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Investigating the thermally transferred optically stimulated luminescence source trap in fired geological quartz

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HIGHLIGHTS

- OSL pre-dose reproducibility test suggested thermal charge transfer.
- Thermal annealing curve also indicated the presence of TT-OSL emission.
- Correlation studies showed 375 and 325 °C peaks to be the source and receptor respectively.
- TT-OSL observed in case of samples which contained 375 °C TL peak.
- Bleaching nature of the signal seems to establish the 375 °C TL as the source trap.

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ABSTRACT

The pre-dosed thermoluminescence (TL) emission of quartz has been found to be useful in retrospective dosimetry and archaeometry. Though the pre-dosed optically stimulated luminescence (OSL) and TL emissions have been reported to be similar, the former has been found to be un-reliable for the equivalent dose estimation. As this measurement protocol involves thermal heating at around 400 °C, the work reported in this paper investigated the influence of this heating on the OSL using fired specimens from various regions. The results suggested that the discrepancy in the behaviour of two emissions is caused by the presence of the thermally transferred optically stimulated luminescence (TT-OSL) induced by thermal-activation involved in the pre-dose treatment. This transferred signal was observed to be very significant in the case of samples containing a prominent higher-temperature TL peak at ~375 °C. The characterization of this signal based on (i) the nature of the glow curves, (ii) thermal-annealing of the OSL trap, (iii) observation of the TT-OSL, (iv) bleaching of the source trap and (v) the correlation between TL and OSL seems to suggest that the trap corresponding to this TL peak is the source trap in the TT-OSL emission mechanism.

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1. Introduction

Quartz grains, once irradiated, either naturally or artificially, release luminescence upon stimulation by light in the visible range. This emission, known as optically stimulated luminescence (OSL), occurs due to recombination of charge, which is released from traps by optical stimulation, with luminescence centres. Optically stimulated luminescence emission has been widely used in retrospective dosimetry and the dating of sediments deposited during the last 100,000 years (Aitken, 1998). The utility of this emission for these applications was made possible due to complete bleaching of the fast component of this signal within a few minutes of exposure to sunlight (Godfrey-Smith et al., 1988).

The OSL of quartz has been observed for a wide range of stimulation wavelengths. The exact nature of the charge kinetics involved in the mechanism of OSL emission in irradiated quartz illuminated with light is complex and not fully understood. OSL traps are emptied sequentially according to their respective cross-sections, but, of course, this process causes an overlap in the decaying OSL components. Similarly, the thermoluminescence (TL) traps, though emptied serially, give rise to overlapping peaks because more than one trap is emptied at the same time. The hypothesis regarding the source of the electrons responsible for the OSL has been diverse. Ditlefsen and Huntley (1994) proposed the participation of a number of traps in the luminescence emission.

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Subsequently, Huntley et al. (1996) were unable to observe any correlation between the 325 °C TL and OSL recorded for optical exposures of 488 and 633 nm. The OSL emission originating from very deep traps, traps which give rise to TL even after a TL read out temperature of 500 °C has been observed for both un-fired and fired quartz (Kitis et al., 2010). However, generally, the initial, fast OSL is believed to be associated with the 325 °C TL glow peak for sedimentary samples. This relation was proposed by Smith et al. (1986) and has subsequently been supported by the experimental work of Wintle and Murray (1997). The signatures of this fast OSL are that it becomes (i) annealed with pre-heating at 360 °C for 10 s and (ii) bleached with a bleaching of a few tens of seconds in the sunlight, which can be replicated using the blue light source of the Risø system (Bøtter-Jensen et al., 1999; Jain et al., 2003; Wintle and Murray, 1998).

