may be ascribed to the growth of Ge centres.

As discussed in earlier section, the higher annealing treatment sensitized to new TL traps over a wider span of measurement temperature in accordance with the usual 110°C TL glow peak. The TL-DRC nature of these TL glow peaks has been also considered. The 400°C and 600°C annealed samples exhibit sublinear ($k < 1$) TL-DRC characteristics of 110°C TL glow peak, whereas TL-DRC nature of 110°C TL glow peak for 800°C and 1000°C annealed samples has been found to be super-linear. (Fig-4.9,4.10,4.11,4.12).

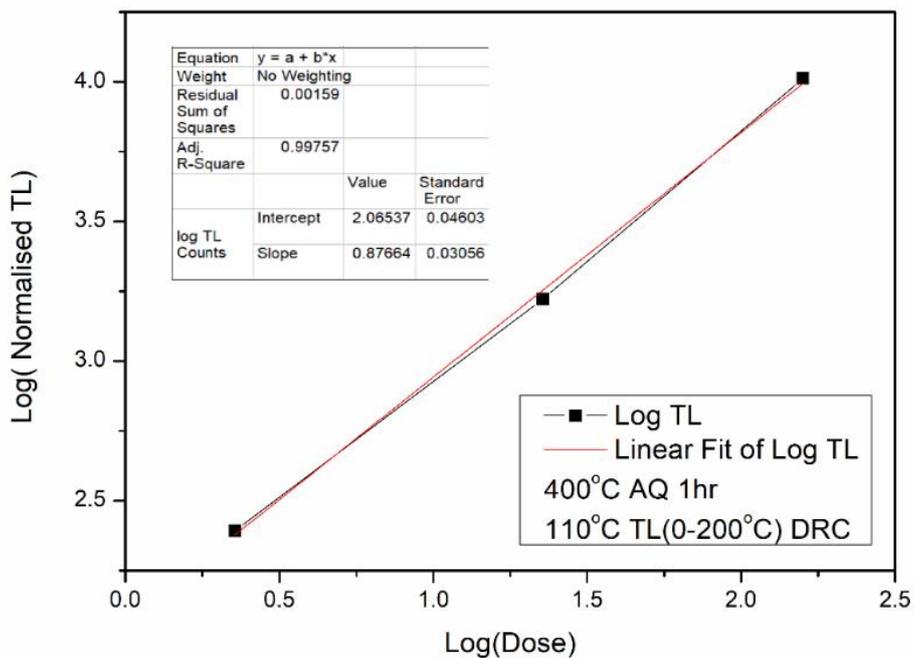


Fig.4.9 TL dose response curve of 110°C TL glow peak for 400°C annealed sample

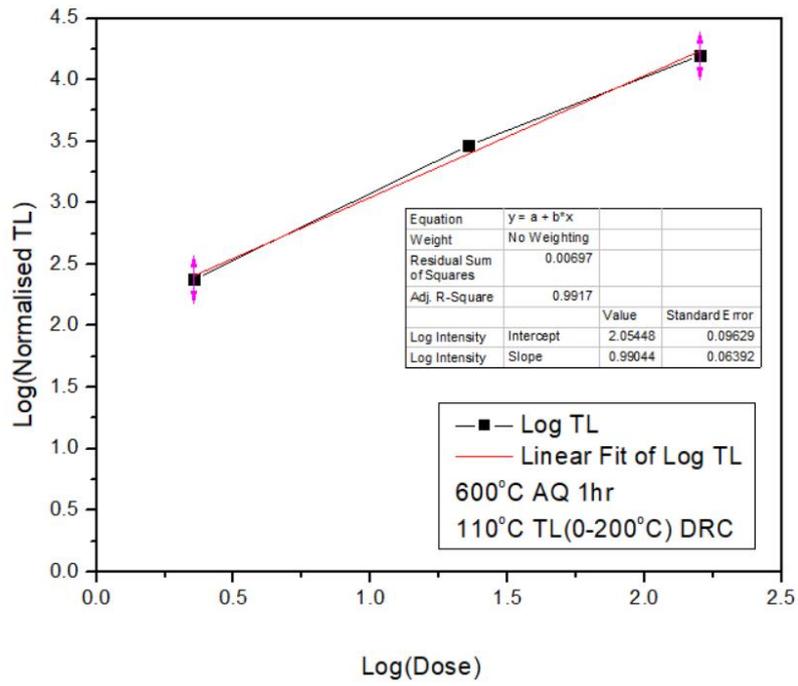


Fig.4.10 TL dose response curve of 110°C TL glow peak for 600°C annealed sample

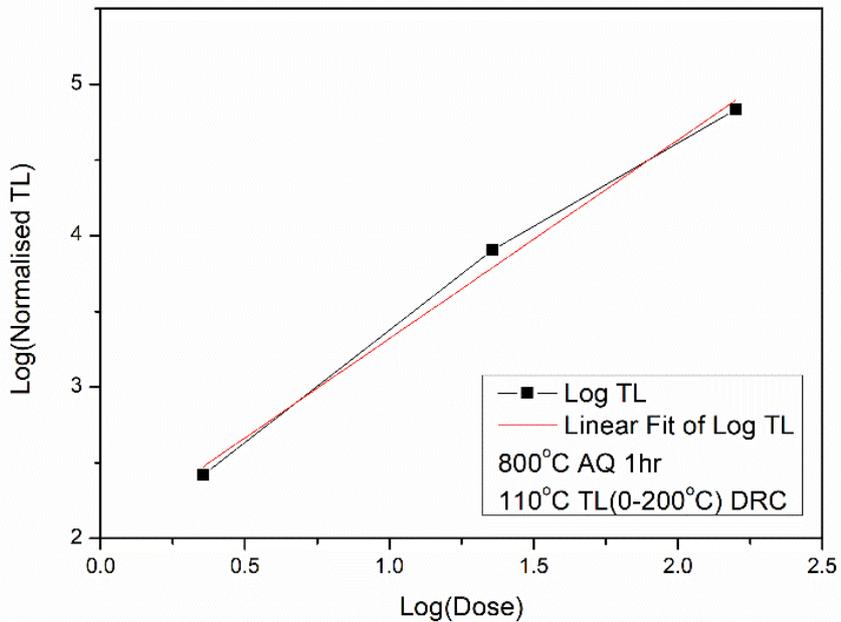


Fig.4.11 TL dose response curve of 110°C TL glow peak for 800°C annealed sample

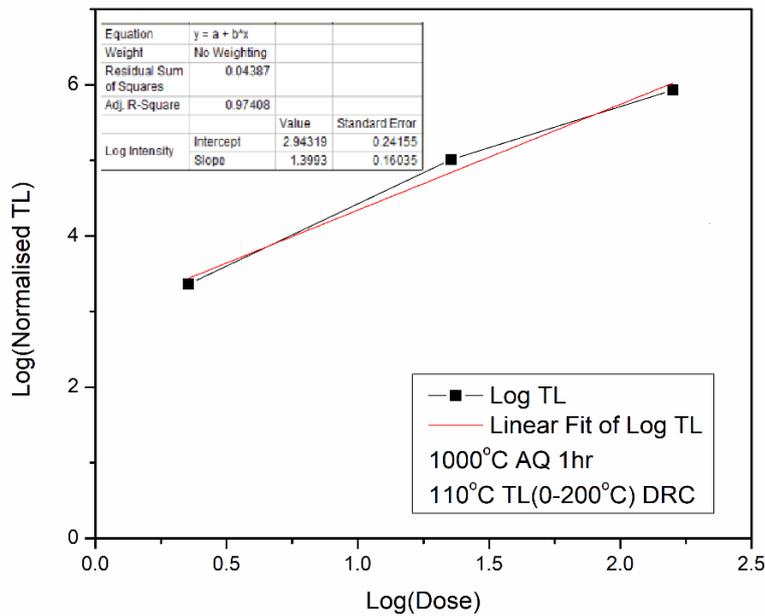


Fig.4.12 TL dose response curve of 110°C TL glow peak for 1000°C annealed sample

However, the 293°C TL glow peak exhibits sublinear nature (by $k = 0.85$) for 600°C annealed sample. Whereas, the 300°C and 207°C TL glow peaks exhibit super linear nature (by $k = 1.18$ and $k = 1.41$) for 800°C and 1000°C annealed sample respectively.

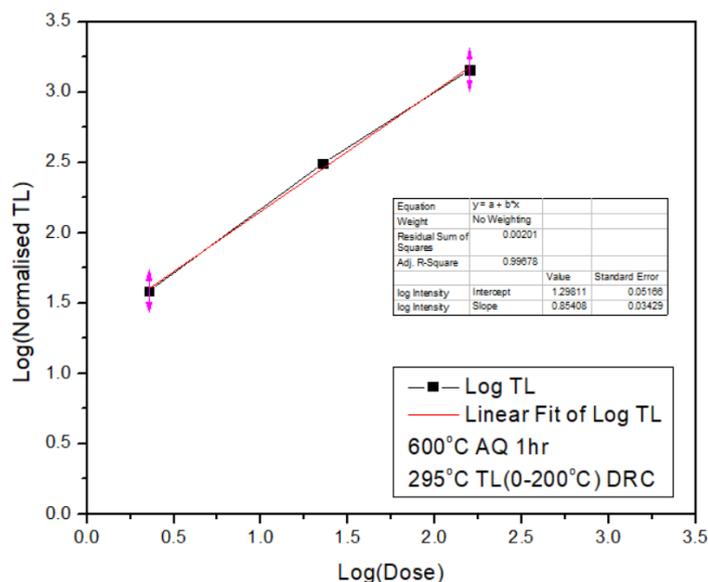


Fig.4.13 TL dose response curve of 295°C TL glow peak for 600°C annealed sample

(Fig-4.13,4.14,4.15). It is suggested that the higher annealing treatment enhances super linear nature of TL-DRC curve of TL glow peaks including TL-DRC curve of 110°C glow peak compared to unannealed sample.

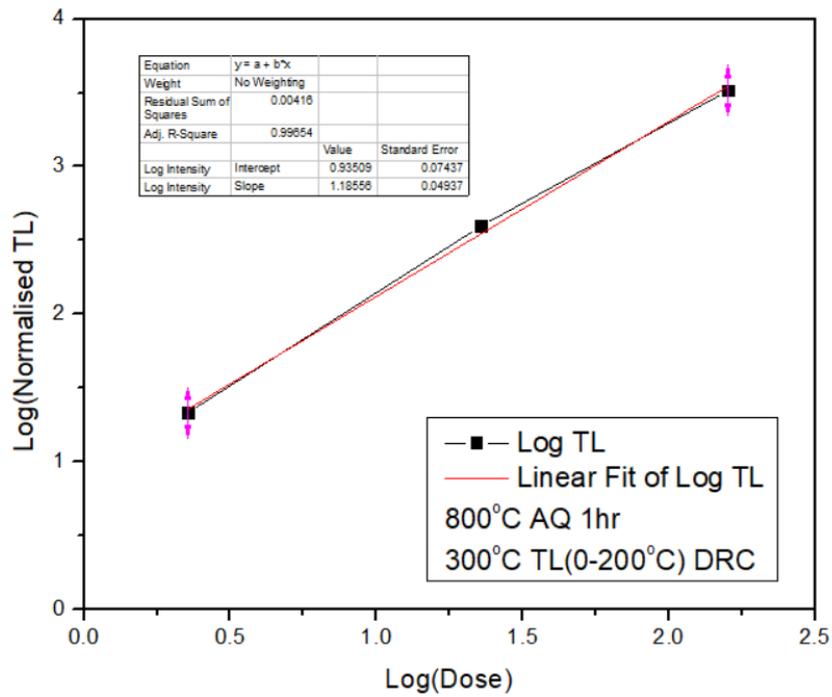


Fig.4.14 TL dose response curve of 300°C TL glow peak for 800°C annealed sample

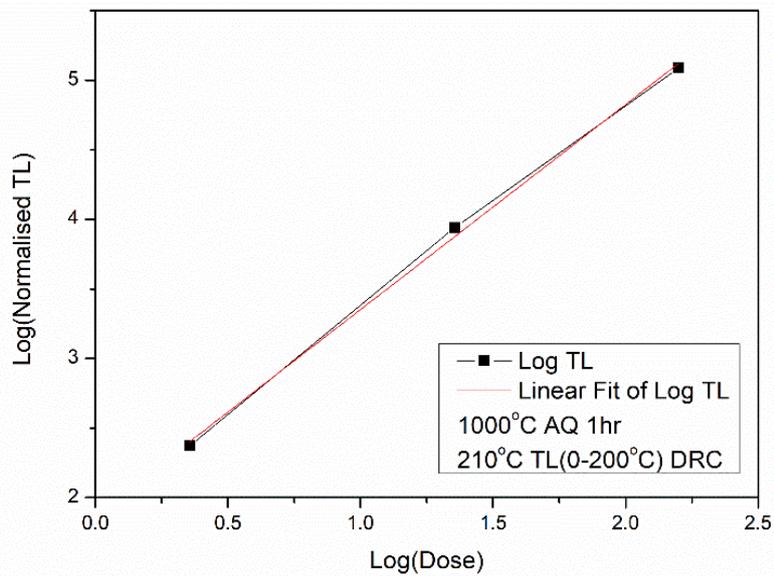


Fig.4.15 TL dose response curve of 200°C TL glow peak for 1000°C annealed sample

Kitis G et al¹⁸ have reported that the synthetic quartz showed that the whole TL dose response behavior is highly dependent on the firing temperature. Kristianpoller N et al¹⁹ reported that annealing sample at high enough temperature increases the sensitivity substantially and changes the dose dependence to be linear or nearly linear. Strong super linearity was attributed to the effect of competition during the heating phase. Present studies revealed that the annealing of synthetic quartz removes the competitor which results in an increase in the sensitivity and a decrease in the degree of super linearity. For another kind of competition, namely competition during excitation, normally expected behavior would consist of a linear-super linear-linear sequence dose response. It means that within certain dose range the degree of super linearity increases with the dose. The removal of the competitor is expected to increase the sensitivity and reduce the slope unity, which indeed occurred in some synthetic quartz crystal too. However, it has been suggested that, in premium Q synthetic quartz material, the slope of dose response curve is about ($k = 1.5$) even after an annealing at 1000°C. One can speculate that since features of both kinds of competition are present the complex results seen are due to the existence of both kinds of competitors in the sample. In present sample, as annealing temperature increases, the degree of super linearity found to be increased from $k = 0.87$ to 1.39 in 110°C TL glow peak and also for higher temperature TL glow peaks. This observation is correlated with the discussion on complexity in competitor for TL dose response and hence it suggestive to be due to existence of both kinds of competitors present in the synthetic quartz sample under annealing treatment.

