THERMAL ASSISTANCE AND THERMAL QUENCHING RELATED TO DEEP ELECTRON TRAPS IN ANNEALED SYNTHETIC QUARTZ.

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ABSTRACT

Thermally assisted optically stimulated luminescence measured from annealed synthetic quartz using continuous-wave optical stimulation (CW-OSL) is reported. The samples were each annealed at 500, 900 and 1000 ºC for 10 minutes before use. The sample annealed at 900 ºC for 10 minutes was further annealed for 30 and 60 minutes to study features related to the duration of annealing. The samples were stimulated using 470 nm blue LEDs at temperatures between 30 and 200 ºC. Prior to optical stimulation, the sample was preheated to 500 ºC. The integrated OSL intensity goes through a peak as a function of measurement temperature. The CW-OSL luminescence intensity increases from 30 ºC to a maximum at 130 ºC and thereafter decrease to 200 ºC, the maximum temperature of the investigations. The increase in OSL intensity is explained in terms of thermal assistance and the decrease is ascribed to thermal quenching. The activation energy of thermal assistance and thermal quenching for sample anneaed at 500 ºC for 10 minutes were evaluated as 0.359 ± 0.030 eV and 0.57 ± 0.02 eV respectively. The annealing temperature and duration of annealing were found to affect the thermal assistance and thermal quenching parameters to some extent.

Keywords: Thermal assistance, Deep traps, Thermal quenching, Synthetic quartz.

INTRODUCTION

Natural and synthetic quartz have been extensively investigated using thermos-luminescence due to their importance in dating and dosimetry (Preusser., et al, 2009) . There are three techniques in optically stimulated luminescence that have been used. Namely, continuous-wave optically stimulated luminescence (CW-OSL) (Bøtter-Jensen., et al, 2003), linearly-modulated optically stimulated luminescence (LM-OSL) (Bulur, 2000) and time-resolved optically stimulated luminescence (TR-OSL) (Chithambo, 2018). Continuous-wave optical stimulation is the most common technique for optical stimulation of luminescence. In this method, luminescence is observed in the presence of stimulating light. For a detailed explanation of each of the techniques, the reader could refer to references (Bøtter-Jensen., et al, 2003; Chithambo, 2018). Deep traps which is the focus of this study. For convenience, we refer to those traps that are thermally inaccessible when the luminescence material is heated from room temperature to a maximum temperature of 500 ºC (Nyirenda and Chithambo, 2017).

However, the OSL signal made from the deep traps can be measured using optically stimulated luminescence (Polymeris., et al, 2010) . The OSL signal observed after preheating to 500 ºC is due to deep traps. Since the shallow and the main traps present in the glow curve which populates to giving rise to OSL signal are empty, the only likely source of the traps giving rise to OSL is the deep traps (DT). The effects of thermally assisted OSL have been reported in some materials like Al_2O_3: C (Polymeris., et al, 2010; Akslrod., et al., 1998, Nyirenda., et al., 2016), Al_2O_3: C, Mg (Kalita and Chithambo., 2017), quartz (Chithambo and Galloway., 2001; Chithambo., 2003; Chithambo., 2011). Measurement of the OSL as a function of temperature provides a means to calculate some kinetic parameters like activation energy of thermal assistance and thermal quenching using OSL (Chithambo., 2007). Study on the effect of annealing on stimulated luminescence of quartz has been the subject of numerus study. Previous studies on quartz using time-resolved luminescence showed that the luminescence lifetime changes with annealing temperature (Chithambo., 2011); the amplitude of emission bands increases with annealing temperature and the intensity increases with duration of annealing temperature above 700 ^{o}C as noted using radioluminescence (Chithambo and Niyonzima., 2017). The activation energy of thermal quenching also seems to decrease with annealing temperature (recombination centres affected) as concluded using thermoluminescence (Dawam and Chithambo., 2018). Recently, Dawam et al. (2021) studied the effect of duration of annealing on kinetic parameters using TL analysis of secondary peak. In their investigation, they found out that activation energy and activation energy of thermal quenching decreases with increased in the duration of annealing.

In this work, we studied the stimulated luminescence associated to deep electron traps in annealed synthetic quartz. The effects of annealing temperature and duration of annealing on the activation energy of thermal assistance to optical stimulation from deep electron traps and the activation energy of thermal quenching were reported.

