

# **CHAPTER-6**

# **CONCLUSIONS**

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The present study carried out with the aim of achieving usual exponential decay curve of optically stimulated luminescence (OSL) from synthetic quartz. For achieving above objective various physical treatment was carried out on the sample of synthetic quartz. Various experimental protocol applied to synthetic quartz material to record TL and OSL from it. A TL and OSL correlation is established along with the thermal transfer effect on the shape of OSL decay curve is studied. Following conclusions are obtained after performing experimental study on synthetic quartz.

### **1) Thermoluminescence (TL) study of synthetic quartz**

#### **a) Effect of beta dose on TL glow curve of unannealed sample.**

Each irradiated sample shows the usual TL glow peak around 110 °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°C, 272°C, and 362°C with feeble intensity under higher beta irradiation of 22.68Gy and 158.76Gy.

#### **b) TL-Dose Response Curve (TL-DRC) of unannealed sample.**

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.

#### **c) Effect of annealing temperature on TL glow curve of sample.**

The lower annealed sample (say 400°C; 1hr) shows usual 110°C TL glow peak, which varies between 99°C -126°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 ~ 97 % of TL sensitization in sample.

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.

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.

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. It is observed 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.

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.

## **2) Effect of cycle of physical treatment on TL glow curve of annealed sample.**

It was found that the 400 °C annealed sample showed that the first TL 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.

After cyclic physical treatment TL 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. 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.

Discrete contribution of 110°C TL glow peak with significant TL intensity as a function beta doses is observed. 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.

TL Record of 400°C annealed sample record exhibits, the background signal. But, under different beta doses (Dn) followed by repetition of cyclic sequence, sample exhibits average TL glow peak around 21°C and 382°C and their TL intensity increase from 28 counts to 695 counts and 21 counts to 448 counts respectively.

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. 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. 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.

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.

### **3) Effect of repetition of cyclic sequence of physical condition on OSL decay.**

The 400°C annealed sample shows usual shape of OSL decay with 28 counts of OSL intensity in OSL record. It increases to 701 counts which is 25 times higher under the influence of different beta doses (Dn) followed by repetition of cyclic sequence. Other significant observation it is found that after returning to initial physical condition counts of the OSL record exhibited double (56 Counts) growth in OSL intensity compared to first OSL Record.

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. It is established clearly 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

It has been observed that 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°C to 212°C, 290°C to 295°C, 300°C, 330°C, 365°C, and glow at 386°C which is discernible while recording TL in measuring temperature range of 0°C -450°C. 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) OSL-Dose Response study under repetition of cyclic sequence of physical condition.**

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.

#### **5) Deconvolution study of OSL at 1250C under sequence of physical condition**

The outcome of OSL deconvolution reports that, for each annealed sample subjected to different beta doses and repetition of cyclic sequence 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 cyclic sequency.

It can be correlated with TL records that the increase in annealing temperature to the sample up to 800°C, the contributions of broad TL glow peaks around 290°C-300°C and

deeper 380°C TL glow peak are prominent. Whereas the proportion of TL glow peaks at 176°C, 228°C, 320°C, and 377°C is observed the annealed sample at 1000°C. In addition, the effect of these TL glow peaks appeared as a result of the cyclic repetition, annealing treatment. Therefore, it is reasonable to suggest that the centres corresponding to 228°C and 320°C are identical and thus may act as a mean component of OSL. The centre corresponding to the 380°C TL peak is associated with the slower components of the OSL which justifies that the higher annealed sample support restoration of the ~ 220°C TL glow peak which is associated with the Ge centre. This offered suggestion is also confirmed by the ESR study.

It is observed that the existence of deeper traps such as 362°C under higher beta irradiation of 22.68Gy and 158.76Gy, 365°C and 386°C with annealing treatment at 600°C, 800°C, and 1000°C followed by above referred beta doses. Also, deeper traps corresponding to 370°C is clearly observed under a cyclic sequence of physical treatment. 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. It is also observed that the fast component of the OSL decay curve has a cross-sectional area of larger 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.

Irradiated samples with different beta doses were subjected to thermal bleach from 0-200 °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 optical bleach, 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 thermal bleaching from 0-200 °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 cyclic sequence 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 cyclic sequence.

#### **6) Effect of thermal bleaching temperature and their cut-off duration on OSL decay**

The 400°C annealed sample subjected to 75.4 Gy beta dose followed by different thermal bleaching temperature range like 0-200°C, 0-300°C and 0°C -400 °C for 10 seconds of cut-off 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.

The charge transfer efficiency depends 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 cut- off 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 is 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.

Further the TL glow curve are recorded for different annealed samples after the 50.29 Gy irradiated sample subjected to TB-1 from 0-200°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.

### **7) ESR Study**

To understand and correlate the bleaching/charge transfer effects on the centre, the ESR spectra were recorded before and after the sun exposure of the specimens for thermally untreated as well as pre-heat treated at elevated temperatures. It was observed that the amplitude of E1' centre reduces remarkably from 6.1 to 4.6a.u after the sun exposure for thermally untreated, 1000 Gy dose specimens, but this centre disappears in annealed sample beyond 600°C, even before sun exposures

To resolve the Ge centre, the ESR was recorded for 5mW microwave power under similar experimental conditions. Such centre shows reduction in ESR signal for thermally untreated specimen, whereas significant enhancement is observed in these centres from 6.1 to 8.2 and 6.7 to 7.7a.u for 600°C and 1000°C annealed specimens before and after UV illumination respectively

The thermally untreated material shows saturation with a very low ESR signal compare to annealed material which is attributed to effect of pre-thermal treatment, such treatment is responsible for changes in OSL/TL properties of quartz specimen. From present investigation of ESR study it is concluded that the E1' and Ge centers play predominant role in the production of TL peaks at 110°C and 220°C. Bleaching study of ESR signal indicate that the reduction of E1' center and growth of Ge center are correlated with enhancement of 210°C-230°C TL peak in material after optical stimulation. Thus, ESR spectra strongly correlate with TL glow curve pattern and OSL properties of synthetic quartz.