4.2.4 Effect of cycle of physical treatment on TL glow curve of annealed sample.

Earlier works reported that, under influence of the annealing temperature and beta dose, the synthetic quartz material showed wide range of TL glow peaks during 0 °C to 450 °C measurement temperature. The TL properties of annealed samples are also examined under the influence of cyclic sequence of physical conditions such as thermal bleaching at desired temperature followed by test beta dose further followed by thermal bleaching at desired temperature. This study will offer the systematic information about thermally stable and unstable TL glow peak. Hence, we can erase the unwanted TL traps by selecting appropriate thermal bleaching temperature or it can transfer the charges to different localities which are optically sensitive. It will be

TB-2 from 0°C -450°C**Step-2 Beta 2.268 Gy (D₀) Do****Step-7 TB-1 from 0°C -200°C TL-5 Record from 0°C -200°C**

In Step-1, the sample was annealed at 400°C, 600°C, 800°C and 1000°C for 1hour duration. In Step-2, each annealed sample was irradiated for 2.268 Gy beta dose (say D₀). Sequentially in Step-3, the sequence (S) of physical treatments such as twice thermal bleaching (TB) at desired temperature (TB-1 from 0°C -200°C and TB-2 from 0°C -450°C) followed by test dose of 0.756 Gy further followed by twice thermal bleaching (TB) at desired temperature (TB-1 from 0°C -200°C and TB-2 from 0°C -450°C).

Similarly, the Step-3 is repeated with the new beta doses (D_n) namely 22.68 Gy(D₁) as Step-4, 75.6 Gy(D₂) and Step-5, 151.2 Gy(D₃) in Step-6 and returned back to Step-2.

During this cyclic sequence of Step-3, for the TB-1 TL glow curve measurement over 0°C -200°C was recorded for four cycles and it is designated as TL-1 record, TL-2 record, TL-3 record and TL-4 record respectively.

Further, after recording four TL measurements sample condition was returned back to Step-2, again the TB-1 was followed prior to the TL glow curve measurement as TL-5 as Step-7. As an effect of cyclic sequence of physical condition, the changes in TL-5 record are compared with TL-1 record.

It was found that the 400°C annealed sample showed that the TL-1 record exhibits, single clear 110°C TL glow peak and its intensity grow systematically from 115 counts to 10537 counts under the influence different beta doses (D_n) followed by repetition of cyclic sequence of Step-3.

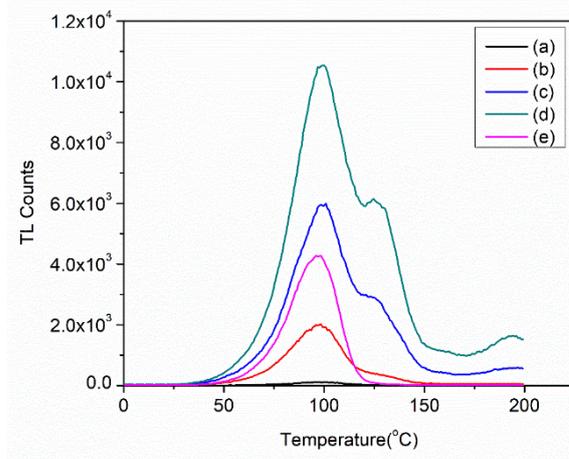


Fig4.16 TL from 0°C -200°C for annealed sample of 400°C at different cyclic sequence of physical condition
(a) D₀ (b) D₀+sequence+D₁ (c) D₀+sequence+D₁+ sequence+D₂ (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ (e)

D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀

Moreover, after returning to Step-2, the TL- 5 record exhibits 4279 counts of 110°C TL glow peak intensity which is found to be 37 times more than 110°C TL glow peak intensity of the TL-1 record. (Fig. 4.16)

This novel protocol has also been implemented to the higher annealed sample of 600°C, 800°C and 1000°C. The changes in TL glow curve pattern have been observed similar to that of the TL records of 400°C annealed sample. The position of 110°C TL glow is sustained with the significant growth of its strength in higher annealed samples compared to that of 400°C annealed sample. (Fig.4.17,4.18,4.19,4.20)

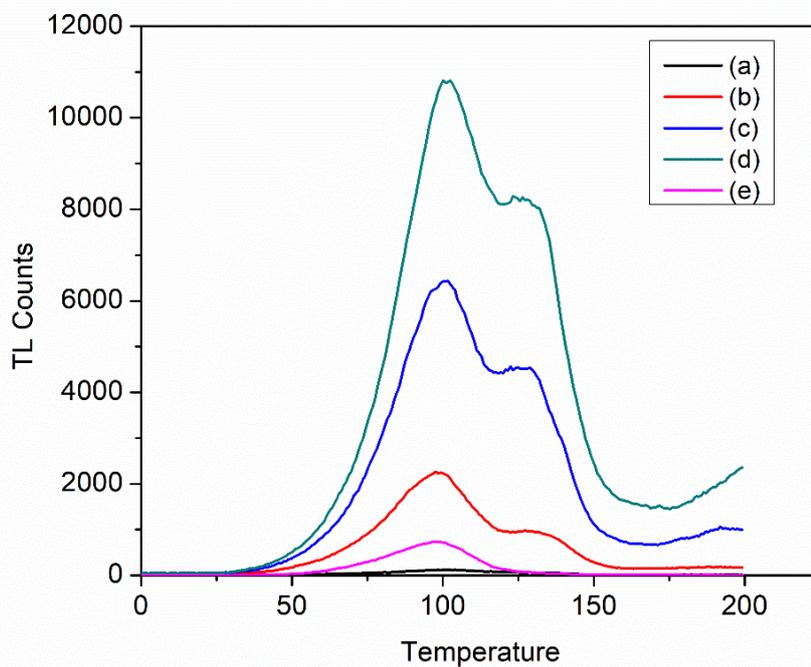


Fig. 4.17 TL from 0°C -200°C for annealed sample of 600°C at different cyclic sequence of physical condition

(a) D₀ (b) D₀+sequence+D₁ (c) D₀+sequence+D₁+ sequence+D₂ (d)

D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀

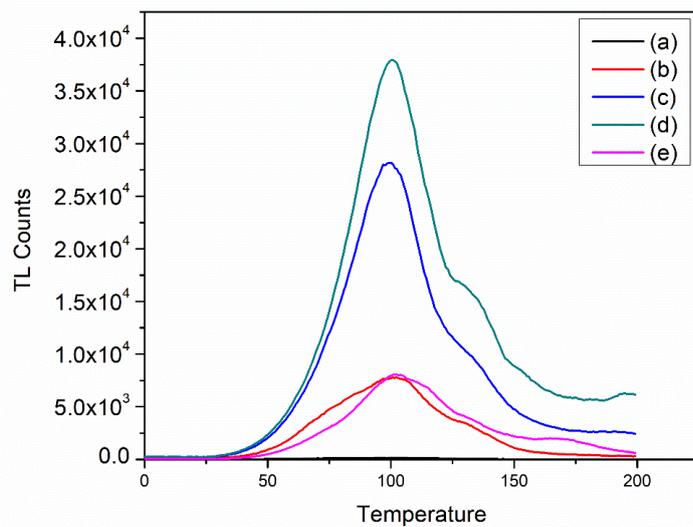


Fig. 4.18 TL from 0°C -200°C for annealed sample of 800°C at different cyclic sequence of physical condition (a) D₀ (b) D₀+sequence+D₁ (c) D₀+sequence +D₁+ sequence+D₂ (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀

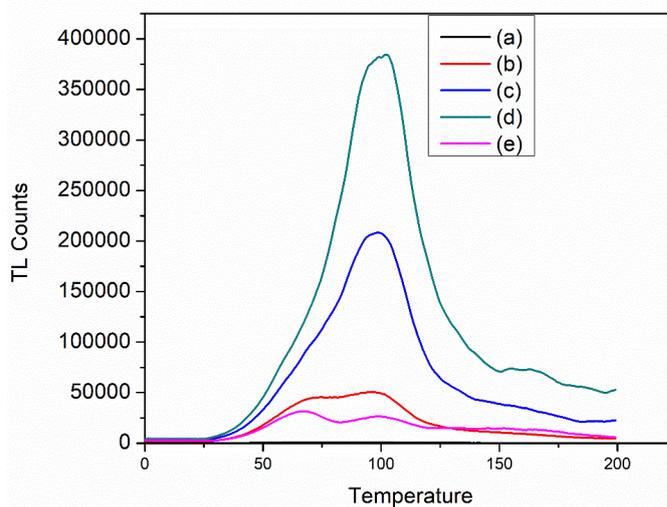


Fig. 4.19 TL from 0°C -200°C for annealed sample of 1000°C at different cyclic sequence of physical condition

(a) D₀ (b) D₀+sequence+D₁ (c) D₀+sequence+D₁+ sequence+D₂ (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀

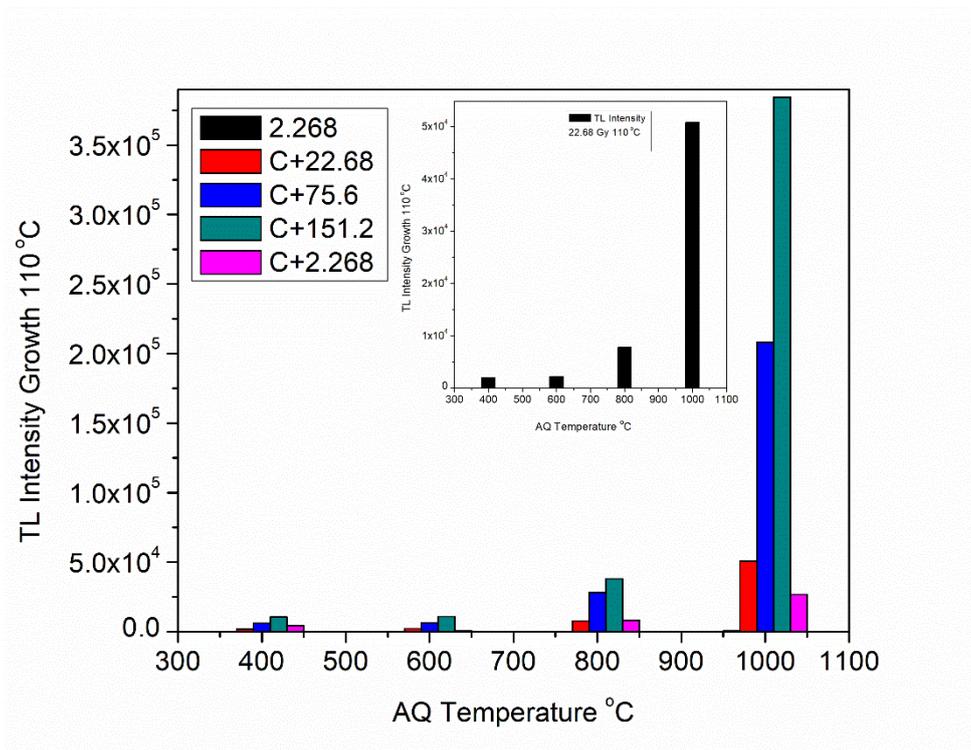


Fig. 4.20 TL sensitivity of 110°C glow peak for different annealed samples at cyclic sequence of physical condition

It is already established that post-excitation thermal treatment erases the TL signal at least up to the treatment temperature on the TL glow curve. However before erasing the TL signal, TL was recorded during the thermal bleaching TB-1 from 0°C -200°C after beta doses, gives all the information of glow peaks between 0°C -200°C measurement temperature. It showed discrete contribution of 110°C TL glow peak with significant TL intensity as a function beta doses. The growth of 110°C TL sensitivity is discernible due to the contribution of annealing temperature in accordance with the cyclic sequence. It might be giving extra thermal sensitization to the sample like pre-dose effect. Additionally, the test dose of lower magnitude may sensitize to particular traps by less mutilation in sample and it may be possible due to the predominant contribution of 110°C TL trap.

In order to study higher temperature glow peaks, the TL properties of samples with following protocols were considered.

	SQ (Grain Size 63-53μm)	
Step-1	Annealing; 400$^{\circ}$C, 600$^{\circ}$C, 800$^{\circ}$C,1000$^{\circ}$C@1hr and quenched at RT	
Step-2	Beta 2.268 Gy (D₀)	Do
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Sequence
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	TL-1 Record from 0$^{\circ}$C -450$^{\circ}$C
	Test Dose (TD)0.756 Gy	
	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	
Step-4	Beta 22.68Gy(D₁)	D₁
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Sequence
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	TL-2 Record from 0$^{\circ}$C -450$^{\circ}$C
	Test Dose (TD)0.756 Gy	
	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	
Step-5	Beta 75.6Gy(D₂)	D₂
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Sequence
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	TL-3 Record from 0$^{\circ}$C -450$^{\circ}$C
	Test Dose (TD)0.756 Gy	
	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	
Step-6	Beta 151.2Gy(D₃)	D₃
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Sequence
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	TL-4 Record from 0$^{\circ}$C -450$^{\circ}$C
	Test Dose (TD)0.756 Gy	
	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	
Step-2	Beta 2.268Gy (D₀)	Do
Step-7	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	

During cyclic sequence of Step-3, the TB-2 from 0°C -450°C as the TL glow curves measured over 0°C -450°C and it is designated as TL-1 record, TL-2 record, TL-3 record and TL-4 record respectively.