EXPERIMENTAL METHOD

Commercially available synthetic quartz (Sawyer Research Products, Ohio, USA) ground into a coarse grain was used. This is the same sample previously studied by Chithambo et al (2011); Dawam and Chithambo (2018). Samples were annealed at 500 ^{o}C for 10 minutes, 900 ^{o}C for 10, 30 and 60 minutes and 1000 ^{o}C for 10 minutes. Measurements were made on similar mass using a RISO TL/OSL DA-20 Luminescence Reader. The luminescence was detected by a photomultiplier tube (EMI 9635QA) through a 7.5 mm Hoya U-340 filter (transmission band 250 - 390 nm FWHM). Samples were irradiated *in-situ* at room temperature using a ^{90}Sr/^{90}Y beta source at a dose rate of 0.10 Gy s^{-1}. Unless otherwise stated, samples were heated at 1 ^{o}C s^{-1} after irradiation to 100 Gy. All the measurements were made in a nitrogen atmosphere to avoid spurious signals from the air and to improve thermal contact between the sample disc and the heater plate. All OSL measurements were carried out in CW-OSL mode using a set of blue LEDs (wavelength, 470 nm; FWHM, 20 nm). LEDs were set to deliver 72 mW cm^{-2} of maximum power at the sample position.

Profile of deep traps in continuous-wave OSL

The characteristics of deep traps were studied by measuring the CW-OSL signal on the samples of synthetic quartz annealed at 900 ^{o}C for 10 minutes using the following steps:

Step 1: The sample was first heated to 500 ^{o}C to clear the background signal

Step 2: The sample was irradiated to 100 Gy at room temperature to populate electron traps

Step 3: The sample was heated to 500 ^{o}C to clear the main traps and all secondary electron traps.

Step 4: The sample was illuminated using 470 nm blue LEDs for 10 s at room temperature and at elevated temperatures during which the CW-OSL from deep traps was recorded.

Step 5: Heating to 500 ^{o}C to record the phototransfered peak induced by phototransfer after the CW-OSL measurement.

The procedure was repeated for the temperature between 30 - 200 ^{o}C. For comparison, the same procedures above were repeated without preheating temperature that is step 3 to monitor conventional OSL from shallower traps.

RESULTS AND DISCUSSION

Fig. 1 shows examples of plots of OSL intensity against stimulation time comparing conventional OSL intensity (shallow traps) with that from deep electron traps measured at 30, 90 150 and 200 °C. It can be observed that the deep trap CW-OSL intensity are different from that of the conventional OSL regardless of the measurement temperature. The CW-OSL from deep traps decays slowly while the conventional OSL signal decays faster as seen from the profiles in Fig.1 for all temperatures measured.

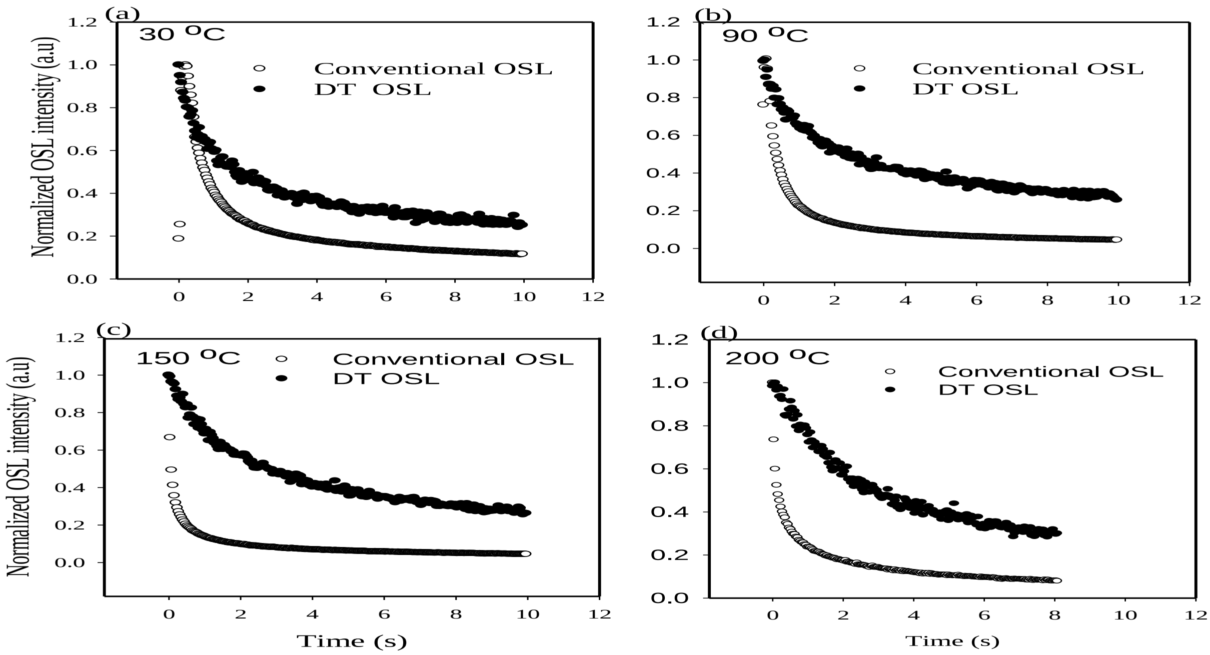

Fig. 1. Examples of conventional OSL and deep traps (DT) CW-OSL decay curves measured from synthetic quartz using 470 nm blue LED for 10 s at 30, 90, 150 and 200 °C following irradiation to 100 Gy.

