Synopsis of the thesis entitled

THERMAL TRANSFER EFFECT ON OSLCURVE OF SYNTHETIC QUARTZ

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INTRODUCTION

Quartz or Silicon Dioxide (SiO_2), the most common constituent of the earth, is known as α-quartz or natural quartz crystal. Due to its well-known piezoelectric properties, it is used for the production of electronic frequency control device as a “resonator” and “filters” in electronic circuits. Apart from these applications, researchers have used this material for ionizing radiation measurement device as a radiation dosimeter and age determination of geological/archaeological sample as a luminescence dating tool. **[1][2]**. Quartz being a natural material, it contains various twins and imperfections which are responsible to degrade its electronic properties and limit its use in applications. To avoid these problems, researchers have grown synthetic quartz crystal by hydrothermal technique at laboratory level.**[1]** The growth condition of the sample keeps the level of impurities, twins and other imperfections at optimum level. Natural quartz shows complex luminescence signals either in Thermo luminescence (TL) or Optically Stimulated Luminescence (OSL) measurement. However, such laboratory grown quartz crystals minimize the complexity in luminescence signals followed by enhanced quality in luminescence outcomes.

As a luminescence aspect, literature shows the various physical treatment to the samples like ionizing radiations, optical bleaching wavelength, annealing temperature, duration of annealing temperature, TL heating rates and grain size are responsible for the noticeable changes of TL glow curve pattern.**[3][4]**. It has been observed from the recent works that, during the measurement the effect of thermal quenching reduces the concentration of deep TL traps and hence enhances the non-radiative transition which gives losses in TL efficiency. However, this problem can be studied and addressed with the help of OSL. The OSL technique has potential advantages over TL technique, particularly in geological dating and radiation dosimetry.

In OSL mechanism, the signal arises due to the release of trapped electrons from previously irradiated crystal under the influence of optical stimulation which recombines with hole at recombination center. The OSL outcome is directly proportional to concentration of trapped electrons which have been influenced by the strength of physical treatment to the sample. Generally, the optically released electron from trap follows the usual path in which it recombines with the hole via conduction band and hence gives usual(exponential) shape of OSL decay curve displayed as the OSL intensity verses time scale. **[5]** Researchers have attempted to segregate and resolve the usual shape OSL decay as multi components of OSL decay curves as fast, medium and slow characteristics of decay. These components find the correlation between OSL and usual TL glow peak.

As TL glow curves get influenced by physical treatments to the sample, the OSL sensitivity and shape of OSL decay also shows influence by such physical treatments. **[6]** During optical stimulation at room temperature, the quartz material exhibits unusual shape of OSL decay curves followed by weaker OSL counts. It is attributed to re-trapping of electrons by shallow traps corresponding to 110^{o}C TL glow peak, which is also suggested due to optically released electrons which do not follow the conventional path to recombine with hole at recombination center.**[7]** However, Kale et al **[8]** have reported that usual shape of OSL decay with significant OSL output is possible at optical stimulation near room temperature with critical level of physical conditions in synthetic quartz sample. But, below these critical level of physical conditions, identical sample showed unusual shape of OSL decay curve and hence the components of OSL are not resolvable.

To avoid these problems, researchers have suggested thermal assistance OSL(TA-OSL) technique **[9]** and thermal transferred OSL (TT-OSL) technique **[10]** to enhance OSL output with usual shape of decay curve. In TA-OSL technique, the optical stimulation around 160^{o}C temperature is to restrict the re-trapping of optically released electron by shallow traps and assist for better OSL. It has been reported that OSL at 160^{o}C exhibits unusual shape of OSL decay curve even below critical level of physical conditions to synthetic quartz material which suggests that re-trapping of optically released electrons still exist **[11].** In TT-OSL technique, the thermal transfer process helps to erase unwanted TL traps which are active in re-trapping process which is responsible for transfer of electrons from optically insensitive trap to optically sensitive trap. **[10]**