Additionally, after returning to Step-2 followed by TB-1 from 0°C -200°C, again the TB-2 from 0°C -450°C as the TL glow curve measured over 0°C -450°C and it is designated as TL-5 record of Step-8. As an effect of cyclic sequence of physical condition, the changes in TL-5 record are compared with TL-1 record.

The 400°C annealed sample shows, in the TL-1 record exhibits, the background signal. But, under different beta doses (D_n) followed by repetition of cyclic sequence of Step-3, sample exhibits average TL glow peak around 210°C and 382°C and their TL intensity increase from 28 counts to 695 counts and 21 counts to 448 counts respectively.

Additionally, after returning to Step-2 followed by TB-1 from 0°C -200°C, the TL-5 record exhibits two separates glow peak around 212°C and 386°C compared to TL-1 record. (Fig.4.21)

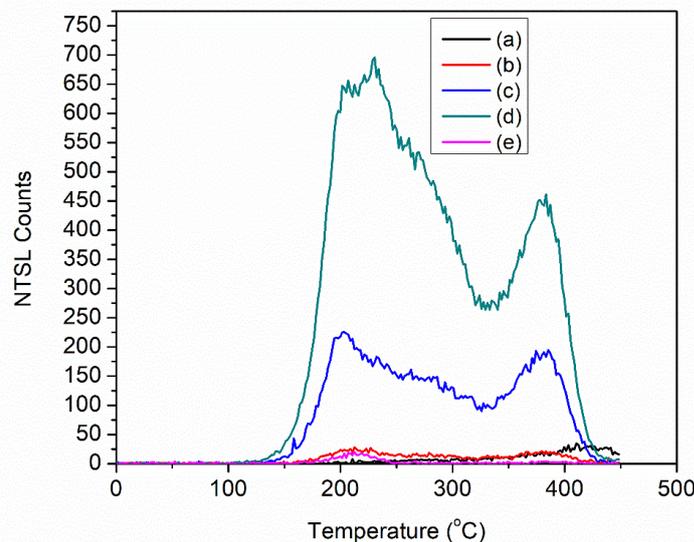


Fig. 4.21 TL from 0°C -450°C for annealed sample of 400°C at different cyclic sequence of physical condition (a) D_0 + TB-1 (b) D_0 + sequence + D_1 + TB-1 (c) D_0 + sequence + D_1 + sequence + D_2 + TB-1 (d) D_0 + sequence + D_1 + sequence + D_2 +

sequence+D₃ + TB-1 (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + TB-1

Further, this novel protocol has implemented to the higher annealed sample of 600°C, 800°C and 1000°C and the changes in TL glow peak pattern have recorded like the TL records of 400°C annealed sample. It is observed that each higher annealed samples exhibit two TL glow peaks with significant TL intensity like 400°C annealed sample. The average TL glow peak around 215°C is more stabilized in 1000°C annealed sample. However, the position of average 382°C TL glow is independent with respect to annealing temperature and their average glow peak position is observed around 290°C, 302°C and 370°C in 600°C, 800°C and 1000°C annealed samples respectively. (Fig.4.22,4.23,4.24)

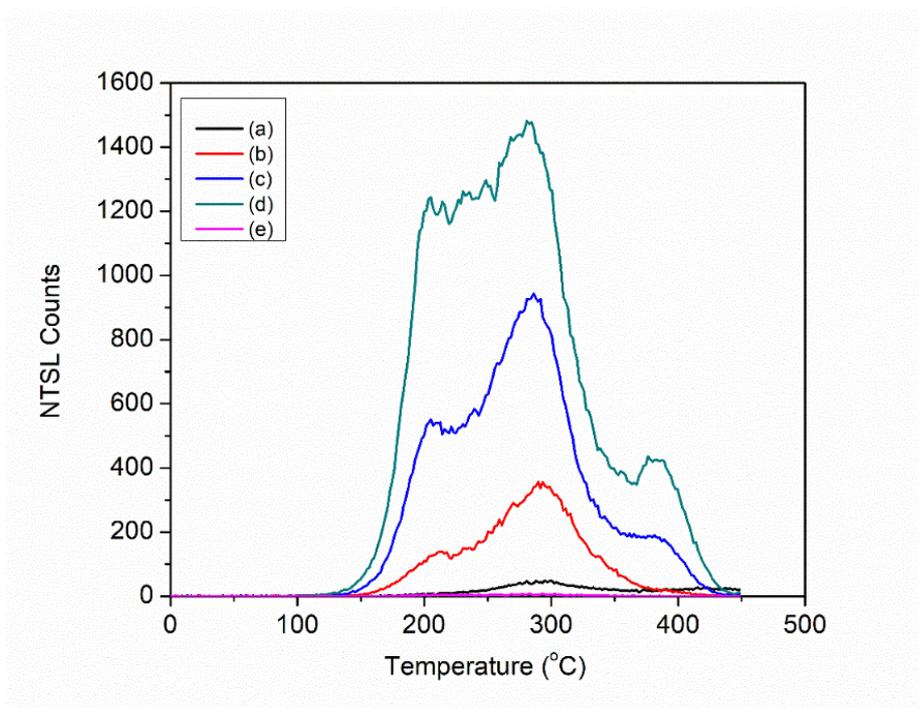


Fig. 4.22 TL from 0°C -450°C for annealed sample of 600°C at different cyclic sequence of physical condition (a) D₀+TB-1 (b) D₀+sequence+D₁ + TB-1 (c) D₀+sequence+D₁+ sequence+D₂ + TB-1 (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ + TB-1 (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + TB-1

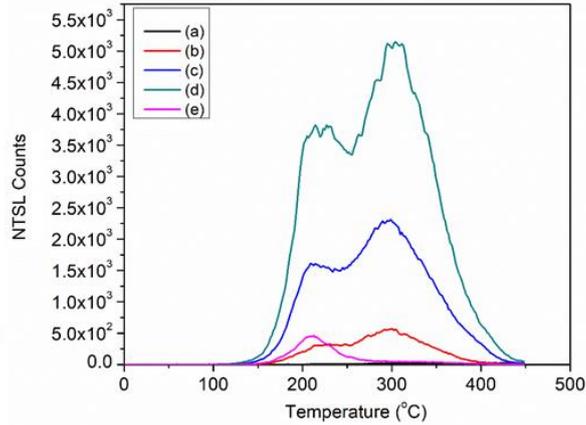


Fig. 4.23 TL from 0°C -450°C for annealed sample of 800°C at different cyclic sequence of physical condition (a) $D_0 + TB-1$ (b) $D_0 + \text{sequence} + D_1 + TB-1$ (c) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + TB-1$ (d) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + TB-1$ (e) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + \text{sequence} + D_0 + TB-1$

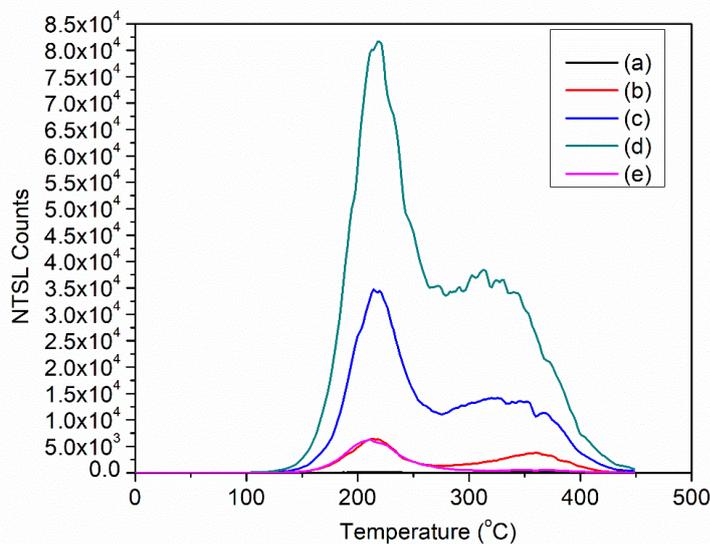
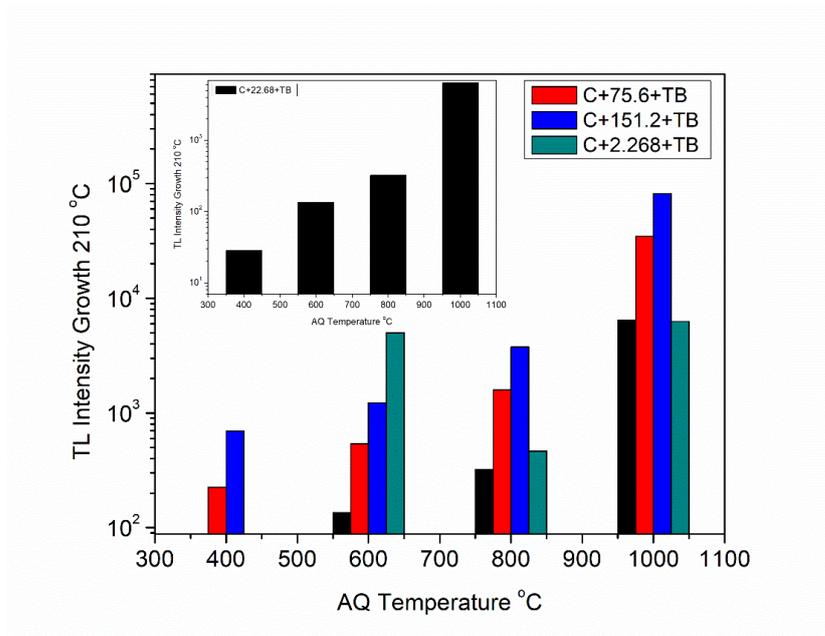


Fig. 4.24 TL from 0°C -450°C for annealed sample of 1000°C at different cyclic sequence of physical condition (a) $D_0 + TB-1$ (b) $D_0 + \text{sequence} + D_1 + TB-1$ (c) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + TB-1$ (d) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + TB-1$ (e) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + \text{sequence} + D_0 + TB-1$



Additionally, the sensitivity of average TL glow peak around 215°C is examined and it is observed that TL sensitivity of this increases with annealing treatment followed by repetition of cyclic sequence of Step-3. Fig.4.25

Fig. 4.25 TL sensitivity of 215°C glow peak for different annealed samples at cyclic sequence of physical condition

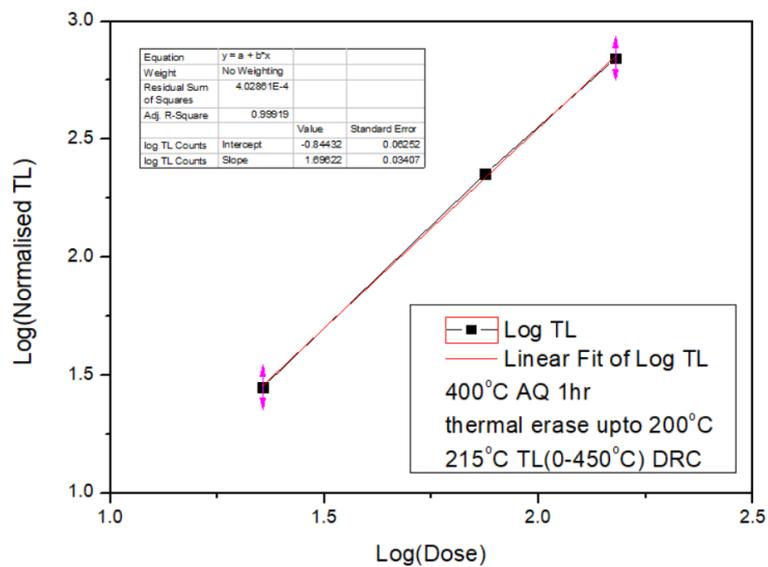


Fig.4.26 TL dose response curve of 215°C TL glow peak for 400°C annealed sample

The TL-DRC nature of average contributed TL glow peak around 215°C glow peak is examined. It shows super linear TL-DRC nature for each annealed sample but the degree of super linearity decreases with rise in annealing temperature.