Dependence of luminescence intensity (CW- OSL) on measurement temperature

The influence of temperature on CW-OSL was investigated. The measurement was carried out on the sample annealed at 900 °C for 10 minutes. The procedure is that the sample was irradiated to 100 Gy prior to preheating to 500 °C to clear the main traps followed by exposure to 470 nm blue LEDs for 10 s at measurement temperature T_i and finally heated to 500 °C to clear residual traps. The measurement was repeated for the temperature between 30 - 200 °C. In the same way, the sequence of the measurements was reversed from 200 – 30 °C. The entire OSL decay curve was integrated to obtain the intensity as a function of measurement temperature. Fig.2 shows the plots of integrated OSL intensity as a function of measurement temperature for the two sequences. In both cases, the variations of integrated OSL intensity against measurement temperature goes through a peak at a maximum of about 130 °C for the two measurements. The difference in OSL intensity between the two runs of the measurements represents a loss of OSL intensity signal as reported by Chithambo and Galloway (2001). This loss in OSL intensity could be due to a sequence of measurements at elevated temperatures. The dependence of the OSL intensity with measurement temperature can be explained in terms of thermal assistance to optical stimulation and thermal quenching. Thermal assistance and thermal quenching are two separable thermodynamics effects that affect the stimulation of luminescence simultaneously (Chithambo and Galloway., 2001). Thermal assistance affects the rate of optical stimulation of charge and thereby affects the luminescence intensity. The overall temperature dependence of luminescence emission affected by thermal quenching and thermal assistance for *n* electron traps can be expressed as

$$I(T) = \frac{I_o \prod_i^n \exp\left(-\frac{E_a}{kT}\right)}{1 + C\exp\left(-\frac{\Delta E}{kT}\right)} \qquad (1)$$

where I is the integrated OSL intensity at temperature T, I_o is the integrated OSL intensity at room temperature, k is the Boltzmann's constant, E_a is the thermal assistance energy to optical stimulation and ΔE is the activation of thermal quenching (Chithambo and Costn., 2017). The constant C is equal to the product of the frequency factor for non-radiative transition and luminescence lifetime corresponding to radiative recombination (Chithambo., 2007). Equation (1) indicates that if OSL is measured at some higher temperature, the initial increase of OSL with temperature is due to dominant thermal assistance to optical stimulation. However, any decrease afterward can be accounted for by dominant thermal quenching. This is the basis for analysis of these processes in α- Al_2O_3:C (Chithambo and Costn., 2017). Since it is not possible to isolate luminescence from particular electron trap (Chithambo and Costn., 2017), equation (1) can only be applied with simplifying assumptions. Under this simplification, equation (1) was used to describe the data shown in Fig. 2. The solid line through the data points in Fig. 2 is the best fit of equation (1). The activation energy of thermal assistance (E_a), thermal quenching (ΔE) and constant C was evaluated from the fits. These values are respectively; E_a = $0.089 \pm 0.004\ eV$; $\Delta E = 0.89 \pm 0.03$ eV and C = 1.42×10^6 for the measurement runs from 30 - 200 °C. When the sequence was reversed, the activation energy of thermal assistance, thermal quenching and the constant C were calculated as $E_a = 0.075 \pm 0.002$ eV ; $\Delta E = 0.84 \pm 0.03$ eV and $C = 5.46 \times 10^6$ respectively. The values of the kinetic parameters for the two runs are consistent. The values of activation energy of thermal quenching reported in the present work are in agreement with those reported by Chithambo (2003) and Chithambo (2004) using time-resolved OSL on the same sample of synthetic quartz.

Analysis based on the conventional OSL

The sample annealed at 900 °C for 10 minutes was irradiated to 100 Gy followed by CW - OSL measurement at temperature T_i for 10 s and finally heated to 500 °C to clear the residual charge from the acceptor traps. The measurement was repeated for temperatures between 30-200 °C. Fig. 3(a) shows the CW-OSL luminescence intensity plotted against stimulation time for measurement temperatures of 30, 130 and 200 °C. It can be observed that for the temperature of 30 °C, the profile of the CW-OSL signal as a function of stimulating time has a peak-like shape. However, as the measurement temperature increases, the CW-OSL signal peak weakens as illustrated in Fig.3 (a). The CW-OSL intensity for each measurement temperature between 30 – 200 °C was obtained again by integrating the entire CW–OSL as a function of simulation time and plotted against measurement temperature (see Fig. 3(b)). It can be observed that OSL intensity increases between 30 -70 ^oC and thereafter decrease from 80 ^oC to the maximum temperature of the measurements. This behaviour is suggesting the simultaneous effects of thermal assistance and thermal quenching. In this case, the change in OSL intensity with measurement temperature could not be satisfactorily fitted with Equation (1). The specific reason is yet not clear however we speculate that the feature observed here is due to involvement of multiple electron traps. Therefore, the simplification used in the fitting process does not valid.