G Adamiec et al have reported two mechanisms for the production of the TT-OSL signal in quartz when all stimulation is carried out at 125 °C. One mechanism is based on the double transfer process previously put forward for OSL recuperation following storage at room temperature. In this mechanism, electrons released by optical stimulation from the trap give rise to the fast OSL component. However, some of them are transferred into a refuge trap (110^{o}C TL) via the conduction band; these electrons are then released from the refuge trap by a thermal treatment and some are re-trapped in the trap responsible for the fast OSL component. The other proposed mechanism is based on a single transfer process in which, electrons are transferred by thermal treatment from a light-sensitive trap via the conduction band to the trap that gives rise to the fast component of the OSL signal, this trap having been emptied by the initial optical stimulation. The analysis of the measured OSL and TT-OSL decay curves suggest that the two signals are derived from the same traps and are dominated by the fast OSL component.**[12]**

AIM OF THE WORK

Since components of OSL curve are not resolvable below critical physical conditions applied to synthetic quartz even though optical stimulation had been carried out at elevated temperature. Present work is aimed to achieve usual shape of OSL curve and hence to resolve its components due to its potential applications. Study was also carried out to set physical conditions in which thermal transfer effect on OSL curve is examined by following various protocols. The OSL outcomes of the present work are discussed in the direction of (i) shape of OSL decay curve (ii) OSL sensitivity and (iii) responsible component of OSL decay curve and their correlation with TL mechanism.

The synthetic quartz material of 63-53µm grain was selected and it had been given various physical treatments like annealing treatment, ionizing radiation doses, thermal bleaching at desired temperature and their cut-off duration, optical bleaching and usual test dose. Such physically treated sample was optically stimulated at 125°C for 40 seconds by 470nm light.

Before investigating the present work on OSL, it is essential to have careful examination of TL study of synthetic quartz material under the influence of sequence of physical treatment. The TL outcomes have provided the details about influence of shallow or unstable TL traps, optically as well as thermally sensitive traps over TL measurement temperature, thermal stability of glow peaks and their TL dose response curve (TL-DRC). The details of experimental protocol are given in flow chart and their outcome are studied as under:

[1]TL recorded over 0°C-450°C for unannealed sample followed by different beta doses.

Synthetic Quartz (Grain Size 63-53µm)	
Unannealed Sample	
Beta Irradiation @Dose Rate	
2.268Gy	Recorded TL-1 (0°C-450°C) @ heating rate 5°C/sec
22.68Gy	Recorded TL-2 (0°C-450°C) @heating rate5°C/sec
158.76Gy	Recorded TL-3 (0°C-450°C) @heating rate5°C/sec

- The sensitivity of 110°C TL glow peak increases with beta doses by 2.268Gy, 22.68Gy and 158.76Gy
- The 110°C TL glow peak shows sublinear nature of TL dose response curve (DRC).
- The higher temperature TL glow peaks appear around 190°C, 273°C and 362°C under identical doses.

[2] TL recorded over 0°C-450°C for annealed samples followed by different beta doses

Synthetic Quartz (Grain Size 63-53µm)	
Annealing Treatments 400°C AQ, 600°C, 800°C, 1000°C AQ; 1hr	
Beta Irradiation @ Dose Rate	
2.268Gy	Recorded TL-1 (0°C-450°C) @ heating rate 5°C/sec
22.68Gy	Recorded TL-2 (0°C-450°C) @ heating rate 5°C/sec
158.76Gy	Recorded TL-3 (0°C-50°C) @ heating rate 5°C/sec

- Each treated sample continues to exhibit usual 110°C TL glow peak. The TL sensitivity of this glow peak increases with rise in annealing temperature.

- The 400°C annealed sample exhibits sub linear TL-DRC nature of 110°C TL glow peak. But it has changed to super linear TL-DRC nature for higher annealed samples.
- Several new TL glow peaks are observed around 194°C and 293°C, 300°C and 207°C for 600°C, 800°C and 1000°C treated samples respectively. The suggested TL glow peaks exhibit noticeable thermal stability and superlinear TL-DRC behaviour.
- The annealing treatments enhance about 95% TL sensitivity of 110°C glow peak compared to unannealed sample.

[3][A] TL recorded over 0°C-200°C for annealed samples followed by cycle of physical conditions and different beta doses.