(Fig. 4.26,4.27,4.28,4.29)

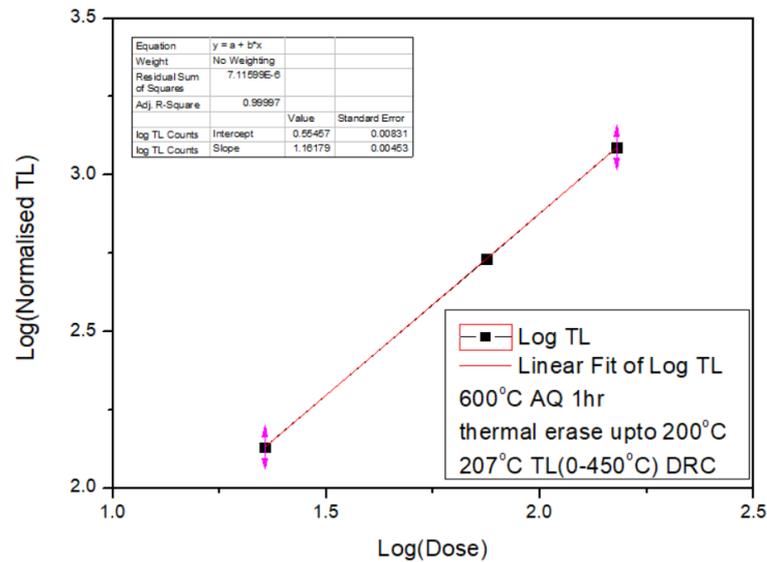


Fig.4.27 TL dose response curve of 207°C TL glow peak for 600°C annealed sample

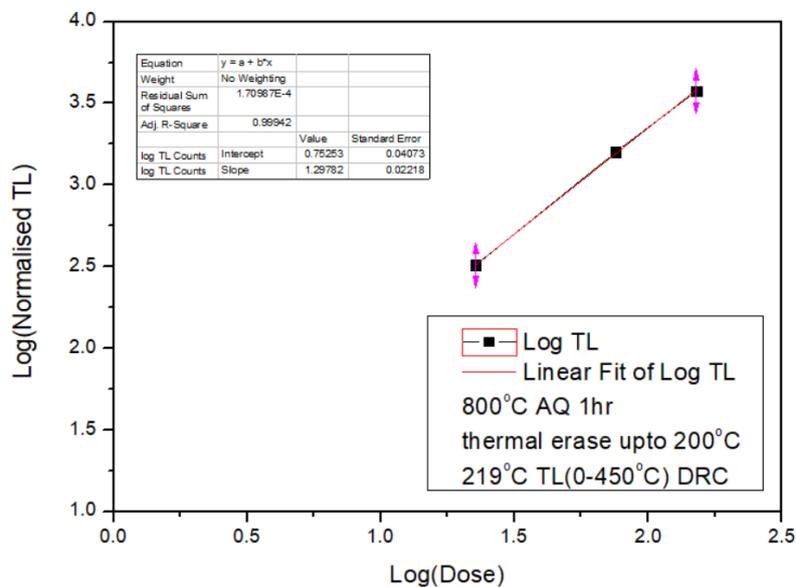


Fig.4.28 TL dose response curve of 219°C TL glow peak for 800°C annealed sample

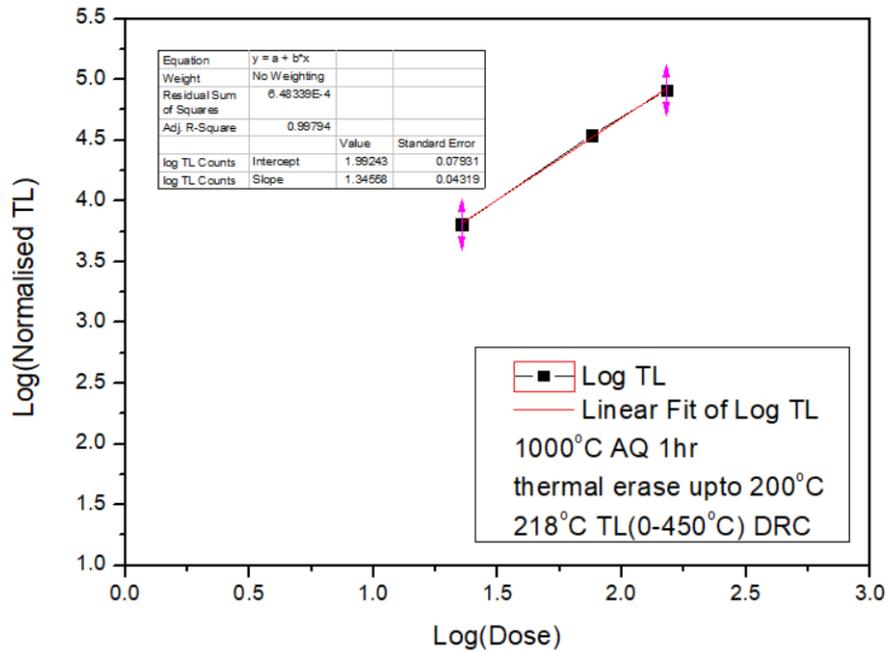


Fig.4.29 TL dose response curve of 219°C TL glow peak for 1000°C annealed sample

Preheating or thermal washing is often used to remove an unstable component in thermoluminescence dating and has been used in the optical dating of quartz. Because the majority of the OSL signal from quartz is correlated to the 325°C TL peak. A preheating procedure was developed which was based on the TL behaviour of this peak. Huntley et al²⁰ have reported that thermally unstable component is not stable at ambient temperature over the relevant geological time period. If this component is not removed by appropriated pre-thermal treatment, equivalent dose and hence age of the sample will be underestimated.

The under influence of cyclic sequence of physical condition on annealed sample followed by dose, in present investigation, represent the contribution of shallow 110°C TL glow peak is predominant and it may contribute as thermally unstable components over 0°C -200°C measurement temperature. The contribution of these TL traps is eliminated by contribution of new TL traps around 202°C -212°C, 290°C -295°C, 300°C, 330°C, 365°C and 386°C glow peaks over 0°C -450°C measurement temperature. It may participate in OSL process and support to enhance OSL efficiency followed by determination of OSL components responsible for OSL process.

4.3 Optically Stimulated Luminescence (OSL) study of synthetic quartz.

Optically stimulated luminescence technique is an established tool for estimating the dose of different phosphors and determining the age of geological / archaeological samples due to some technical advantages over TL technique. It can minimize TL loss either by re-capturing electrons with low traps, the same traps, different locations and deep TL traps, or a heat treatment process. In OSL technique, during optical stimulation, optically sensitive electron traps are depleted and electrons are recombined to a hole in the recombination center through a conduction band having a conventional (exponential) shape of the OSL decay curve over time.²¹

The pattern of OSL decay curve reflects the electron recombination tendency from trap to the hole at recombination center. It is resolved by deconvolution of the OSL decay curve and thus determining the decay tendency by means of fast, medium and slow components. Like TL, these OSL results are affected by physical treatment of the sample or experimental conditions such as ionizing radiation, annealing temperature, stimulation wavelength, stimulation temperature, thermal bleaching at the desired temperature. During optical stimulation at room temperature, the quartz sample shows an unusually shaped OSL decay curve followed by a weaker amount of OSL, representing the re-combination of optically released electrons at low traps corresponding to 110°C TL traps rather than recombination at the center which arises the loss of OSL signal with poor efficiency.²² For dating and other applications, this loss of OSL signal with poor efficiency problem was addressed by attempting various protocols to the sample. To optimise the OSL output usual exponential OSL decay curve is desired, as it avoids the contribution of 110°C TL trap contribution and enhances the chances of output from slowly bleachable peaks.

Kale Y et al²³ showed that below critical physical conditions (either lower annealed sample irradiated by higher beta dose or higher annealed sample irradiated by lower beta dose) in the sample, the material did not show the conventional shape of the OSL decay curve and did not yield better OSL readings. To avoid this problem, recommendation received from various researchers to use thermally-assisted optically stimulated luminescence (TA-OSL) technique. This technique is being performed on various luminescence phosphor either natural or synthetic. In the TA-OSL technique, OSL is performed in a combined operation of thermal and optical stimulation, and thus a conventional OSL decay curve is possible with a better OSL

outcome due to removal of re-trapping of an optically released electron by shallow traps corresponding to 110°C TL trap.²⁴

Kitis G et al²⁵ reported complex nature of luminescence signals arising from quartz samples in TL and OSL studies. They attributed it to these signals originating from several electron and hole traps, as well as from several recombination centres. For OSL dating application, the signal is measured at an elevated temperature of 125°C, in order to avoid problems associated with the 110°C TL trap. It is assumed that the OSL signal measured at 125°C consists of several light-sensitive components, commonly referred to as fast, medium, and slow OSL components.

However, as a TA-OSL measurement, researchers have reported that optical stimulation at ~ 160°C is sufficient to limit the recombination of electron with low traps and to aid better OSL. Based on these, below the critical level of physical conditions, the unusual shape of the OSL decay curve recorded at 160°C is still observed in synthetic quartz material. It is suggested that the optically released electron did not follow the normal recombination pathway and had been trapped at sites other than the normally low traps Kale Y.²⁶ In addition, researchers have recommended thermally transferred OSL (TT-OSL) technique, in which the largest contribution of an electron is obtained from deep optically sensitive traps.²⁷

On natural / laboratory irradiated sample, preheating or thermal washing at the desired temperature, supports electron heat transfer / removal from unwanted TL traps, low traps, and optically insensitive traps to optically sensitive traps, provides significant effective OSL data. But below the critical level of the physical conditions of the synthetic quartz sample, an unusual shape of the OSL decay curve was still observed, even if the exposed sample was preheated to 290°C for the desired time, followed by optical stimulation at 160°C. Therefore, it means synthetic quartz crystals need a suitable revised protocol that provides normal decay curve of OSL at a significant intensity and resolves the components responsible for OSL decay. However, in this study, OSL decay curves are recorded at 125°C according to the new proposed protocol for synthetic quartz material.

4.3.1 Effect of repetition of cyclic sequence of physical condition on OSL decay.

Earlier works reported that the combined effect of annealing treatment and irradiation on synthetic quartz material has a wide measuring range of TL glow peaks. In addition to low TL traps, the contributing higher temperature TL glow peaks can be dual in nature, such as thermally and optically sensitive. The effect of these traps may be possible for significant OSL followed by the conventional form of decay curve, and it may be possible to estimate the responsible contribution of such traps for OSL decay. Therefore, efforts have been made to obtain the maximum proportion of these traps in OSL, after which a correlation with TL has been found.

In view of these, the following protocol is followed to record the OSL decay curves at 125°C for 40 seconds of stimulation time and OSL output obtained in terms of shape of OSL decay, intensity and the responsible components OSL decay are investigated systematically.

	SQ (Grain Size 63-53µm)	
Step-1	400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour	
Step-2	Beta 2.268Gy (D₀)	D₀
Step-3	Thermal Bleaching-1(0°C -200°C)	Sequence
	Optical Bleaching for 40 sec at 125°C	Recorded OSL-1 at 125°C
	TD 0.756Gy	
	Thermal Bleaching-1(0°C -200°C)	
	Optical Bleaching for 40 sec at 125°C	
Step-4	Beta 22.68Gy(D₁)	D₁
Step-3	Thermal Bleaching-1(0°C -200°C)	Sequence
	Optical Bleaching for 40 sec at 125°C	Recorded OSL-2 at 125°C
	TD 0.756Gy	
	Thermal Bleaching-1(0°C -200°C)	

	Optical Bleaching for 40 sec at 125°C	
Step-5	Beta 75.6Gy(D₂)	D₂
Step-3	Thermal Bleaching-1(0°C -200°C)	Sequence
	Optical Bleaching for 40 sec at 125°C	Recorded OSL-3 at 125°C
	TD 0.756Gy	
	Thermal Bleaching-1(0°C -200°C)	
	Optical Bleaching for 40 sec at 125°C	
Step-6	Beta 151.2Gy(D₃)	D₃
Step-3	Thermal Bleaching-1(0°C -200°C)	Sequence
	Optical Bleaching for 40 sec at 125°C	Recorded OSL-4 at 125°C
	TD 0.756Gy	
	Thermal Bleaching-1(0°C -200°C)	
	Optical Bleaching for 40 sec at 125°C	
Step-2	Beta 2.268Gy(D₀)	D₀
Step-6	Thermal Bleaching-1(0°C -200°C)	
Step-7	Optical Bleaching for 40 sec at 125°C	Recorded OSL-5 at 125°C

In Step-1, the sample annealed at 400°C, 600°C, 800°C and 1000°C for 1hour duration. In Step-2, each annealed sample was irradiated by 2.268 Gy beta dose (say D₀). The sequence of physical treatments such as thermal bleaching (TB) at selected temperature (TB-1 from 0°C -200°C) followed by Optical Bleaching (OB-1) at 125°C for 40 seconds followed by test dose (0.756 Gy) followed by thermal bleaching (TB) at selected temperature (TB-1 from 0°C -200°C) followed by Optical Bleaching (OB-1) at 125°C for 40 seconds is implemented as Step-3. The Step-3 is repeated after the new beta doses (D_n) namely 22.68 Gy(D₁) in Step-4, 75.6 Gy(D₂) in Step-5, 151.2 Gy(D₃) in Step-6 and then returned back to Step-2, where again sample was irradiated by 2.268 Gy beta dose as D₀.

During cyclic sequence of Step-3, for each beta doses (D_n) and TB-1 from 0°C -200°C, the OSL decay curves were recorded at 125°C for 40 seconds. These OSL records are represented as OSL-1 record, OSL-2 record, OSL-3 record and OSL-4 record respectively. Further, after returning to Step-2, again TB-1 from 0°C -200°C followed by OSL decay curve recorded at 125°C for 40 seconds as one more optical bleaching, which is represented as OSL-5 in Step-7. To bring out effect of cyclic sequence of physical condition on synthetic quartz the changes in OSL-5 decay curves are compared with OSL-1 record.