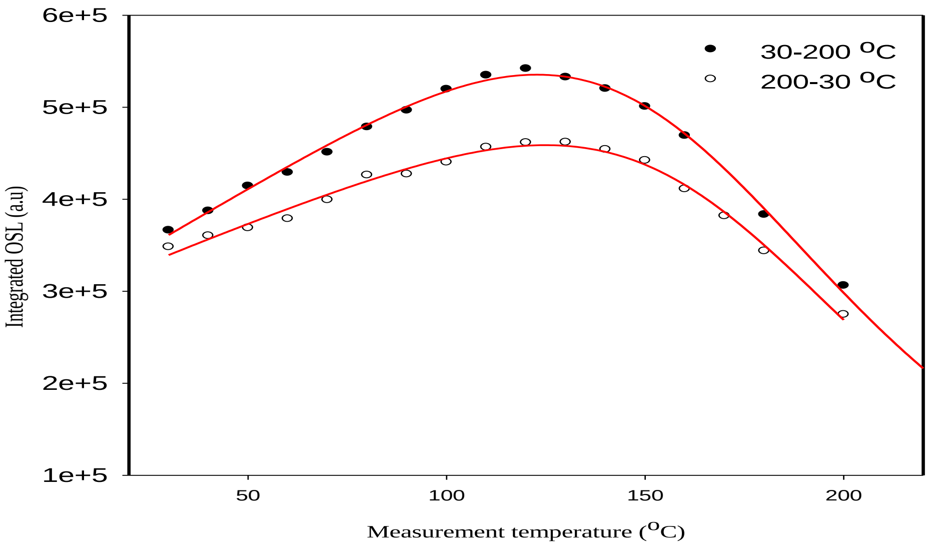

Fig. 2. The relationship between integrated OSL and measurement temperature made from 30-200 ^oC and in turn 200 - 30 ^oC. The solid lines correspond to the best fit of Equation (1).

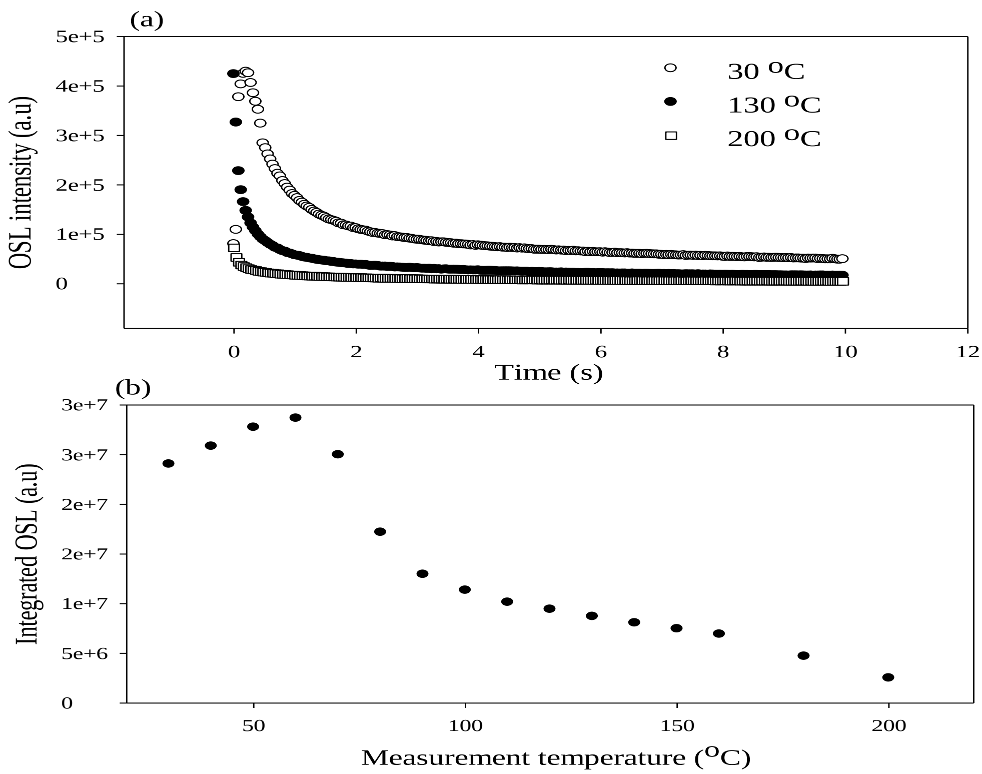

Fig. 3. The plots of OSL intensity as a function stimulation time for measurement temperature at 30, 130 and 200 °C((a) . Integrated OSL versus measurement temperature (b).

