



THERMOLUMINESCENCE STUDY FOR DIFFERENT GRAIN SIZES OF ANNEALED SYNTHETIC QUARTZ MATERIAL

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Keywords: synthetic quartz, grain size, thermal treatment, beta dose, thermally stimulated luminescence

Abstract

The thermally stimulated luminescence (TSL) glow curves have been recorded at room temperature for different grain sizes of annealed synthetic quartz material followed by beta irradiations. The significant shifting of higher temperature TL peak is observed from 357°C to 245°C with decrease in particle size from 125-106µm to 0.422µm of 600°C annealed sample. It is still appearing up to 45 µm in higher annealed sample of 1000°C material. Below 45µm of grain size, the higher temperature TL peak is disappeared. The TL peak of 110°C still exist both annealed samples. The TL intensity of 110°C and higher temperature peak are increased with decrease in particle size up to 45µm. It is significantly decreased by further reduced in grain size to 0.42µm.

1. Introduction

Researchers have reported that the TL intensity depend upon beta dose, annealing treatment, uv-illumination and particle size [1].

In present paper the TL intensity is recorded to different grain sizes of annealed synthetic quartz. The noticeable changes are observed either in TL intensity or peak position by further decrease in particle size from 45µm to 0.422µm.

2. Experimental Details

Grinding:

- (i) With Mortar and Pestle and prepared the grain size of 125-45µm
- (ii) With Ball Milling (Planetary Mono Mill, Pulverisette-6, Fritsch, Germany) technique and prepared the grain size of 0.422µm; MBR(Material-Ball-Ratio) was 1:8, RPM-350; for 4hrs

Treatment:

The sample of 0.422µm

↓
Dissolve in HCL+ Washed with distilled water

↓
Filtered by paper and dried at 40°C.

↓
Sample was annealed at 600 and at 1000° for 1hour duration.

Instrument: Nucleonix PC based TL reader

3. Results and Discussion

The 600°C annealed sample shows the growth in TL intensity with decrease in particle size from 125µm to 45µm for 110°C and 350°C TL peaks. It is decreased with particle size to 0.422µm. The 350°C peak is shifted to 250°C with decrease in particle size by keeping an identical position of 110°C TL peak. The 1000°C exhibits an identical pattern of TL growth curve to 600°C. The strength in TL signal is quite noticeable rather than 600°C. The shifting of higher temperature TL peak is observed with particle size from 125µm to 45µm. It is disappeared by further decrease in sample to 0.422µm of grain size **Fig1-5**

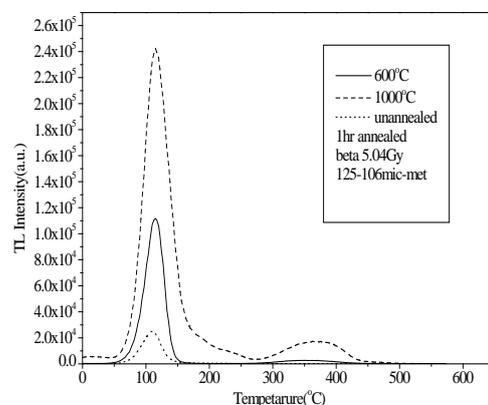


Fig.1 TL glow curve measured at room temperature for annealed and unannealed sample of 125-106mic-met.

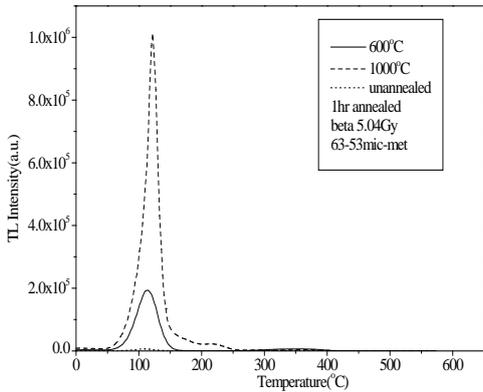


Fig.2 TL glow curve measured at room temperature for 63-53mic-met of annealed and unannealed samples.