The 400°C annealed sample shows usual shape of OSL decay with 28 counts of OSL intensity in OSL-1 record. It increases to 701 counts in OSL-4 record which is 25 times higher under the influence of different beta doses (D_n) followed by repetition of cyclic sequence of Step-3. In yet another significant observation it is found that after returning to Step-2 counts of the OSL-5 record exhibited double (56 Counts) growth in OSL intensity compared to OSL-1 record. (Fig.4.30)

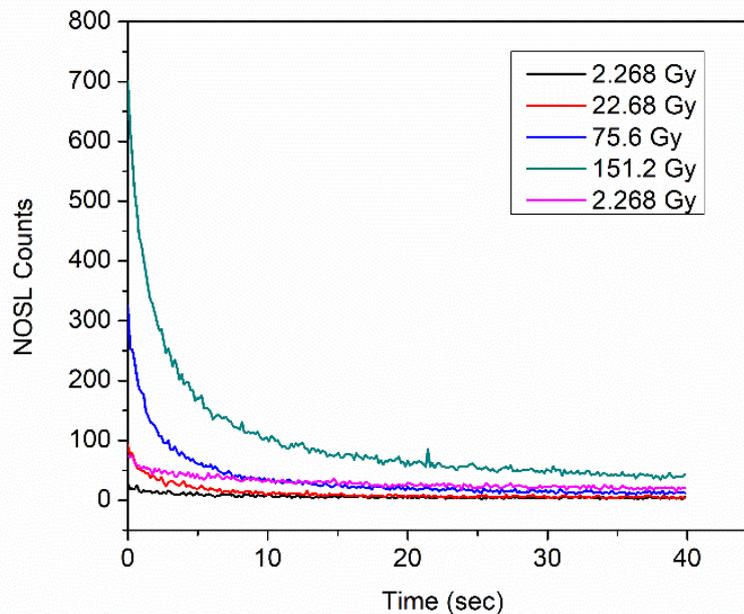


Fig. 4.30 OSL at 125°C for annealed sample of 400°C at different cyclic sequence of physical condition

- (a) D_0 + TB-1 (b) D_0 + sequence + D_1 + TB-1 (c) D_0 + sequence + D_1 + sequence + D_2 + TB-1 (d) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + TB-1 (e) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence + D_0 + TB-1**

Further, the identical protocol has been implemented to the higher annealed sample of 600°C, 800°C and 1000°C also. The changes in shape of OSL decay pattern have been observed like the OSL records of 400°C annealed specimen. The shape of OSL decay pattern is identical with significant strength in higher annealed samples compared to 400°C annealed sample. **(Fig.4.31,4.32,4.33)**

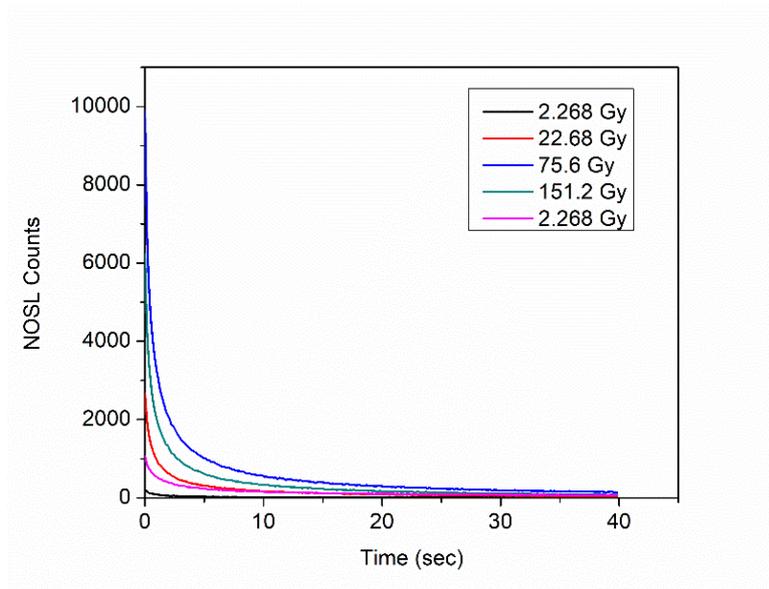


Fig. 4.31 OSL at 125°C for annealed sample of 600°C at different cyclic sequence of physical condition (a) $D_0 + TB-1$ (b) $D_0 + \text{sequence} + D_1 + TB-1$ (c) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + TB-1$ (d) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + TB-1$ (e) $D_0 + \text{sequence} + D_1 + \text{sequence} + D_2 + \text{sequence} + D_3 + \text{sequence} + D_0 + TB-1$

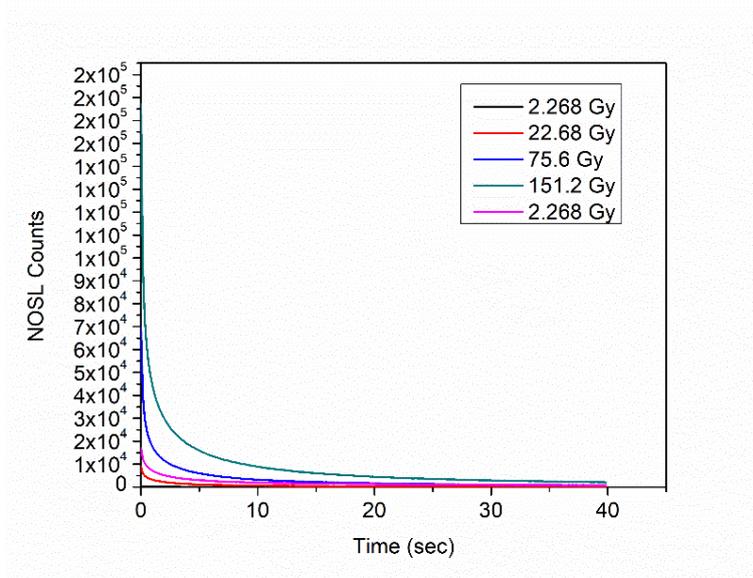


Fig. 4.32OSL at 125°C for annealed sample of 800°C at different cyclic sequence of physical condition (a) D₀+TB-1 (b) D₀+sequence+D₁ + TB-1 (c) D₀+sequence+D₁+ sequence+D₂ + TB-1 (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ + TB-1 (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + TB-1

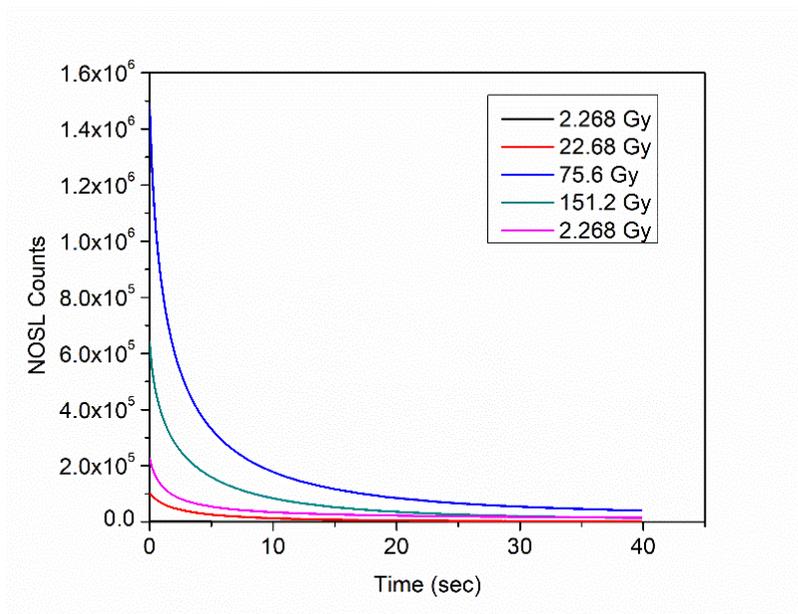


Fig. 4.33 OSL at 125°C for annealed sample of 1000°C at different cyclic sequence of physical condition (a) D₀+TB-1 (b) D₀+sequence+D₁ + TB-1 (c) D₀+sequence+D₁+ sequence+D₂ + TB-1 (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ + TB-1 (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + TB-1

The present study clearly reveals that the OSL intensity increases by more than 95 % of sensitization with annealing treatment followed by repetition of cyclic sequence of Step-3 to the sample. (Fig.4.34)

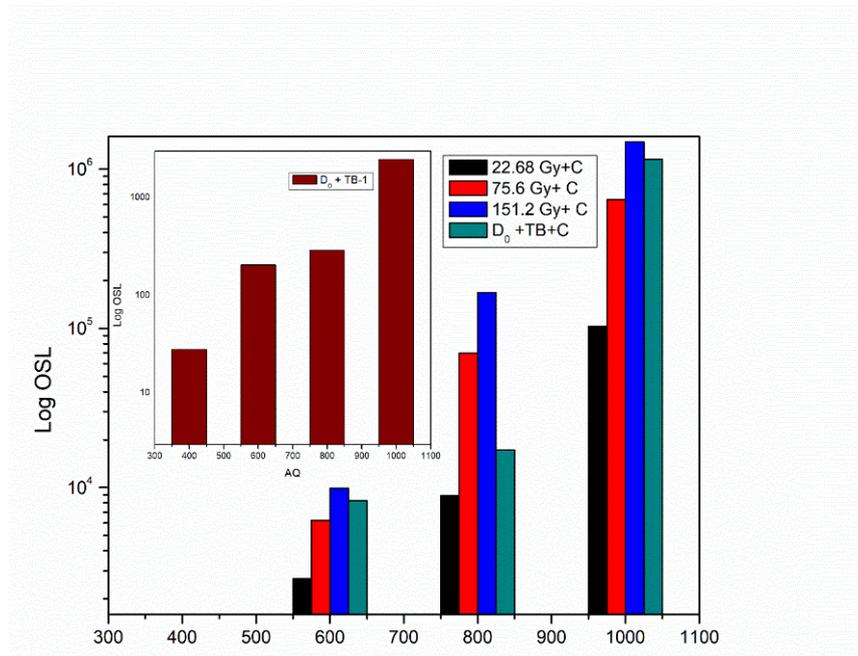


Fig. 4.34 Sensitivity of OSL at 125°C for different annealed samples at cyclic sequence of physical condition

(a) D₀ +TB-1 (b) D₀+sequence+D₁ + TB-1(c) D₀+sequence+D₁+ sequence+D₂ + TB-1 (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ + TB-1 (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + TB-1

In optical dating, the purpose of thermal annealing or preheating was to isolate a thermally stable component, and thus it has been proposed to properly determine the corresponding dose for preheating both natural and laboratory irradiated samples. The effects of preheating a quartz sample are complex; natural and laboratory irradiated samples often show an enhancement of their OSL signals when they are annealed at temperatures above 200°C. It has been attributed to both thermal charge transfer from light insensitive traps to the OSL traps and sensitivity changes due to increase of luminescence efficiency.²⁸ Vartanian E et al²⁹ have reported that preheating is necessary to remove unstable components of OSL. During preheating, charges from low traps are thermally depleted so that only long-lived traps remain.

According to the new proposed protocol, each sample has a common OSL decay properties with significant findings that the strength of annealing treatment supports to minimize competitor in the sample. Desired thermal bleaching eliminates the contribution of shallow TL traps in TL or transfer the charges to deeper traps which have been observed as wider span of higher temperature TL, which might have been contributed as optically sensitive traps. These proposed mechanisms behind the charge transfer process in the sample in the double heat treatment and their repetition as a cyclic sequence may be responsible for the desired result of conventional form of decay with significant amounts of OSL.

As mentioned in the literature, during preheating at the desired temperature, the charges of the low traps are thermally discharged so that only long-term trapped charges remain. Obviously, these long-lived traps may have sufficient strength to hold heat transfers. In the present TL results for annealing treatment and repeating the cyclic sequence of the physical state in step 3, significant new higher temperature TL traps are developed at about 202^oC to 212^oC, 290^oC to 295^oC, 300^oC, 330^oC, 365^oC, and glow at 386 ^oC which is discernible while recording TL in measuring temperature range of 0^oC -450^oC. These proposed TL traps could be long-lasting and optical in nature. It may be responsible for the normal form of OSL decay with enhanced OSL intensity.

4.3.2 OSL-Dose Response study under repetition of cyclic sequence of physical condition.

Like well-known technique as TL for dosimetry, OSL is also gaining attraction for dosimetry application. In a previous work, a sample of different exposures (D_n), followed by TB-1 from 0-200^oC during step 3, the electrons accumulated in traps are thermally transferred to higher temperature traps above 200^oC. The higher temperature traps those are optical sensitive in nature, obviously is participated in OSL process predominantly and hence their OSL-DRC can be obtained. Therefore, it is necessary to record the OSL-DRC dose response curve of optical sensitive traps in synthetic quartz materials.

Similar to TL-DRC measurement, the OSL-DRC follows

$$OSL = a(D)^k$$

where, *a* is constant, *D* is applied dose and *k* value is related to slope of the DRC and it determines the nature of the curve over log *OSL* against log *D* scale.

It is observed that nearly each annealed sample displays the growth in OSL counts with rise in the beta doses. Here, the slope value of OSL-DRC is observed as $k > 1$ and hence it represents super linear nature of OSL dose response curve of synthetic quartz material. It is also noteworthy to observe that with rise in annealing temperature to the sample, the degree of super linearity also increases. (Fig.4.35,4.36,4.37,4.38).