Effect of annealing temperature on thermally assisted CW-OSL intensity

Samples of synthetic quartz annealed at 500, 900 and 1000 °C for 10 minutes were used. Samples were irradiated to 100 Gy, preheated to 500 °C at 1 °C s^{-1} followed by CW-OSL measurement for 10 s and finally heated to 500 °C to measure the residual TL signal. In each of the measurements, integrated OSL intensity was obtained and plotted as a function of measurement temperature.

Fig. 4 shows the observed luminescence intensity for different annealing temperatures. As expected the sample annealed at 500 °C shows moderately low intensity compared to the sample annealed at 900 and 1000 °C. The CW-OSL intensity increases with annealing at 500, 900 and 1000 °C. This is important because annealing temperature induces luminescence sensitivity changes in the sample (Preusser., et al, 2009). The annealing of quartz undergoes phase inversion at 573 and 870 °C (Chithambo and Costn., 2017) leading to significant changes in the OSL signal.

The kinetic parameters for the samples corresponding to different annealing temperatures were calculated again by fitting with Equation (1). The resulting values of the activation energy of thermal assistance E_a and the activation energy of thermal quenching ΔE for the sample annealed at 500 °C are respectively as follows; $E_a = 0.36 \pm 0.03$ eV; $\Delta E = 0.57 \pm 0.02$ eV. The constant C for the 500 °C annealed sample was calculated to be 2.26×10^7. For the sample annealed at 900 °C the kinetic parameters are respectively $E_a = 0.08 \pm 0.01$ eV; $\Delta E = 0.82 \pm 0.02$ eV and $C = 5.46 \times 10^5$. Similarly, the activation energy of thermal assistance, thermal quenching and the constant C for the sample annealed at 1000 °C are found as $E_a = 0.22 \pm 0.01$ eV; $\Delta E = 0.59 \pm 0.03$ eV and $C = 3.40 \times 10^7$. The values of the activation energy of thermal quenching are consistent for the annealing temperature of 500 and 1000 °C and increases for annealed at 900 °C. However, the activation energy of thermal assistance is much higher for samples annealed at 500 and 1000 °C. The differences in the values of E_a observed in different annealed samples are related to the phase inversion of the sample at 573 and 870 °C.

Influence of duration of annealing on thermal assistance and thermal quenching

The influence of the duration of annealing on synthetic quartz was investigated. Samples were annealed at 900 °C for 10, 30 and 60 minutes prior to use. The same experimental procedure reported in section 3.1 was used for this investigation. Fig. 5 shows the graphs of integrated OSL intensity against measurements temperature for the samples annealed for 10, 30 and 60 minutes. The intensity decreases with an increase in the duration of annealing as seen in Fig. 5. At a given measurement temperature, the OSL intensity for the sample annealed for 10 minutes is higher than that of the samples annealed for 30 and 60 minutes. The OSL intensity is the least for the sample annealed for 60 minutes. This indicates that the concentration of deep traps decreases due to prolong annealing. The thermal assistance and thermal quenching were also observed from the rising and the falling part respectively of the peaks shown in Fig 5.