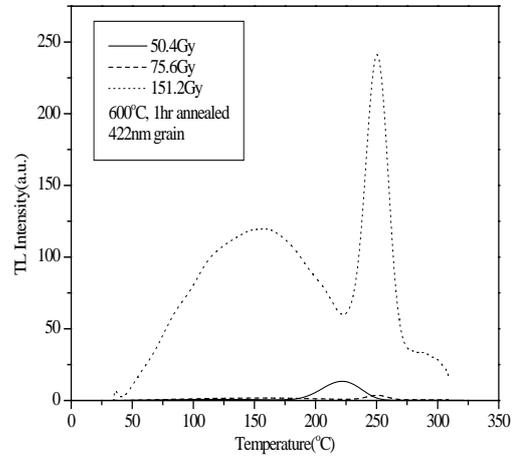


Fig.4 TL recorded at room temperature for 0.422mic-met grain of 600°C annealed sample

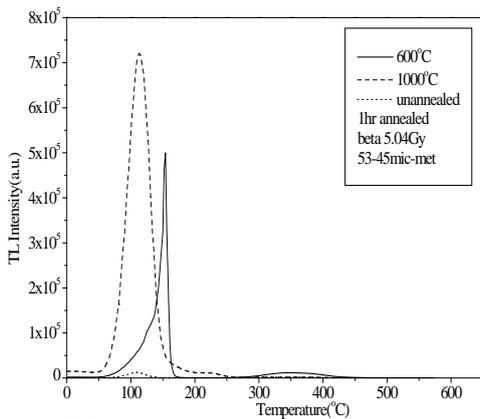


Fig.3 TL glow curve measured at room temperature for 53-45mic-met of annealed and un-annealed temperatures for 53-45mic-met.

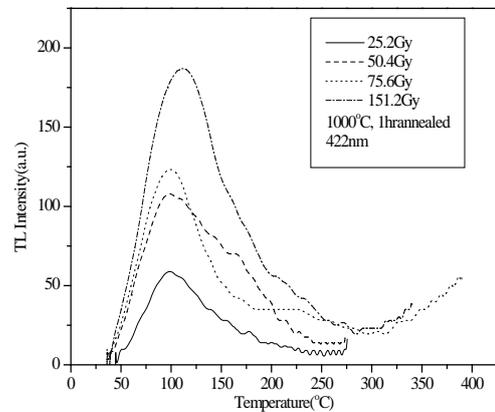


Fig.5 TL recorded at room temperature for 0.422mic-met grain of 1000°C annealed sample at different doses.

The increase in TL intensity with decrease in grain sizes may due to an increase in the surface area per unit mass [2]. Loss of TL is may be due to decrease in available trapping center.

Conclusion: The changes TL signals are responsible for combine effect of radiation and annealing on different particle sizes of synthetic quartz.



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**DEVELOPMENT OF PROTOCOLS TO OPTICALLY STIMULATED
LUMINESCENCE (OSL) OF SYNTHETIC QUARTZ**

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Abstract

The majority optically stimulated luminescence (OSL) signals are obtained from quartz material is responsible to the rapidly bleachable TL peak (325°C). The changes are observed in the OSL decay curve pattern established the correlation of this peak with the 110°C thermoluminescence (TL) peak of quartz material. Researchers were developed various protocols to resolve these correlation under influence of physical condition to the specimen and experimental condition. However, the OSL was recorded at room temperature and at elevated followed by different physical condition to the specimen. The pre-heat treatment at 290°C for 10sec followed by beta exposure and cyclic optical stimulation at 160°C were performed. The OSL results are interpreted to the growth Ge center, effect of phase transformation and thermal transfer of the charges into optically sensitive traps. The TL was recorded before and after the OSL to support the present work.