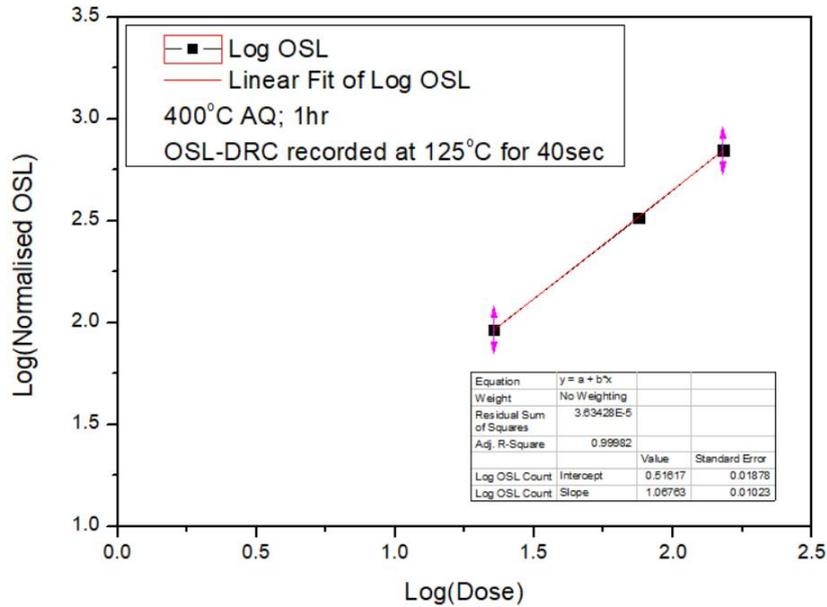


Fig.4.35 OSL dose response for 400°C annealed sample

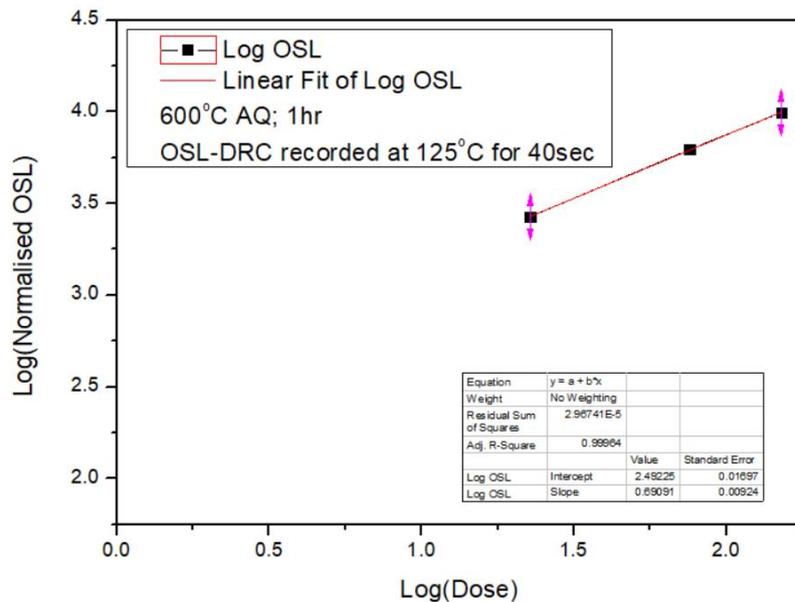


Fig.4.36 OSL dose response for 600°C annealed sample

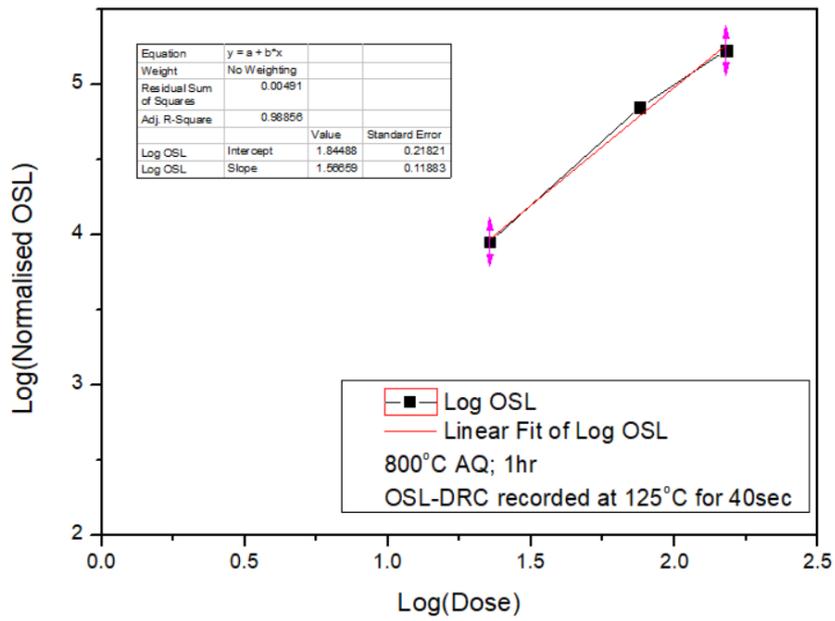


Fig.4.37 OSL dose response for 800°C annealed sample

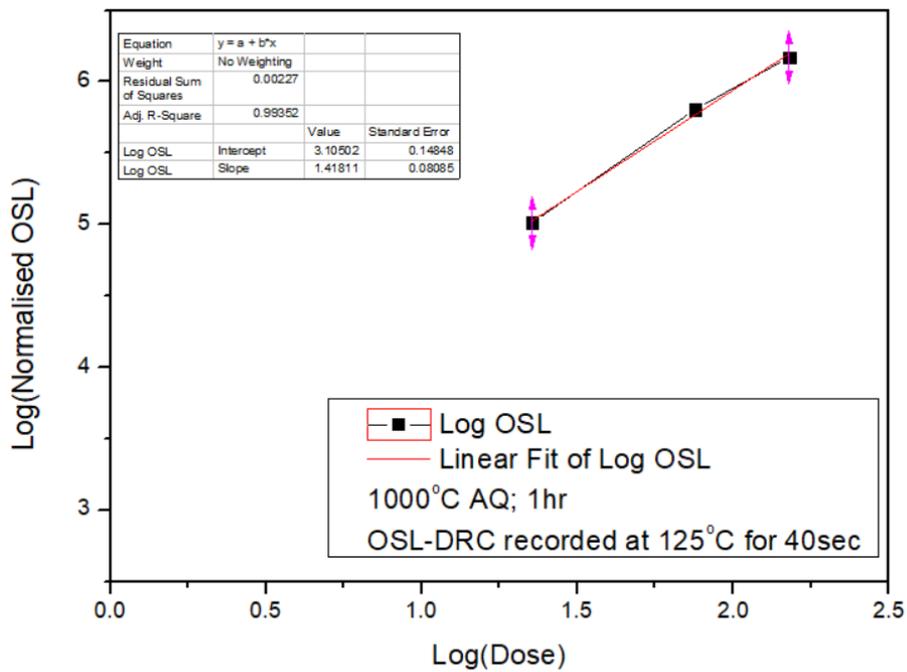


Fig.4.38 OSL dose response for 1000°C annealed sample

It has been suggested that like TL-DRC under repetition of cyclic sequence of physical condition for annealed sample, the OSL-DRC also shows super linear nature for each annealed sample. However, it does not give the noticeable relation between the changes in degree of super linearity in OSL-DRC nature with rise in annealing temperature. Therefore, the interpretation for OSL-DRC under repetition of cyclic sequence of physical treatments might be considered as identical as interpretation of TL-DRC under at different annealing temperature.

4.3.3 Deconvolution study of OSL at 125^oC under sequence of physical condition

In general, the nature of the OSL decay curve is conventional (exponential), which means that electron-hole recombination occurs via a common path. The pattern of OSL decay curves represent the nature of recombination of electron from trap into hole at recombination center and it is classified as fast(F), medium(M) and slow(S) components of OSL. The nature of the recombination is greatly influenced by the physical condition of the sample and the experiment.

According to Bailey et al.³⁰ three major components have been observed in many quartz samples and are informally referred to as fast, medium, and slow components. The contributing percentage of distribution of these components of OSL decay determines whether electrons are depleted from fast, slow, medium bleaching traps or shallow unstable traps. Like the overlapping TL glow curve pattern of quartz, it is necessary to solve the complexity model of the OSL decay curve that will help to understand the responsible component for OSL or optically sensitive traps. It may become interpretation line for OSL and TL correlation.

In present investigations, the attempts have made to resolve the OSL components by fitting first order exponential equation $y = A_1 * \exp\left(\frac{-x}{t_1}\right) + A_2 * \exp\left(\frac{-x}{t_2}\right) + A_3 * \exp\left(\frac{-x}{t_3}\right) + y_0$ to the exponential shape of decay curve through ORIGIN8 commercial software.³¹

The output of equation represented y as Intensity, x as stimulation time, A_1 , A_2 and A_3 as amplitudes, t_1 , t_2 and t_3 as factors required for electrons to recombine with hole at recombination center and their reciprocal represent the value of decay constant which is denoted by (f) or (λ). Offset intensity is associated with background signal and it represents by y_0 . Results of these studies support the discussion line

about the nature (fast, medium and slow) of decay or optically sensitive traps and contribution percentage of the components in OSL process.

The outcome of OSL deconvolution reports that, for each annealed sample subjected to different beta doses and repetition of Step-3 gives major contribution of slow components and moderate contribution of medium components of OSL decay curve. However, these contribution patterns remain identical with the changes in annealing treatment subjected to given beta doses and repetition of Step-3.

										OSL components			
										F(%)	M (%)	S(%)	
400°C AQ; 1hr													
D0	TB-1									OSL-1 Record at 125°C	0.87	10.66	88.47
D0	Sequence	D1	TB-1							OSL-2 Record at 125°C	7.6	41.21	51.18
D0	Sequence	D1	Sequence	D2	TB-1					OSL-3 Record at 125°C	9	36.5	54.54
D0	Sequence	D1	Sequence	D2	Sequence	D3	TB-1			OSL-4 Record at 125°C	4	32.94	63.06
D0	Sequence	D1	Sequence	D2	Sequence	D3	Sequence	D0	TB-1	OSL-5 Record at 125°C	5.59	-	94.4
600°C AQ; 1hr													
D0	TB-1									OSL-1 Record at 125°C	7.63	28.81	63.56
D0	Sequence	D1	TB-1							OSL-2 Record at 125°C	7.07	29.45	63.47
D0	Sequence	D1	Sequence	D2	TB-1					OSL-3 Record at 125°C	7.78	30.29	61.63
D0	Sequence	D1	Sequence	D2	Sequence	D3	TB-1			OSL-4 Record at 125°C	6.95	29.37	63.68
D0	Sequence	D1	Sequence	D2	Sequence	D3	Sequence	D0	TB-1	OSL-5 Record at 125°C	3.34	25.02	71.64
800°C AQ; 1hr													
D0	TB-1									OSL-1 Record at 125°C	1.8	18.55	79.65

D0 Sequence	D1 TB-1							OSL-2 Record at 125°C	5.08	69.18	25.74
D0 Sequence	D1 Sequence	D2 TB-1						OSL-3 Record at 125°C	8.5	23.85	67.65
D0 Sequence	D1 Sequence	D2 Sequence	D3 TB-1					OSL-4 Record at 125°C	7.74	22.6	69.66
D0 Sequence	D1 Sequence	D2 Sequence	D3 Sequence	D0 TB-1				OSL-5 Record at 125°C	4	28.86	71.04
1000°C AQ; 1hr									F(%)	M	S(%)
									(%)	(%)	(%)
D0 TB-1								OSL-1 Record at 125°C	6.56	52.67	40.77
D0 Sequence	D1 TB-1							OSL-2 Record at 125°C	5.57	33.3	61.12
D0 Sequence	D1 Sequence	D2 TB-1						OSL-3 Record at 125°C	2.54	22.75	74.71
D0 Sequence	D1 Sequence	D2 Sequence	D3 TB-1					OSL-4 Record at 125°C	3.94	27.13	68.9
D0 Sequence	D1 Sequence	D2 Sequence	D3 Sequence	D0 TB-1				OSL-5 Record at 125°C	6.2	29.1	64.7

D₀=2.268Gy

D₁=22.68Gy

D₂=75.6Gy

D₃=151.2Gy

Sequence=TB-1 from 0°C -200°C + OB-1 at 125°C for 40sec + Test Dose 0.75Gy + TB-1 from 0°C -200°C + OB-1 at 125°C for 40sec

Table. 4.1 Components of OSL decay under cyclic sequence and thermal bleaching

Peng Jun and Wang Xulong³², explained the OSL production of the medium component for a coarse-grained quartz sample. Analysis of OSL decay curves measured at different stimulation temperatures revealed that there was a strong interaction between the initial states of the fast and medium OSL components. Preheating experiments showed that the thermal stability of the medium OSL component was almost the same as that of the fast OSL component. These results suggested that both fast and medium OSL components originated from the same source trap. It is remarkable to note that the production of the medium OSL component was simulated using a kinetic model in which the medium OSL signal was originated from the 325^oC TL trap and the corresponding charge was re-trapped at the 170^oC TL trap due to the phototransfer effect. A good match between measured and simulated OSL decay curves was observed.

It can be correlated from the Figure 4.46 that the increase in annealing temperature to the sample up to 800^oC, the contributions of broad TL glow peaks around 290^oC -300^oC and deeper 380^oC TL glow peak are prominent. Whereas the proportion of TL glow peaks at 176^oC, 228^oC, 320^oC, and 377^oC is observed the annealed sample at 1000^oC. In addition, the effect of these TL glow peaks appeared as a result of the cyclic repetition, cycle of annealing treatment and physical treatment in step 3. Therefore, it is reasonable to suggest that the centers corresponding to 228^oC and 320^oC are identical and thus may act as a mean component of OSL. The center corresponding to the 380^oC TL peak is associated with the slower components of the OSL which justifies that the higher annealed sample support restoration of the ~ 220^oC TL glow peak which is associated with the Ge center. This offered suggestion is also confirmed by the ESR study.

Bailey R M has reported that³³ that the slow component of quartz OSL exhibits several properties that clearly distinguish it from the main part of the quartz OSL signal ('fast bleaching') traditionally used for dating. These properties include very high thermal stability, dose saturation level, and charge concentration dependence in both signal form and decay rate. The physical mechanism responsible for the slow component is currently believed to comprise a direct donor and acceptor recombination component perhaps associated with competing pathways below, and possibly up to, the conduction band.

Present work reveals that the existence of deeper traps such as 362⁰C under higher beta irradiation of 22.68Gy and 158.76Gy, 365⁰C and 386⁰C with annealing treatment at 600⁰C, 800⁰C, and 1000⁰C followed by above referred beta doses. Also deeper traps corresponding to 370⁰C is clearly observed under a cyclic sequence of physical treatment in Step-3. It could be supported to correlate with the thermal stability of TL glow peak, intensity, and slower bleachability of TL glow which is in well agreement with the slower components of OSL decay.

In the OSL process, it is very important to consider the role of photoionization cross section (PIC). It can explain whether a particular trap is optically sensitive / active or not and whether all TL traps are optically active or not. These questions can be answered with lower or higher PIC values at a given stimulation wavelength. Photoionization cross section (PIC) describes how stimulation light interacts with an electron trapped in a potential well to be released optically. The PIC area is calculated for the fast, medium, and slow components of the OSL decay curves by repeating the physical cyclic cycle in step 3. A well-established photoionization cross section (PIC) equation was used in this calculation.³⁴

$$f = (\sigma_p) \left(\frac{I}{h\nu} \right)$$

where; f is decay constant in second⁻¹ σ_p is photoionization cross section in meter², I is Intensity of optical stimulation light in Watt/meter², h is Plank Constant in Joule. Second and ν is frequency of optical stimulation light in Hertz. Re-arrange the equation to make σ_p as a subject of equation and hence the photoionization cross-section (PIC) has been calculated by

$$\sigma_p = \left(\frac{hc}{I\lambda} \right) (f)$$

where; $\nu = c/\lambda$ and c is speed of light in *meter per second* and λ is wavelength of optical stimulation light in *meter*.