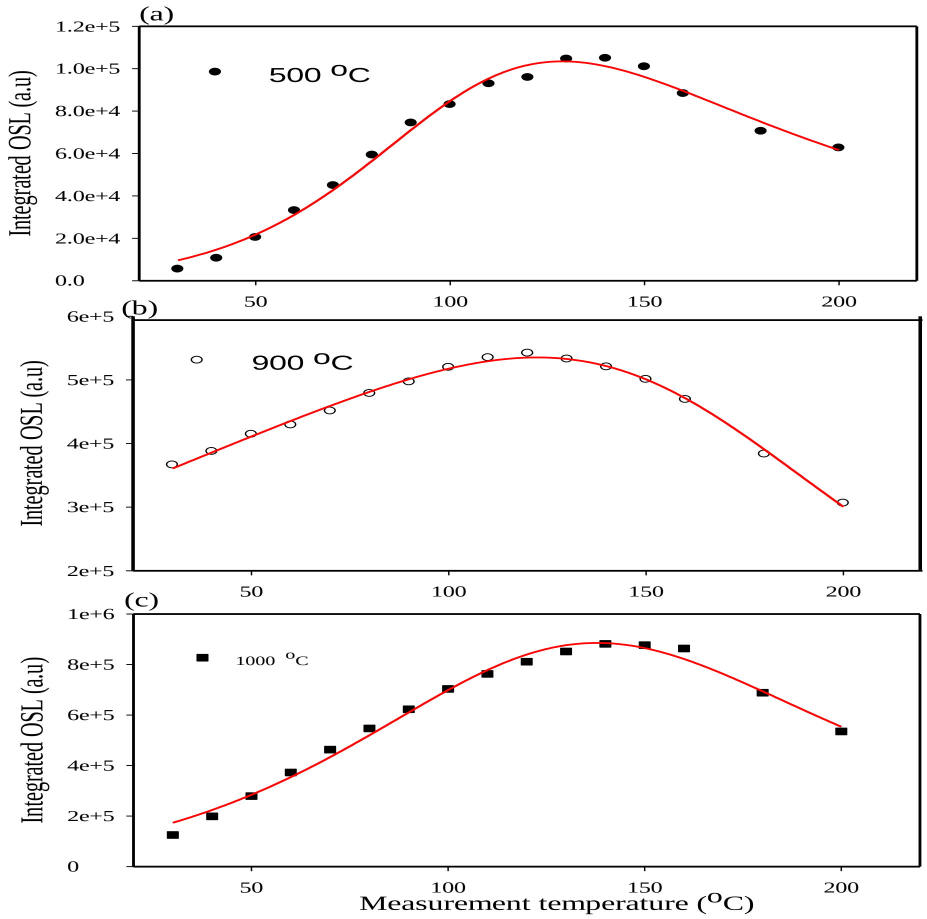

Fig. 4. The variation of integrated OSL intensity as a function of measurement temperature for samples annealed at 500, 900 and 1000 °C for 10 minutes. The solid line in each shows the best fit of Equation (1).

The activation energy of thermal assistance and thermal quenching for the sample annealed for 10 minutes are determined as $E_a = 0.074 \pm 0.002$ eV, $\Delta E = 0.65 \pm 0.02$ eV. When the duration of annealing was increased to 30 and 60 minutes, the kinetic parameters corresponding 30 minutes annealed sample were found to be $E_a = 0.094 \pm 0.005$ eV, $\Delta E = 0.74 \pm 0.03$ eV and those of 60 minutes annealed sample be $E_a = 0.063 \pm 0.004$ eV, $\Delta E = 0.75 \pm 0.02$ eV respectively.

These values of the activation energy of thermal assistance and thermal quenching are consistent for the three different duration of annealing. The values of the activation energy of thermal quenching are slightly increased with duration of annealing. This signifies that the recombination centre is affected by the duration of annealing as suggested by Chithambo and Niyonzima (2017).

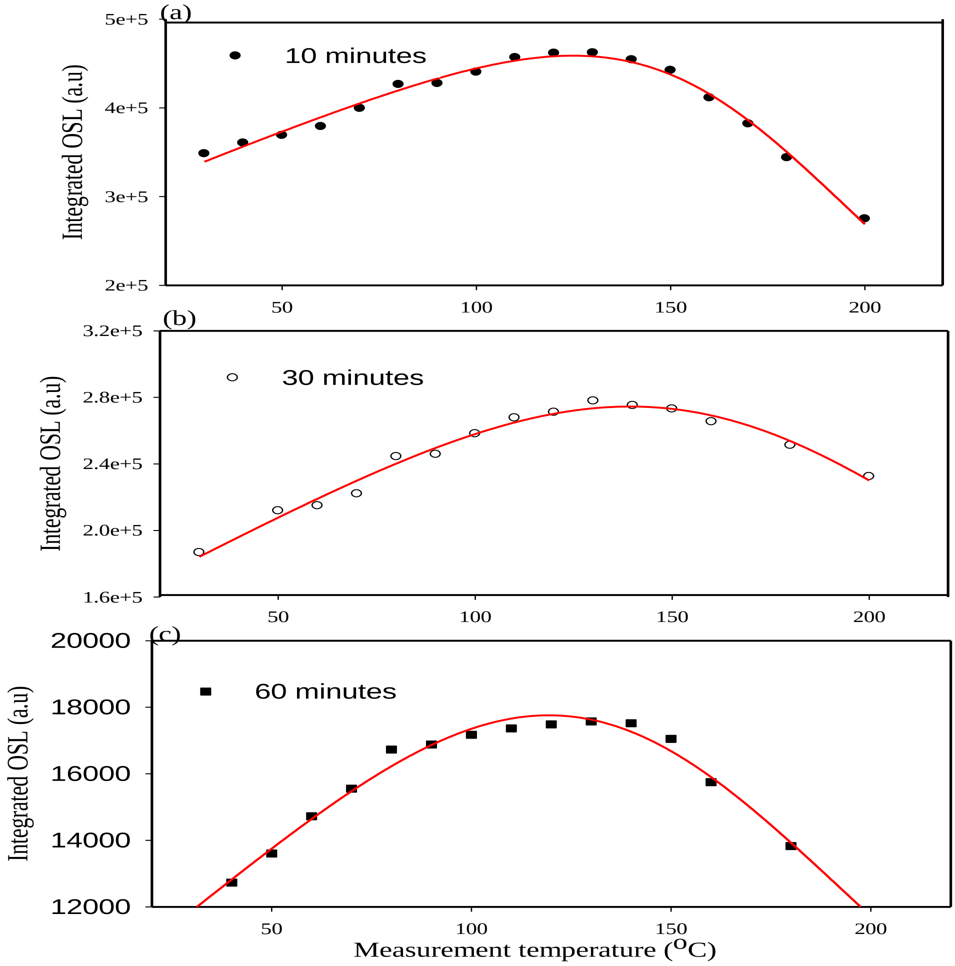

Fig. 5. The plots of integrated OSL intensity against measurement temperature for the sample annealed at 900 °C for the duration of 10, 30 and 60 minutes. The solid line in each shows the best fit of Equation (1).