Thermal treatment, which is necessary for OSL measurements of quartz, has been observed to introduce the unwanted phenomenon of the thermal transfer of charge. Rhodes (2000), in his work on glacigenic quartz, also observed significant TT-OSL signals induced by pre-heating at temperatures in the range of 200–360 °C in a number of samples. This signal was observed to be solely related to natural dosing effects. Li and Li (2006) while studying the thermal transfer over a wide temperature range suggested the association of the basic transfer with three types of source traps: shallow, medium and deep. It was Wang et al. (2006) who found this thermally transferred OSL (TT-OSL) to be a valuable signal for significantly extending the upper limit of the dating of sediments based on the fast OSL signal. The nature of the OSL and TT-OSL decay-curves suggest that both of these signals are derived from the same trap, though in case of the latter the charge is thermally transferred from the source trap to the 325 °C TL trap (Adamiec et al., 2008). A single transfer mechanism has been found to explain the production of the thermally transferred optically stimulated luminescence signal, TT-OSL, observed for stimulation above 120 °C in quartz (Adamiec et al., 2008). The simulations that have been carried out so far, also, support the single transfer mechanism (Pagonis et al., 2008). The characterization of the source trap, which included studying the separation of high temperature TL peaks and the trap parameters, has led to hypothesis that the source trap is a trap associated with one of the higher-temperature TL peaks, 375 or 480 °C (Adamiec et al., 2010). The heating temperature involved in the zeroing of the charge responsible for the TT-OSL also suggested that the source trap is a deep trap. This trap has also been reported to be very difficult to bleach out completely, even with a bleaching duration of two weeks (Kim et al., 2009). A good account of this phenomenon, which addresses the various aspects of this emission, has been compiled by Duller and Wintle (2012). Attempts have been made to investigate the contribution of lower-temperature TL peaks (160, 220 and 280 °C) to the transferred signal by lowering the pre-heating temperature during OSL measurement in the TT-OSL SAR protocol from 260 to 180 °C (Porat et al., 2009). Although the transferred signal was seen to be enhanced, no appreciable change in the D_e was observed noticed once charge-transfer was undertaken at higher-temperatures (Porat et al., 2009; Jacobs et al., 2011). Duller and Wintle (2012) have attempted to explain this observation in two ways. Their first suggestion is that the contribution of these low-temperature peaks to the TT-OSL is small compared to that of the deeper traps once the transfer is performed at higher-temperature. The second is based on the preferential transfer of charge to the 325 °C TL trap. In this case, it is hypothesized that the low-temperature TL peaks are not spatially associated with this trap.

A number of studies have been undertaken by various researchers to observe the relation between the 110 °C TL peak and the OSL signal of quartz (Stoneham and Stokes, 1991; Stokes, 1994; Koul, 2012). The primary interest in such studies has been to utilize the 110 °C TL peak to monitor the variation in the OSL emission sensitivity as a function of heating. This TL glow peak has been successfully used as the sensitivity monitor in a single aliquot additive-dose protocol. Again, in the development of the single aliquot regenerative-dose (SAR) protocol for OSL dating the 110 °C TL peak has been found to be proportional to the OSL signal (Murray and Roberts, 1998). The linear relation between the two emissions has been established by many researchers for high heating temperatures of up to ~1000 °C (Chen et al., 2001; Schilles et al., 2001; Li, 2002; Koul, 2012). The impact of various stimuli on the pre-dosed TL and OSL emissions of fired quartz specimen has also been observed to be similar (Koul and Chougaonkar, 2007; Koul, 2012). A good relation between the two emissions was observed up to the highest applied heating temperature of 900 °C. These correlation studies have been undertaken by incorporating the fast component of the CW-OSL signal. However, the pre-dosed OSL has been found to be unfeasible for equivalent dose estimation (Koul et al., 2010). Additionally, this relationship between the two emission phenomena might not be valid for other components of the OSL signal (Jain et al., 2003).

The pre-dose based technique involving the 110 °C TL emission has been found to be useful in a variety of applications, such as retrospective dosimetry and the dating of artifacts (Bailiff and Haskell, 1984; Koul, 2008). The pre-dose sensitization mechanism involves the integrated administration of radiation and heat treatments (Bailiff, 1994; Kitis et al., 2006; Koul, 2008). The sensitivity of crystalline quartz, in this case, is governed by the previous radiation dose that it has experienced at room temperature and the subsequent thermal-activation. Zimmerman (1971) established the pre-dose model based on the transfer of charges from one recombination centre to another. The pre-dose (natural or laboratory-administered dose) populates the reservoir centre, R, rather than the luminescence centre, L, with holes because of the former's presumably higher capture cross-section. The subsequent thermal treatment transfers the holes from R to L centres. This charge-transfer enhances the recombination rate by means of increased population of holes trapped in the recombination centre and results in the sensitization of the signal, known as pre-dosed sensitization.

In this work, a pre-dose reproducibility test of OSL signals was undertaken to determine the feasibility of the use this signal in equivalent-dose measurement. This observation suggested the occurrence of charge-transfer at 400 °C, the thermal-activation temperature employed for the pre-dosed signal. This assumption regarding the charge-transfer was substantiated by recording an appreciable signal from a pre-bleached specimen with pre-heating at 400 °C. The recording of the thermal-annealing of the OSL emission with pre-heating in the range of 250–500 °C exhibited signature of thermal transfer initiated at ~325 °C. Interestingly, the specimen that did not contain the higher-temperature TL peak, sample D, was distinct from the rest of the samples in all observations, which also included the measurement of the TT-OSL. The similarity in the nature of the shine down curves for all pre-heating temperature indicated that the luminescence was derived from the trap associated with 325 °C TL peak. Correlation studies involving the relationship between (i) the OSL measured for various time periods and the subsequent TL and (ii) the 375 °C TL and TT-OSL growth curves seemed to demonstrate the 325 and 375 °C TL traps to be the receptor and source traps in the TT-OSL mechanism. The bleaching nature of this emission was seen to be similar to that reported in the literature.

2. Experimental details

The luminescence measurements were performed using an automatic Risø TL/OSL, TL-DA-15 system having a blue light-

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