These studies provide information on the effect of OSL components by the physical treatment to the sample. They are represented as the photoionized cross-sectional area in which optically sensitized charges being accumulated. Therefore, present studies reveal that the fast component of the OSL decay curve has a cross-sectional area of lager photoionization rather than the photoionization cross-sectional area of the medium and slow components. It has been also established that an optically

sensitive trap is slow and moderately bleaching in nature. In addition to this, the present work aim to investigate the effect of test dose followed by 0-200°C TB-1 on the OSL decay curve. For these OSL measurements, the experimental protocol is identical to that of previous one, but the OSL-1 record, OSL-2 record, OSL-3 record, and OSL-4 record and OSL-5 are recorded at 125 °C for the test dose (0.756 Gy) and TB-1 between 0°C -200°C during step 3.

SQ (Grain Size 63-53µm)

Step-1	400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour and quenched at RT	
Step-2	Beta 2.268Gy (D₀) @ Dose Rate	D₀
Step-3	Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C TD 0.756Gy Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C	Sequence Recorded OSL-1 at 125°C
Step-4	Beta 22.68Gy(D₁)	D₁
Step-3	Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C TD 0.756Gy Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C	Sequence Recorded OSL-2 at 125°C
Step-5	Beta 75.6Gy(D₂)	D₂
Step-3	Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C TD 0.756Gy Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C	Sequence Recorded OSL-3 at 125°C
Step-6	Beta 151.2Gy (D₃)	D₃
Step-3	Thermal Bleaching-1(0°C -200°C) Optical Bleaching for 40 sec at 125°C	Sequence

TD 0.756Gy

Thermal Bleaching-1(0°C -200°C)

Optical Bleaching for 40 sec at 125°C

Recorded OSL-4 at 125°C

Step-2 Beta 2.268Gy(D₀)

D₀

Step-3 Thermal Bleaching-1(0°C -200°C)

Optical Bleaching for 40 sec at 125°C

TD 0.756Gy

Thermal Bleaching-1(0°C -200°C)

Optical Bleaching for 40 sec at 125°C

Recorded OSL-5 at 125°C

It is observed that the 400°C annealed sample shows unusual shape of OSL decay with weaker OSL records. It has been achieved usual shape of OSL decay curve with significant growth in OSL records with changes in annealing temperature such as 600°C, 800°C and 1000°C annealed samples. (Fig.39,40,41,42)

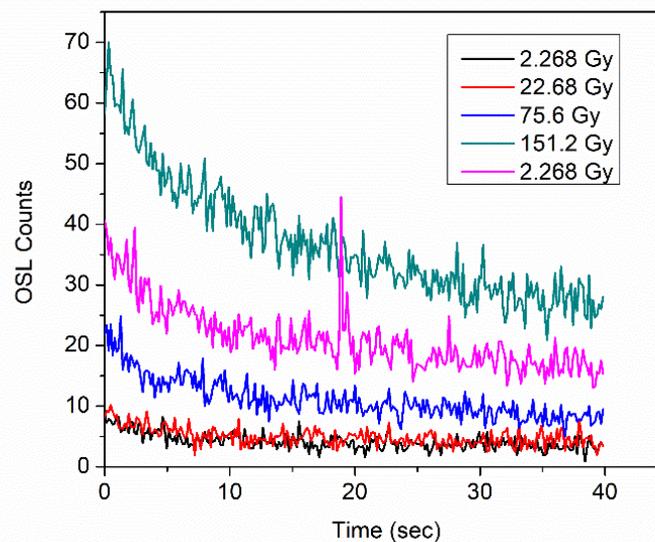


Fig. 4.39 OSL at 125°C for annealed sample of 400°C at different cyclic sequence of physical condition (a) D₀ +sequence (b) D₀+sequence+D₁ + sequence (c) D₀+sequence+D₁+ sequence+D₂ + sequence (d) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃ + sequence (e) D₀+sequence+D₁+ sequence+D₂+ sequence+D₃+ sequence+D₀ + sequence

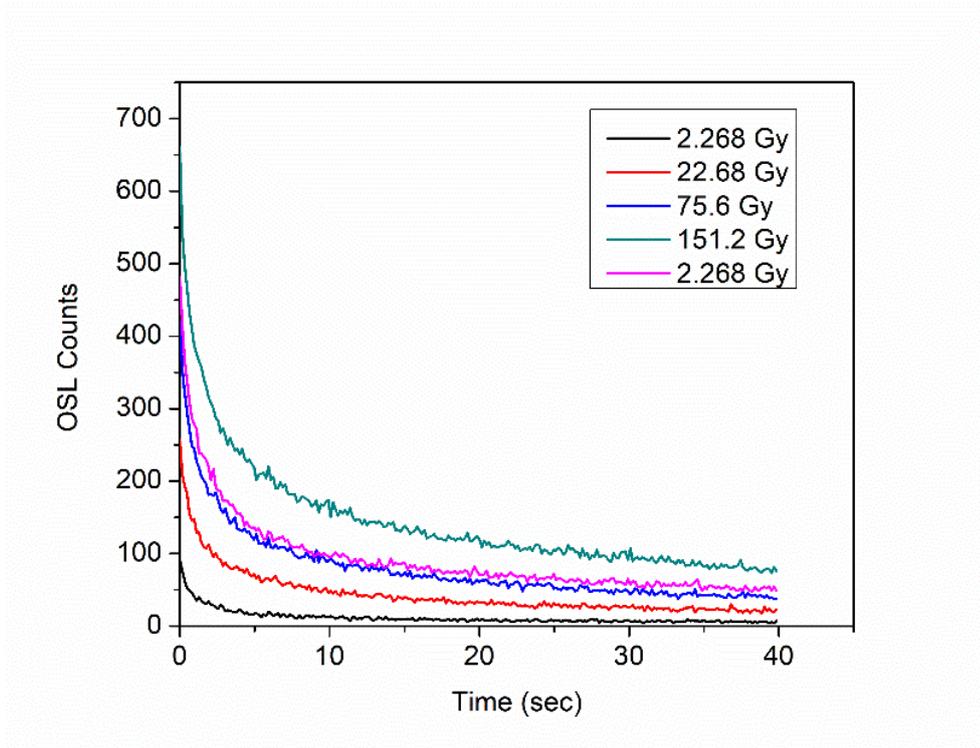


Fig. 4.40 OSL at 125°C for annealed sample of 600°C at different cyclic sequence of physical condition (a) D_0 + sequence (b) D_0 + sequence + D_1 + sequence (c) D_0 + sequence + D_1 + sequence + D_2 + sequence (d) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence (e) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence + D_0 + sequence

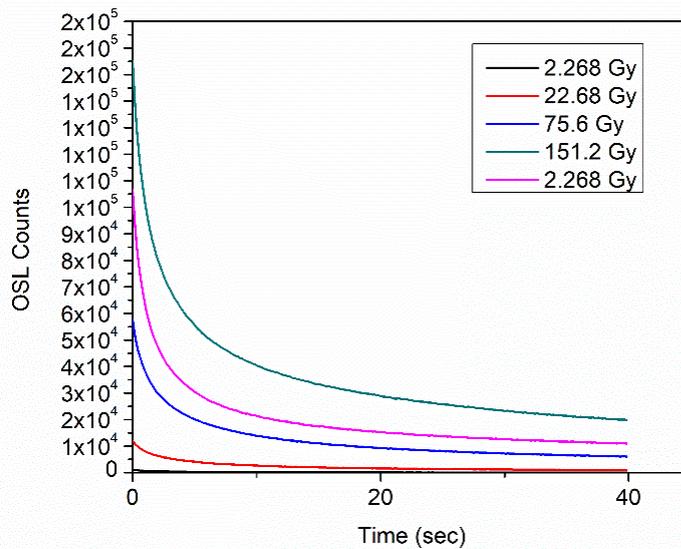


Fig. 4.41 OSL at 125°C for annealed sample of 800°C at different cyclic sequence of physical condition (a) D_0 + sequence (b) D_0 + sequence + D_1 + sequence (c) D_0 + sequence + D_1 + sequence + D_2 + sequence (d) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence (e) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence + D_0 + sequence

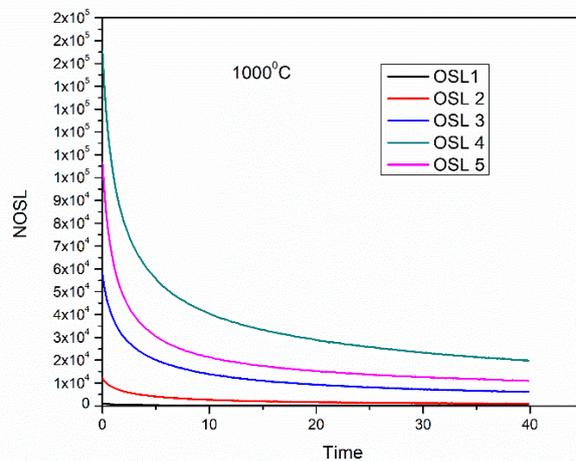


Fig. 4.42 OSL at 125°C for annealed sample of 1000°C at different cyclic sequence of physical condition (a) D_0 + sequence (b) D_0 + sequence + D_1 + sequence (c) D_0 + sequence + D_1 + sequence + D_2 + sequence (d) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence (e) D_0 + sequence + D_1 + sequence + D_2 + sequence + D_3 + sequence + D_0 + sequence

Murray A S et al³⁵ have suggested that while estimating an equivalent dose with OSL data using a single aliquot regeneration dose protocol should keep the test dose low compared to the natural dose to minimize the risk of thermal transfer of charge inserted into shallow, light insensitive traps by such test dose. It was believed that this could be transferred to the OSL trap during subsequent high temperature preheating. It was implicitly assumed that a small test dose always caused the same trapped charge population, so changes in luminescence response could be interpreted as changes in luminescence recombination probability.

Pagonis V et al³⁶ reported that although the fast component of the OSL signal in quartz has been found to be the basis for accurate luminescence dating, its use is limited by the saturation of the measured OSL signal. However, another OSL signal is proposed as the basis for the new OSL data, namely thermally transferred OSL (TT-OSL), which is measured after optical bleaching of the irradiated quartz and then preheat.

Bo Li et al³⁷ showed that thermal transfer effects play an important role in quartz optical dating. There are two types of thermal transfer, basic transfer, and recuperation. The basic transfer is a 'single transfer' effect, which results from the movement of charges from thermally stable, optically insensitive trap into optically sensitive traps during preheating. However, the recuperation, refers to a 'double transfer' effect, which is the combination of phototransfer during optical bleaching and thermal transfer during preheating.

For the present work, samples irradiated with different beta doses were subjected to TB-1 from 0°C-200°C. During this treatment, electrons might have thermally transferred from thermally stable, optically insensitive trap into the traps above 200°C. These traps might be thermally or optically sensitive traps. However, during the OB-1 at 125°C for 40 seconds, the electrons get depleted from optically insensitive traps and gets re-trapped into other optically sensitive traps or may re-trap into thermally sensitive traps as part of recuperation process. Further, under influence of test dose which always gave rise to the same trapped charge population followed by TB-1 from 0°C -200°C, might support to populate main OSL trap and again it may transfer the charges from thermally sensitive traps to OSL traps. This suggested process may correlate to the mechanism of TT-OSL which helps to achieve efficient usual OSL decay of synthetic quartz with their three components.

The outcome of OSL deconvolution suggests that, for each annealed sample subjected to given beta test dose and repetition of Step-3 gives major contribution of slow components and moderate contribution of medium components of OSL decay curve. However, these contribution pattern remains identical as changes in given annealing treatment to the specimen followed by beta doses and repetition of Step-3.

Analysis (400°C AQ; 1hr)										Components			
										F(%)	M (%)	S(%)	
D ₀	Sequence									OSL-1 Record at 125°C	1.97	1.53	96.5
D ₀	Sequence	D ₁	Sequence							OSL-2 Record at 125°C	9.5	9.5	81
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence					OSL-3 Record at 125°C	0.8	3.03	96.17
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence			OSL-4 Record at 125°C	4.9	47.5	47.6
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence	D ₀	Sequence	OSL-5 Record at 125°C	1.36	49.06	49.58
											4.29	15.39	80.32
Analysis (600°C; 1hr)										F(%)	M (%)	S(%)	
D ₀	Sequence									OSL-1 Record at 125°C	5.66	27.14	67.2
D ₀	Sequence	D ₁	Sequence							OSL-2 Record at 125°C	6.6	19.32	74.1
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence					OSL-3 Record at 125°C	2.57	17.65	79.78
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence			OSL-4 Record at 125°C	1.46	16.17	82.37
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence	D ₀	Sequence	OSL-5 Record at 125°C	2.82	23.34	73.84
											4.07	20.07	75.86
Analysis (800°C AQ; 1hr)										F(%)	M (%)	S(%)	
D ₀	Sequence									OSL-1 Record at 125°C	3.9	28.25	67.84
D ₀	Sequence	D ₁	Sequence							OSL-2 Record at 125°C	1.51	20	78.5
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence					OSL-3 Record at 125°C	8.5	23.85	67.65
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence			OSL-4 Record at 125°C	2.2	19.11	78.69
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence	D ₀	Sequence	OSL-5 Record at 125°C	3.58	23.13	73.29
											4.03	22.80	73.17
Analysis (1000°C AQ; 1hr)										F(%)	M (%)	S(%)	
D ₀	Sequence									OSL-1 Record at 125°C	6.76	48.84	44.41
D ₀	Sequence	D ₁	Sequence							OSL-2 Record at 125°C	3.75	22.85	73.4
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence					OSL-3 Record at 125°C	2.54	22.75	74.71
D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence			OSL-4 Record at 125°C	3.78	19.34	76.87