Effect of illumination temperature on photo-transferred thermos-luminescence

The influence of illumination temperature on photo-transferred thermos-luminescence (PTTL) induced by the CW-OSL was investigated on the sample annealed at 900 °C for 10 minutes. The experimental procedure was that the sample was irradiated to 100 Gy followed by preheating to 300 °C to remove some glow peaks from the glow curve observed below 300 °C and measurement of complete glow curve after illumination at a given temperature by 470 nm blue light for 10 s. Fig 6(a) shows a conventional TL glow curve and Fig. 6(b) shows a PTTL glow curve measured from the sample annealed at 900 °C for 10 minutes. Three PTTL peaks were reproduced at 80, 136 and 200 °C labelled as A1, A2 and A3 respectively. The PTTL peaks were monitored for each illumination (i.e., OSL measurement) temperature between 30 and 200 °C for constant illumination time of 10 s. The PTTL peak positions were constant with the temperature of illumination.

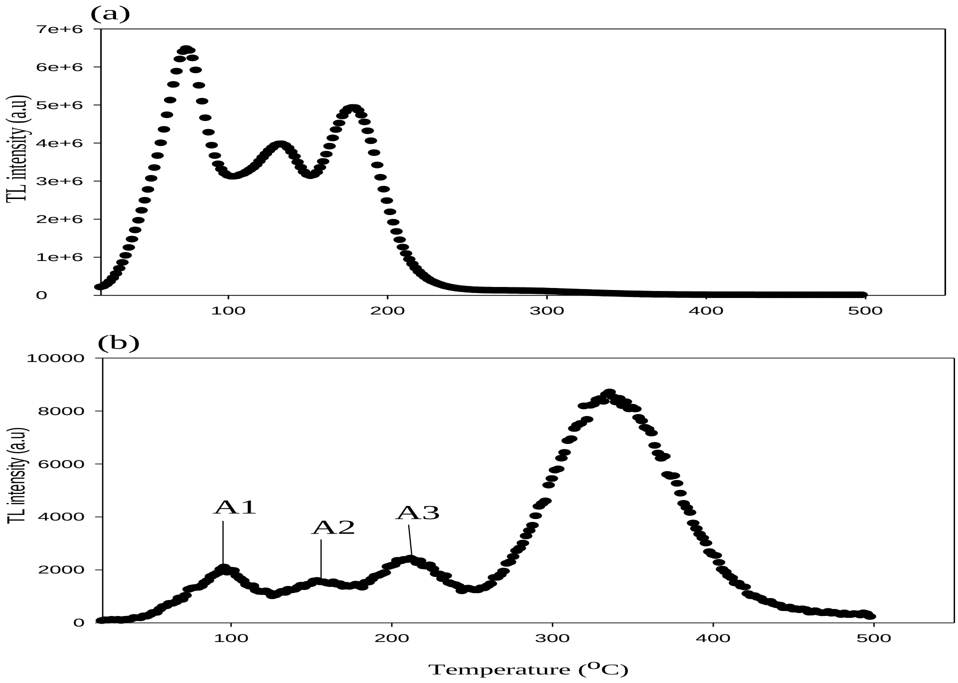

Fig. 6. A conventional TL glow curve following the irradiation to 100 Gy of the sample annealed at 900 °C for 10 minutes (a). A PTTL glow curve measured after preheating to 300 °C (b).