	Synthetic Quartz (Grain Size 63-53µm)	
Step-1	Annealing treatment 400°C, 600°C, 800°C, 1000°C; 1hour	
Step-2	Beta 2.268Gy (Do)@Dose Rate	Do
Step-3	Recorded TL-1(0-200°C) @heating rate @5°C/sec	TL-1(0-200°C)
	Thermal Bleaching (0-450°C)	C1
	Test Dose 0.756Gy	
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-4	Beta 22.68Gy(D1)	D1
Step-5	Recorded TL-2(0-200°C) @heating rate @5°C/sec	TL-2(0-200°C)
	Thermal Bleaching (0-450°C)	C2
	Test Dose 0.756Gy	
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-6	Beta 75.6Gy(D2)	D2
Step-7	Recorded TL-3(0-200°C) @heating rate @5°C/sec	TL-3(0-200°C)
	Thermal Bleaching (0-450°C)	C3
	Test Dose 0.756Gy	
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-8	Beta 151.2Gy(D3)	D3
Step-9	Recorded TL-4(0-200°C) @heating rate @5°C/sec	TL-4(0-200°C)
	Thermal Bleaching (0-450°C)	C4
	Test Dose 0.756Gy	
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-10	Beta 2.268Gy (Do)	Do
Step-11	Recorded TL-5(0-200°C) @heating rate @5°C/sec	TL-5(0-200°C)

- Each annealed sample either exposed by 2.268Gy beta dose or influenced by cycle C1 followed by Step-4 to Step-5, cycle C2 followed by Step-6 to Step-7, cycle C3 followed by Step-8 to Step-9 and cycle C4 followed by Step-10 exhibits only 110°C TL glow peak in TL readout at Step-3 and Step-11. The sensitivity of 110°C TL glow peak increase from 115.8 counts (TL readout of Step-3) to 4279.66 counts (TL readout of Step-11).
- The influence of C1, C2 and C3 cycles of physical conditions followed by 22.68Gy, 75.6Gy and 151.2Gy beta doses on TL readout of Step-3 enhances the strength in TL

intensity of 110°C glow peak by 2020 counts, 5995.93 counts and 10537 counts respectively.

- The influence of different annealing temperature on 110°C glow peak followed by cycle C1 followed by Step-4-5, cycle C2 followed by Step-6-7 and cycle C3 followed by Step-8-9 show systematic growth in TL counts of 110°C glow peak.
- The repetitions of cycle of physical conditions followed by 22.68Gy, 75.6Gy and 151.2Gy beta doses shows superlinear TL-DRC nature of 110°C TL glow peak for the 1000°C annealed sample.

[3][B] TL recorded over 0°C-450°C for annealed sample followed by cycle of physical conditions and different beta doses.

	Synthetic Quartz (Grain Size 63-53µm)	
Step-1	Annealing treatment 400°C, 600°C, 800°C, 1000°C; 1hour	
Step-2	Beta 2.268Gy (Do)	Do
Step-3	Thermal Bleaching (0-200°C)	TB(0-200°C)
Step-4	Recorded TL-1 (0-450°C) @heating rate @5°C/sec	TL-1 (0-450°C)
	Test Dose 0.756Gy	C1
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-5	Beta 22.68Gy(D1)	D1
Step-6	Thermal Bleaching(0-200°C)	TB(0-200°C)
Step-7	Recorded TL-2 (0-450°C) @heating rate @5oC/sec	TL-2 (0-450°C)
	Test Dose 0.756Gy	C2
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-8	Beta 75.6Gy(D2)	D2
Step-9	Thermal Bleaching(0-200°C)	TB(0-200°C)
Step-10	Recorded TL-2 (0-450oC) @heating rate @5oC/sec	TL-3 (0-450°C)
	Test Dose 0.756Gy	C3
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-11	Beta 151.2Gy(D1)	D3
Step-12	Thermal Bleaching(0-200°C)	TB(0-200°C)
Step-13	Recorded TL-2 (0-450°C) @heating rate @5oC/sec	TL-4 (0-450°C)
	Test Dose 0.756Gy	C4
	Thermal Bleaching (0-200°C)	
	Thermal Bleaching (0-450°C)	
Step-14	Beta 2.268Gy (Do)	Do
Step-15	Thermal Bleaching(0-200°C)	TB(0-200°C)
Step-16	Recorded TL-2 (0-450°C) @heating rate @5oC/sec	TL-4 (0-450°C)

- Individual annealed sample either exposed to 2.268Gy beta dose followed by Step-3-4 or influenced by C1 followed by Step-5 to Step-7, C2 followed by Step-8 to Step-10, C3 followed by Step-11 to Step-13 and cycle C4 followed by Step-14 to Step-16 exhibits various TL glow peaks around 207°C-219°C, 290°C,302°C, 367°C and 380°C along with the elimination of 110°C TL glow peak.