D ₀	Sequence	D ₁	Sequence	D ₂	Sequence	D ₃	Sequence	D ₀	Sequence	OSL-5 Record at 125°C	5.83	25.17	69
											4.21	28.45	67.35
D ₀ =2.268Gy		D ₁ =22.68Gy		D ₂ =75.6Gy		D ₃ =151.2Gy							
Sequence=TB-1 from 0°C -200°C + OB-1 at 125°C for 40sec + Test Dose 0.75Gy + TB-1 from 0°C -200°C + OB-1 at 125°C for 40sec													

Table. 4.2 Components of OSL decay under cyclic sequence of physical condition

4.3.4 Effect of thermal bleaching temperature and their cut-off duration on OSL decay

As reported in earlier, the thermal bleaching at desired temperature helps to erase the shallow/unwanted TL traps and transferred the charges to active optically sensitive traps which is responsible for better OSL. The influence of different thermal bleaching temperature and their cut-off duration on OSL decay are also being considered to establish the correlation between TL and OSL. The outcomes from these investigations could support to the discussion on earlier TL and OSL works. The 400°C annealed sample subjected to 75.4 Gy beta dose followed by different thermal bleaching temperature range like 0°C -200°C, 0°C -300°C and 0°C -400 °C for 10 seconds of cutoff duration. It is observed that as increase in the temperature range of thermal bleaching, the OSL intensity decreases from 136.93 counts to 4.83 counts even though usual shape of OSL decay. But the trend of OSL decay pattern is similar for 600°C, 800°C and 1000°C annealed samples with the rise in strength of OSL count. (Fig.4.43(A), 43(B))

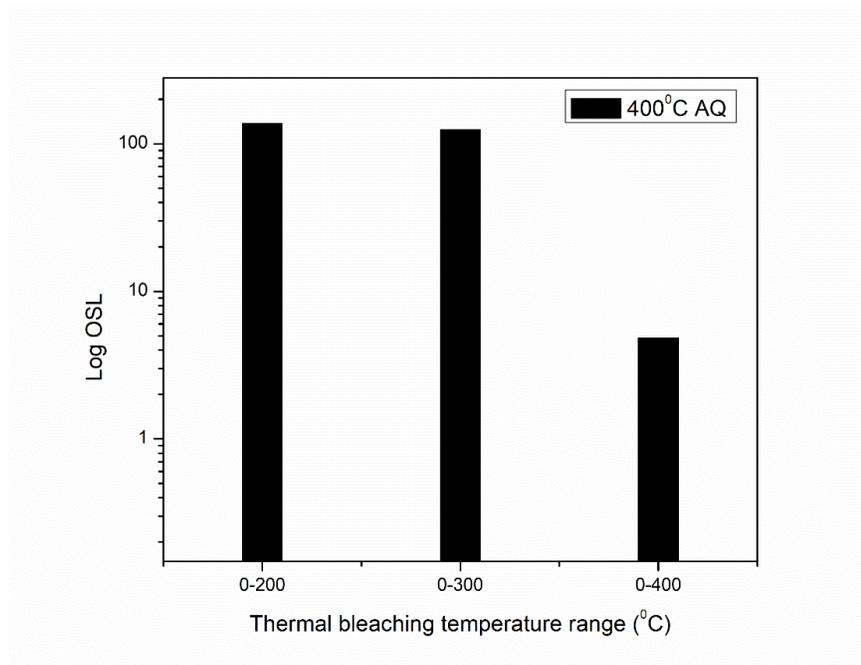


Fig. 4.43(A) Effect of thermal bleaching temperature on OSL Intensity

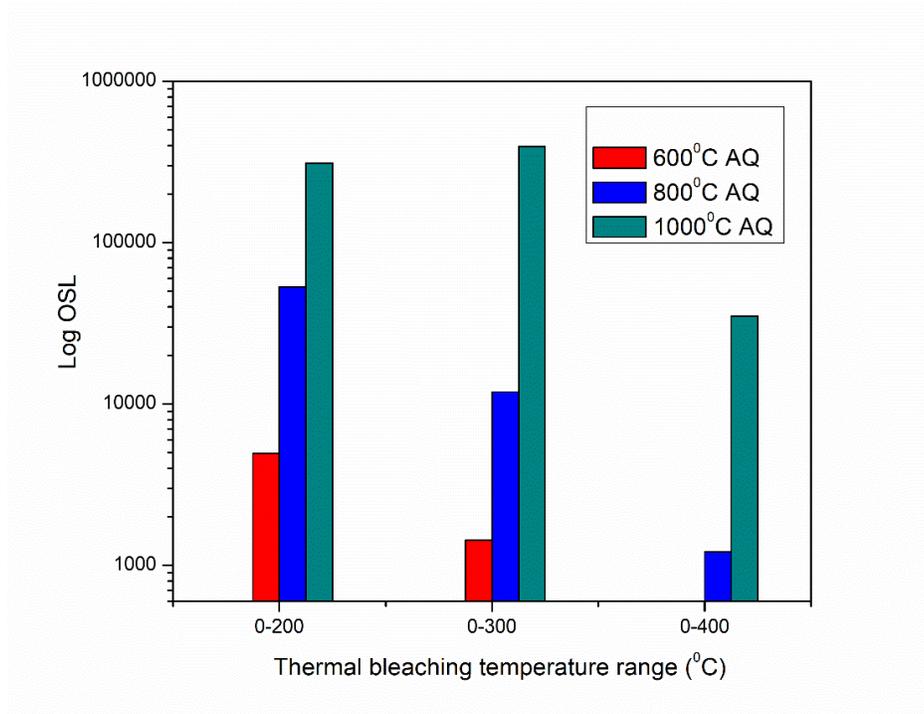


Fig. 4.43(B) Effect of thermal bleaching temperature on OSL Intensity

The charge transfer efficiency may depend on the population of the traps and nature of traps whether it is optically or thermally sensitive. However, as increase in the range of thermal bleaching temperature with its identical cutoff duration, the charges are erased from traps at least up to the thermal bleaching temperature and it might be completely depleted from light sensitive traps or shifted to other localities which are less light insensitive nature. It may be responsible for the loss of OSL event though it has sensitized by previously annealing treatment.

Apart from this, it is corroborated that as we increase the annealing temperature, at identical beta dose and range of thermal bleaching temperature and their cut-off duration, the OSL intensity increases. Therefore, it is reasonable to suggest that proposed range of thermal bleaching temperature is effective for the transfer of charges to main or active OSL trap. Hence it is clearly discernible that the slower component is predominant and medium components are moderate in OSL decay curve. (Fig. 4.44)

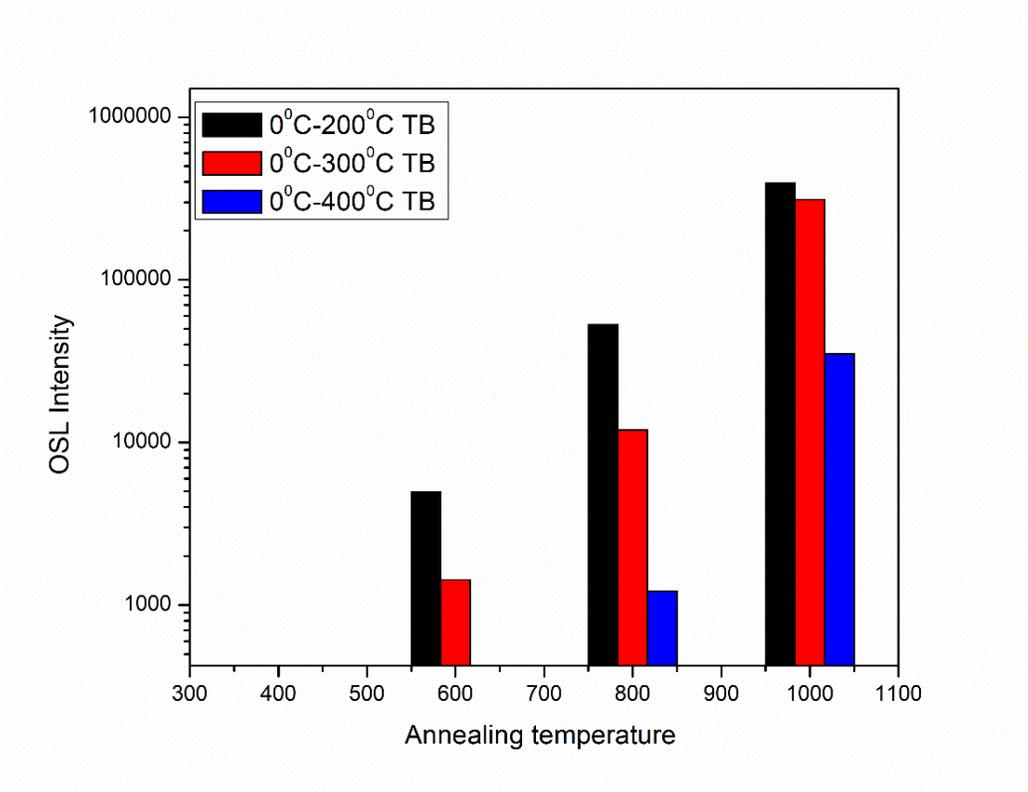


Fig. 4.44 Effect of annealing temperature on OSL Intensity at different thermal bleaching temperature

The OSL decay curves are recorded for different annealed samples followed 50.29 Gy beta dose and TB-1 from 0°C -200°C. It observed that OSL intensity increases from 72.43 counts to 1960767 counts with rise in annealing temperature. The contribution for this significant OSL growth is examined by resolving responsible components of OSL. It is reported that as rise in annealing temperature, the contribution of fast and medium components percentage decreases from 16.03% to 3.32%. This pattern of loss of medium components from 36.05% to 24.52 % is observed up to 800°C annealed sample and it enhances by further rise in annealing temperature to 1000°C. But, the slow component of OSL increases from 47.91% to 70.16 % with annealing temperature up to 800°C but it shows little decrease with further rise in annealing temperature. (Fig.4.45)

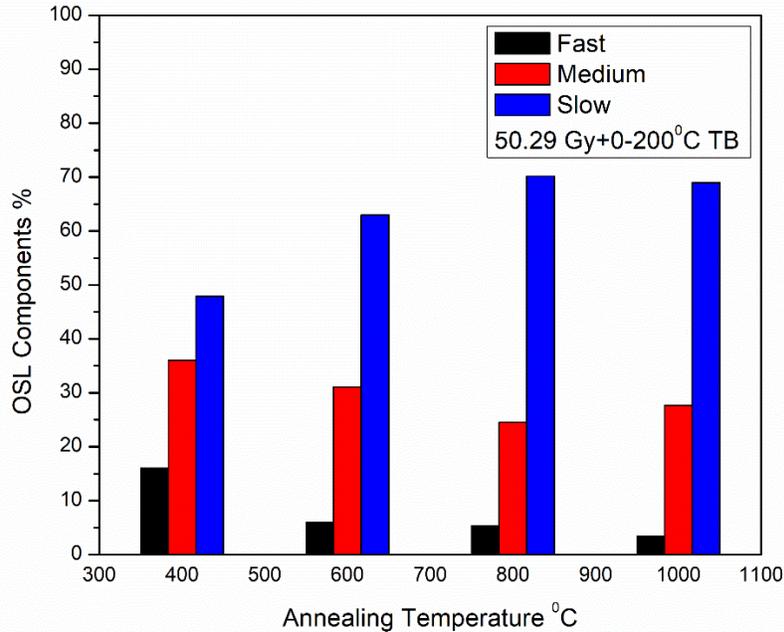


Fig. 4.45 Effect of annealing temperature on components of OSL decay curve at identical dose and thermal bleaching temperature

Further the TL glow curve are recorded for different annealed samples after the 50.29 Gy irradiated sample subjected to TB-1 from 0°C -200°C and OB-1 at 125°C for 40 seconds. It showed that as we increase the annealing temperature up to 800°C, the growth of broad TL glow peak around 290°C -300°C in accordance with deeper 380°C TL glow peak. The positions of the broad glow peak is shifted to 320°C TL glow peak in accordance with new contribution of 176°C, 228°C and 377°C TL glow peaks in 1000°C annealed sample. It explicitly justifies that higher temperature annealed sample supports reestablishment of the ~220°C TL glow peak associated with Ge center. This offered suggestion is in well agreement with the ESR study.

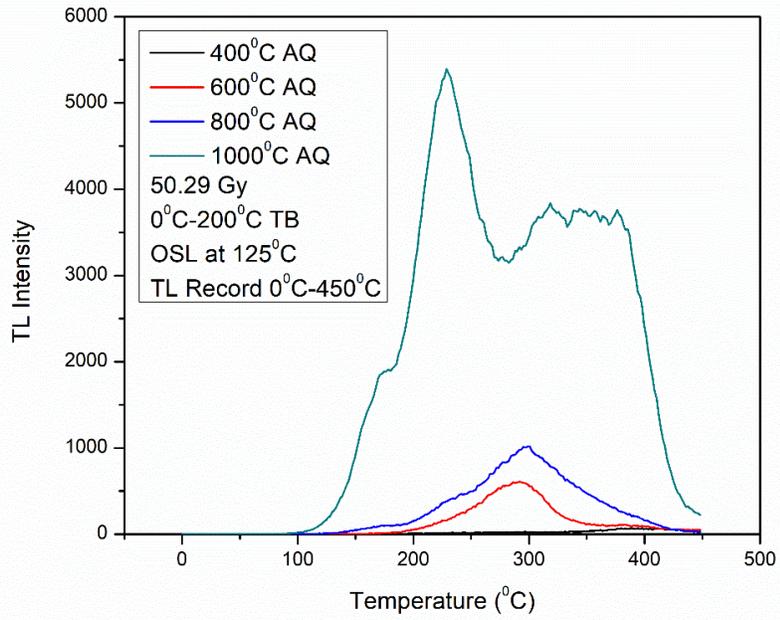


Fig 4.46 TL Recorded from 0°C to 450°C for different annealed temperature subjected to dose, TB, and Optical stimulation

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