Keywords: Annealing, Irradiation, thermal transfer, TL, OSL

1. Introduction

The crystal structure of materials is affected by various physical conditions like temperature, pressure, mechanical actions (grinding and crushing) and radiation etc [1]. However, it has been utilized to establish the definite correlation between defects and luminescence intensity [2]. Researchers have studied the role of annealing treatment prior to beta irradiation and pre-heat treatment followed by beta irradiation on changes in luminescence sensitivity of quartz.[3] Several workers[4][5] have reported the annealing temperature up to 600°C leads to dramatic alterations in the OSL sensitivity in quartz. It is due to alterations in the recombination center concentration and effect of phase inversion in quartz. In present work leads to explain the suitable physical condition to the material for better OSL output. The interpretations have made on the basis of thermal transfer process and growth of Ge center with support of the TL after OSL.

2. Experimental Details

Synthetic Quartz (SQ) crystal [6] of 0.063-0.053mm was held at 1000°C in muffle furnace for 1hour to annealing-quenching (AQ) process.

(i) SQ + AQ + 5.04Gy + TL at RT (25°C). (ii)SQ + AQ + 5.04Gy + OSL at RT, 160°C + TL at RT. (iii)SQ + AQ + 5.04Gy + pre-heat to 290°C for 10seconds (post irradiation heat treatment; PIHT) + OSL at 160°C + TL at RT. (iv)SQ + AQ + 25Gy + cyclic OSL at 160°C + TL at RT. The OSL decay curves were recorded through RISO (TL/OSL-DA-15) system, which has a facility of an optical stimulation by blue light 470nm.

3. Results and Discussion

Table-1 reports the annealed sample exhibited two separate peaks at 122°C and 218°C.

Figures 1-4 show the OSL decay curve at room temperature (25°C) as well as at 160°C.

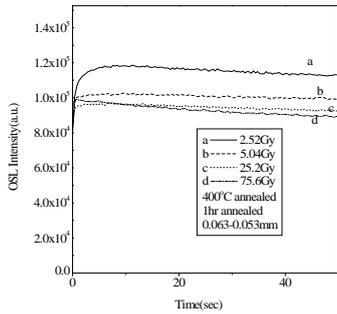


Fig.1 OSL measured at room temperature of 400°C annealed sample for 1hr duration at different doses.

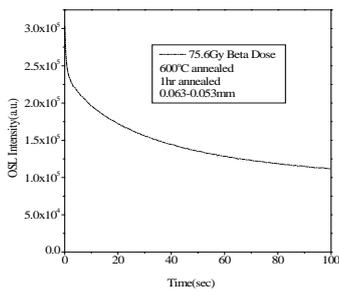


Fig. 2 OSL measured at room temperature of 600°C annealed sample for 1hr duration at 75.6Gy

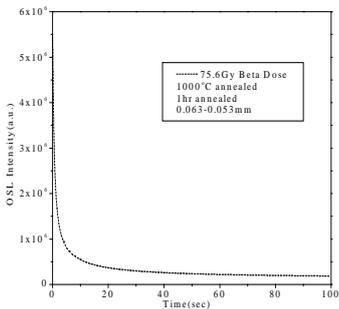


Fig. 3 OSL measured at room temperature of 1000°C annealed sample for 1hr duration at 75.6Gy.

