

ABSTRACT

**THERMAL TRANSFER EFFECT ON SHAPE OF OSL
CURVE OF SYNTHETIC QUARTZ**

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By

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Quartz or Silicon Dioxide (SiO_2), is technologically very important material. 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.

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.

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°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.

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°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. Present 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. The details of experimental protocol are given in flow chart and their outcomes are studied as under:

1) Effect of beta dose on TL glow curve of unannealed sample.

SQ (grain size 63-53μm)	
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.

2) Effect of annealing temperature on TL glow curve of sample.

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 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

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.

3) Effect of cyclic physical treatment on TL glow curve of annealed sample.

	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	Seque	TL-1 Record from 0$^{\circ}$C -
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	nce	200$^{\circ}$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 22.68 Gy(D₁)	D₁	
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Seque	TL-2 Record from 0$^{\circ}$C -
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	nce	200$^{\circ}$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 75.6 Gy(D₂)	D₂	
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Seque	TL-3 Record from 0$^{\circ}$C -
	TB-2 from 0$^{\circ}$C -450$^{\circ}$C	nce	200$^{\circ}$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 151.2 Gy(D₃)	D₃	
Step-3	TB-1 from 0$^{\circ}$C -200$^{\circ}$C	Seque	TL-4 Record from 0$^{\circ}$C -
		nce	200$^{\circ}$C

	TB-2 from 0°C -450°C	
	Test Dose (TD)0.756 Gy	
	TB-1 from 0°C -200°C	
	TB-2 from 0°C -450°C	
Step-2	Beta 2.268 Gy (D₀)	D₀
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.
- **4) TL from 0°C -450°C for annealed sampled at different cyclic sequence of physical condition**

	SQ (Grain Size 63-53µm)
Step-1	Annealing; 400°C, 600°C, 800°C,1000°C@1hr and quenched at RT

Step-2	Beta 2.268 Gy (D₀)	D₀	
Step-3	TB-1 from 0°C -200°C	Sequence	
	TB-2 from 0°C -450°C		TL-1 Record from 0°C - 450°C
	Test Dose (TD)0.756 Gy		
	TB-1 from 0°C -200°C		
	TB-2 from 0°C -450°C		
Step-4	Beta 22.68Gy(D₁)	D₁	
Step-3	TB-1 from 0°C -200°C	Sequence	
	TB-2 from 0°C -450°C		TL-2 Record from 0°C - 450°C
	Test Dose (TD)0.756 Gy		
	TB-1 from 0°C -200°C		
	TB-2 from 0°C -450°C		
Step-5	Beta 75.6Gy(D₂)	D₂	
Step-3	TB-1 from 0°C -200°C	Sequence	
	TB-2 from 0°C -450°C		TL-3 Record from 0°C - 450°C
	Test Dose (TD)0.756 Gy		
	TB-1 from 0°C -200°C		
	TB-2 from 0°C -450°C		
Step-6	Beta 151.2Gy(D₃)	D₃	
Step-3	TB-1 from 0°C -200°C	Sequence	
	TB-2 from 0°C -450°C		TL-4 Record from 0°C - 450°C
	Test Dose (TD)0.756 Gy		

	TB-1 from 0°C -200°C	
	TB-2 from 0°C -450°C	
Step-2	Beta 2.268Gy (D₀)	Do
Step-7	TB-1 from 0°C -200°C	
Step-8	TB-2 from 0°C -450°C	TL-5 Record from 0°C - 450°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.

5) Effect of repetition of cyclic sequence of physical condition on OSL decay.

	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)	Do
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	

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(D_1) in Step-4, 75.6 Gy(D_2) in Step-5, 151.2 Gy(D_3) in Step-6 and then returned back to Step-2, where again sample was irradiated by 2.268 Gy beta dose as D_0 .

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.

6) Deconvolution study of OSL at 125 oC under sequence of physical condition

SQ (Grain Size 63-53 μ m)		
Step-1	400°C, 600°C, 800°C and 1000°C Annealed @ 1 hour and quenched at RT	
Step-2	Beta 2.268Gy (D_0) @ Dose Rate	D_0
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(D_1) Thermal Bleaching-1(0°C -200°C)	D_1 Sequence

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

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