- As increase in annealing temperature followed by cycle of physical conditions shows strength and growth in TL sensitivity of suggested TL glow peak.
- The suggested TL glow peaks around 207°C-219°C, 290°C, 302°C, 367°C and 380°C exhibits super linear of TL-DRC nature.

[4] OSL recorded at 125°C for annealed samples followed cycle of physical conditions and beta doses.

	Synthetic Quartz (Grain Size 63-53μm)	
Step-1	400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour	
Step-2	Beta 2.268Gy (Do)	Do
Step-3	TB(0-200°C)	TB(0-200°C)
Step-4	Recorded OSL-1 at 125°C	Recorded OSL-1 at 125°C
	TD 0.756Gy	C1
	TB(0-200°C)	
	OB at 125°C for 40seconds	
Step-5	Beta 22.68Gy	D1
Step-6	TB(0-200°C)	TB(0-200°C)
Step-7	Recorded OSL-2 at 125°C	Recorded OSL-2 at 125°C
	TD 0.756Gy	C2
	TB(0-200°C)	
	OB at 125°C for 40seconds	
Step-8	Beta 75.6Gy	D2
Step-9	TB(0-200°C)	TB(0-200°C)
Step-10	Recorded OSL-3 at 125°C	Recorded OSL-3 at 125°C
	TD 0.756Gy	C3
	TB(0-200°C)	
	OB at 125°C for 40seconds	
Step-11	Beta 151.2Gy	D3
Step-12	TB(0-200°C)	TB(0-200°C)
Step-13	Recorded OSL-4 at 125°C	Recorded OSL-4 at 125°C
	TD 0.756Gy	C4
	TE-1(0-200°C)	
	OB at 125°C for 40seconds	
Step-14	Beta 2.268Gy	Do
Step-15	TB(0-200°C)	TB(0-200°C)
Step-16	Recorded OSL-5 at 125°C	Recorded OSL-5 at 125°C

- Each annealed sample exposed by 2.268Gy beta dose followed by thermal bleaching(0°C-200°C) shows usual shape of OSL decay at 125°C. (Step-4).
- The OSL intensity significant arise in OSL readout of Step-16 under influence of cycle C1 followed by Step-5 to Step-7, cycle C2 followed by Step-8 to Step-10, cycle C3 followed by Step-11 to Step-13 and cycle C4 followed by Step-14 to Step-16 compared to OSL readout of Step-4.
- The OSL counts at 125°C increases with noticeable counts under cycle of C1 followed by Step-5 to Step-7, cycle C2 followed by Step-8 to Step-10, cycle C3 followed by Step-11 to Step-13 compared to the OSL counts at 125°C followed by Step-4 readout. The usual

shape of OSL decay under these protocol exhibits predominant contribution of medium and slow components of OSL decy curve

- The influence of different annealing temperature on OSL readout of Set-4 followed by cycles of physical conditions and Step-5 to Step-7 shows significant OSL count. Further, it increases by following Step-8 and Step-11.

- The 400°C and 600°C annealed sample followed by cycles of physical conditions exhibit sublinear OSL-DRC nature. However, it has changed to super linear OSL-DRC nature for 800°C and 1000°C annealed samples.

[5][A] TL recorded over 0°C-200°C, 0°C-300°C and 0°C-400°C measurement temperature for different annealed samples followed by 75.4Gy beta dose.

Synthetic Quartz (Grain Size 63-53µm)		
400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour		
Beta Irradiation @ Dose Rate		
75.4Gy		
Recorded TL-1(0°C-200°C) @ heating rate 5°C/sec	Recorded TL-2(0°C-300°C) @ heating rate 5°C/sec	Recorded TL-3(0°C-400°C) @ heating rate 5°C/sec

- Either increase in the range of TL measurements temperature (0°C-200°C, 0°C-300°C and 0°C-400°C) for individual annealed sample followed by 75.4Gy beta does or increase in the annealing temperature followed by 75.4Gy beta does and each TL measurement temperature shows contribution of usual 110°C TL glow peak with significant TL sensitivity.
- The contribution of 200°C TL glow peak with significant TL sensitivity is observed in each annealed sample followed by 75.4Gy beta does and at higher thermal bleaching temperature.

[5][B] TL recorded over 0°C-450°C for different annealed samples followed by 75.4Gy beta dose and different thermal bleaching temperature with their cut-off duration of 10seconds.