Table 1: TL glow curves were recorded at RT before and after OSL for different protocol; Beta: 5.04Gy and 25.2Gy; Grain Size: 0.063-0.053mm; Annealing: 1000°C for 1hr

	Tm ₁ (°C)	Tm ₂ (°C)	Tm ₃ (°C)	I ₁ (a.u.)	I ₂ (a.u.)	I ₃ (a.u.)
TL before OSL at RT.	122	218	-	1.01×10 ⁶	19212	-
TL after OSL at RT.	113	171	223	53514	8584	4982
TL after OSL at 160°C.	218	382	-	857	161	-
TL after PIHT+ OSL.	223	364	-	3592	1821	-
TL after Cyclic OSL at 16°C.	239	377	-	22520	4097	-

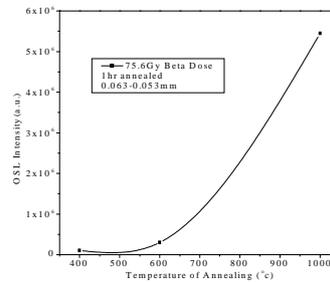


Fig. 4 OSL sensitivity measured at 75.6Gy dose for 1hr annealed sample.

The OSL intensity (160500a.u.) at elevated temperature is lower than OSL intensity (297259a.u.) was recorded at room temperature. Also, the both OSL decay curves show non-exponential pattern for 0.4sec initial stimulation peak time (Table-2). The OSL was recorded at 160°C followed by prior to pre-heat at 290°C for 10sec followed by 5.04Gy beta exposed sample show significant growth of OSL intensity(556041a.u.) compare to without post irradiation heat treatment (Table-2)

Table 2: OSL recoded at room temperature and elevated temperature for different protocol. Grain Size: 0.063-0.053mm; Annealing: 1000°C for 1hr; Beta: 5.04Gy.

OSL (a.u.) at RT.	OSL (a.u.) at 160°C.	OSL (a.u.) at 160°C after PIHT at 290°C for 10sec.	Cyclic OSL (a.u.) at 160°C.
297259	160500	556041	4.1×10^6
			122071
			106120

The OSL intensity reduces during cyclic stimulation at 160°C and shows the stability of 210°C TL peak. The non-exponential pattern of decay curve is due to the growth of 110°C TL peak during optical stimulation at room temperature. It reduces the OSL intensity due to re-trapping of electrons in to shallow levels. On account of this concept, elevated temperature protocols were implemented to avoid the re-trapping of optically released electron [7]. Present work suggests the novel observation regarding the elevated temperature OSL study. It shows non-exponential shape for 0.4 sec initial stimulation peak time and loss of OSL intensity. It may be due to second order kinetics or re-trapping of electrons still exists during stimulation. The enhancement of OSL intensity in post irradiation heat treatment protocols may link with the thermal transfer process [8] of electron. Such transferring of an electron may be into rapidly bleachable trap which give rise OSL for these identical protocols. The cyclic stimulation gives the loss of signals during repetition of stimulation. But it exhibits the stability of TL peaks corresponding 210°C (confirmed by the ESR at room temperature; growth of Ge centers (**Fig.5 and Fig.6**)) and 375°C

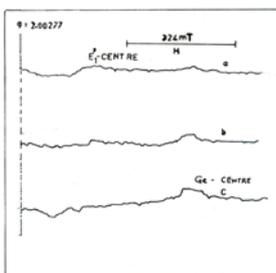


Fig. 5 Room temperature ESR spectra of E1' center and the growth of new Ge center in annealed synthetic quartz for (a) 400°C (b) 600°C (c) 1000°C using 0.1mW microwave power.

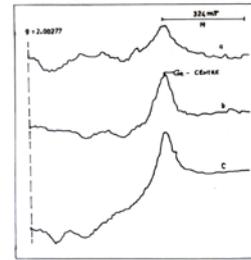


Fig. 6 Room temperature ESR spectra of new Ge center in annealed synthetic quartz for (a) 400°C (b) 600°C (c) 1000°C using 5mW microwave power.

4. Conclusions

The rise in OSL Intensity for 1000°C annealing treatment and post irradiation heat treatment are responsible for the effect of phase transform, and the growth of Ge center as well as the effect of thermal transfer into optically sensitive traps. The non-exponential shape of decay at elevated temperature is due to second order kinetics. The cyclic stimulation suggests the stability of 210°C and 375°C TL peaks.

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