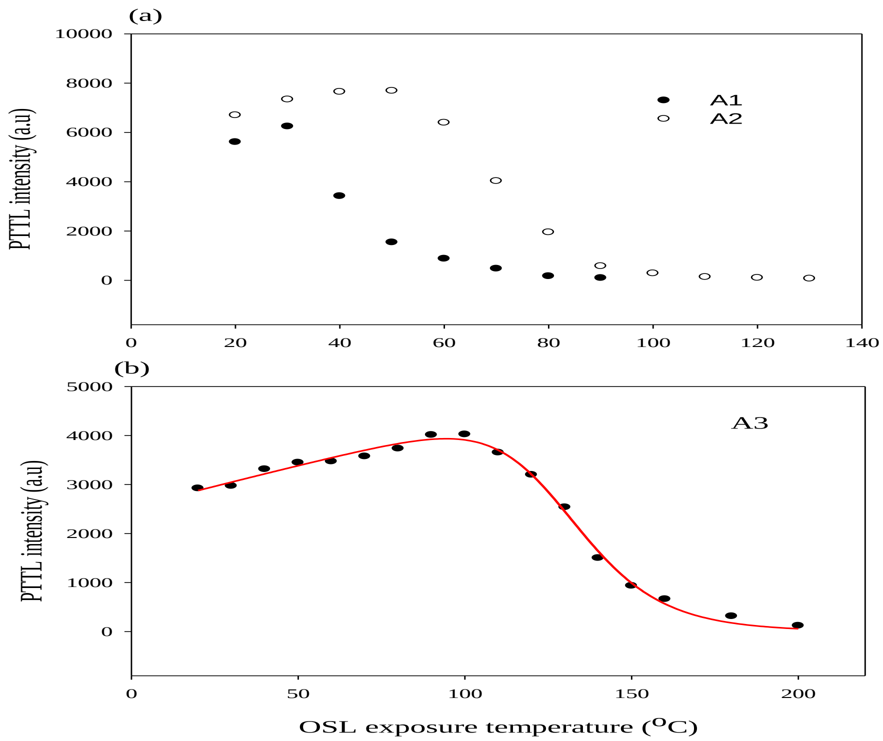

Fig. 7. Plots of PTTL intensity as a function of illumination temperature for PTTL peaks A1, A2 and A3. The solid line in (b) is the best fit of Equation (1).

Fig. 7 shows the plots of PTTL intensity against illumination temperature for the three PTTL peaks reproduced. As shown in Fig. 7(a) the PTTL intensity initially increases with illumination temperature and thereafter decreases monotonically for peaks A1 and A2. However, for peak A3, the PTTL intensity increases with illumination temperature between 30 -100 ^{o}C and then subsequently decreases monotonically to 200 ^{o}C. The increase in the PTTL intensity with measurement temperature is associated with the phenomenon of thermal assistance. The decrease beyond 100 ^{o}C may be due to the combined effect of less retrapping at the main trap of charges stimulated from the deep traps and thermal quenching at recombination sites at higher temperature.

The activation energy of thermal assistance and thermal quenching of peak A3 was evaluated by fitting the data of PTTL intensity as a function of illumination temperature using Equation (1) as shown in Fig. 7(b). The parameters were found as E_a = 0.044 $\pm$0.002 eV, ΔE = 0.76 $\pm$0.01 eV and $C = 4.39 \times 10^7$. The values of the activation energy of thermal assistance and thermal quenching are consistent with the calculated ones reported in the previous sections. The detailed explanation regarding PTTL profiles and their analysis was reported elsewhere by Chithambo and Dawam (2020) and Dawam (2020).

CONCLUSION

Thermally assisted CW-OSL from annealed synthetic quartz measured using 470 nm blue LEDs has been studied. The thermally assisted OSL was measured from a temperature between 30 – 200 ^{o}C. It was observed that the CW-OSL from deep electron trap decays slower than that of the conventional OSL from intermediate traps. The values of the activation energy of thermal quenching increases with increase in duration of annealing. This signifies that the recombination centre is affected by the duration of annealing. The values of the activation energy of thermal quenching are consistent for the annealing temperature of 500 and 1000 ^{o}C and increases for sample annealed at 900 ^{o}C (see Table 1).

Table 1. A comparison of thermal assistance and thermal quenching parameters evaluated from the fits using CW-OSL integrated intensity and measurements temperature in synthetic quartz.

Temp. range (^{o}C)	Annealing time (minutes)	Annealing temp. (^{o}C)	Preheat (^{o}C)	$E_a(eV)$	ΔE(eV)	C	Ref.
30-200	10	900	500	0.089 ± 0.004	0.89 ± 0.03	1.42×10^6	Fig. 2
200-30	10	900	500	0.075 ± 0.002	0.84 ± 0.03	5.46×10^6	Fig. 2
30-200	10	500	500	0.359 ± 0.030	0.57 ± 0.02	2.26×10^7	Fig. 4(a)
30-200	10	900	500	0.075 ± 0.005	0.82 ± 0.02	5.46×10^5	Fig. 4(b)
30-200	10	1000	500	0.216 ± 0.020	0.59 ± 0.03	3.40×10^7	Fig. 4(c)
30-200	10	900	500	0.074 ± 0.020	0.65 ± 0.02	3.91×10^6	Fig. 5(a)
30-200	30	900	500	0.074 ± 0.005	0.74 ± 0.03	1.89×10^6	Fig. 5(b)
30-200	60	900	500	0.063 ± 0.004	0.75 ± 0.02	3.70×10^6	Fig. 5(c)
30-200	10	900	300	0.044 ± 0.002	0.76 ± 0.01	4.39×10^7	Fig. 7(b)

Furthermore, the annealing temperature and duration of annealing were also found to affect the activation energy of thermal assistance to optical stimulation. An experiment using PTTL reveals that the intensity of a PTTL peak at 200 ^{o}C initially increases with illumination temperature up to 100 ^{o}C and thereafter decreases monotonically in a manner that signifies the existence of thermal assistance and thermal quenching.

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