Synthetic Quartz (Grain Size 63-53µm)		
400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour		
Beta Irradiation @ Dose Rate		
75.4Gy		
Thermal Bleaching(0°C-200°C)	Thermal Bleaching(0°C-300°C)	Thermal Bleaching(0°C-400°C)
cut-off at 200°C for duration of 10seconds	cut-off at 200°C for duration of 10seconds	cut-off at 200°C for duration of 10seconds
Recorded TL-1(0°C-450°C) @ heating rate 5°C/sec	Recorded TL-2(0°C-450°C) @ heating rate 5°C/sec	Recorded TL-3(0°C-450°C) @ heating rate 5°C/sec

- As increase in annealing temperature followed by 75.4Gy beta dose for thermal bleaching up to 200°C with cut off duration of 10seconds shows the noticeable sensitization between 290°C-300°C TL glow peak over 0°C-450°C TL measurement temperature.

[6] OSL recorded at 125°C for different annealed samples followed by 74.4Gy beta dose

and different thermal bleaching temperature of 200°C, 300°C, 400°C with their cut-off duration of 10seconds.

Synthetic Quartz (Grain Size 63-53μm)		
400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour		
Beta Irradiation @ Dose Rate		
75.4Gy		
Thermal Bleaching(0°C-200°C)	Thermal Bleaching(0°C-300°C)	Thermal Bleaching(0°C-400°C)
cut-off at 200°C for duration of 10seconds	cut-off at 300°C for duration of 10seconds	cut-off at 400°C for duration of 10seconds
Recorded OSL-1 at 125°C for 40seconds	Recorded OSL-2 at 125°C for 40seconds	Recorded OSL-3 at 125°C for 40seconds

- Each treated sample thermally bleached up to 200°C or 300°C or 400°C with their cut-off duration of 10second exhibits usual shape of OSL decay curve. However, it shows losses in OSL counts with rise in thermal bleaching temperature with their identical cut-off duration of 10second. But, the strength in OSL counts remains superior in 1000°C annealed sample than lower annealed sample.
- The contributions of medium components of OSL found moderate and contribution of slow components found predominant in each annealed sample. While, the contribution of fast component of OSL decay is predominant in 1000°C annealed sample only.
- As rise in annealing temperature at distinct thermal bleaching temperature with their cut-off duration of 10second, exhibits the significant growth in OSL counts. However, the strength in OSL counts is found superior in thermal bleaching temperature of 200°C and 300°C with their cut-off duration of 10second. It exhibits major contribution of slow component of OSL compared to fast and medium component of OSL.

[7]TL recorded over 0°C-450°C for different annealed samples followed by 50.29Gybeta dose, thermal bleaching of 200°C and optical bleaching at 125°C

Synthetic Quartz (Grain Size 63-53μm)		
400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour		
Beta Irradiation @ Dose Rate		
50.29Gy		
Thermal Bleaching(0°C-200°C)	Thermal Bleaching(0°C-200°C)	Thermal Bleaching(0°C-200°C)
Optical Bleaching at 125°C for 40seconds	Optical Bleaching at 125°C for 40seconds	Optical Bleaching at 125°C for 40seconds
Recorded TL-1(0°C-450°C) @ heating rate 5°C/sec	Recorded TL-2(0°C-450°C) @ heating rate 5°C/sec	Recorded TL-3(0°C-450°C) @ heating rate 5°C/sec

- The contribution of broad TL glow peak is observed between 290°C-320°C with increase in annealing temperature.

- Under influence of different annealing treatments, the TL intensity of this glow peak increases from 27.7 counts to 3793.44 counts which is about 95% of growth in TL sensitivity.

Above results were also cross examined with TG, DTG and TGA study with an identical physical treatment on the specimen. The results were corroborated with XRD and ESR studies as well to confirm suggested revelations.

Plan of the thesis:

Research work of the thesis is presented in the form of five chapters.

Chapter-1: It describes literature survey, scope and objective of the present work

Chapter-2: It explains the details about sample preparation, physical treatment to the sample (suggested protocols) and experimental setup.

Chapter-3: This chapter presents the results from TL and OSL measurements under different physical treatments/ protocols.

Chapter-4: Presents allied studies in the support of TL and OSL outcomes.

Chapter-5: Conclusions

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