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# Performance of pulsed OSL stimulation for minimising the feldspar signal contamination in quartz samples

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## HIGHLIGHTS

- The performance of pulsed OSL on feldspar contaminated quartzes were investigated.
- Time resolved OSL was used to check the dominance of the quartz OSL in the off-time.
- The quartz OSL was dominated in the off time for all samples.
- Some samples showed anomalous fading in the quartz OSL.

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## ABSTRACT

The use of pulsed optically stimulated luminescence (POSL) stimulation to remove contaminated feldspar OSL signal in quartz fractions has been tested either on artificial or natural mixtures of quartz and feldspar grains, using samples with good luminescence characteristics of quartz. The problem of feldspar contamination in quartz, however, is often accompanied by other undesirable luminescence characteristics such as a dim OSL signal and slow OSL decay. We report here, on the performance of POSL for nine sand-sized quartz samples from different parts of the Earth, which show severe feldspar contamination in the quartz OSL signal. The time resolved (TR)-OSL signal of these samples was measured to check the dominance of quartz OSL in their off-time signals. The fitting results of the off-time TR-OSL signal showed that the quartz OSL signal dominated during the off-time. The POSL $D_e$ values for these quartz samples are on average ~30% larger than the continuous wave (CW)-OSL $D_e$ values due to the anomalous fading of the feldspar OSL signal. Furthermore the POSL ages were compared with post-infrared infrared stimulated luminescence (pIRIR) ages from K-feldspar for the samples and in most cases the two ages were consistent. These results indicate that POSL is an effective tool to remove feldspar contamination even for dim quartz samples. However, there are quartz samples which show severe anomalous fading in the quartz OSL signal itself. For such samples feldspar may be a better dosimeter.


## 1. Introduction

Time resolved optically stimulated luminescence (TR-OSL) using pulsed stimulations has been studied by various authors to understand recombination processes of quartz and feldspar (e.g., Ankjærgaard et al. 2009, 2010; Clark et al., 1997; Chithambo and Galloway, 2000a,b; Jain and Ankjærgaard, 2011; Pagonis et al., 2009; Sanderson and Clark, 1994; Tsukamoto et al., 2006, 2010). The quartz TR-OSL signal has been first measured and reported by Bailiff (2000) and Chithambo and Galloway (2000a,b). Ankjærgaard et al. (2010) investigated the variation of the lifetime from 30 natural quartz samples measured at 125 °C. They reported that the main component of most quartz samples lies between 35 and 40 μs, but minor shorter and longer lifetime components exist (Ankjærgaard et al., 2010). The lifetimes of quartz as well as feldspar and salt measured by TR-OSL and TR-optically stimulated exoelectron (OSE) emission have been compared by Tsukamoto et al. (2010). They concluded that the relatively long OSL lifetime of quartz originated from de-excitation of the exited state of the recombination centre, because OSE lifetimes were much shorter than OSL lifetimes.

TR-OSL of feldspar has been measured mainly with IR-laser or

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IR-LEDs as stimulation sources (Clark et al., 1997; Clark and Bailiff, 1998; Tsukamoto et al., 2006). Ankjærgaard et al. (2009) studied TR-OSL of various feldspars stimulated with a green laser and blue LEDs and demonstrated that the signals can be expressed as a sum of 4 exponentially decaying components, with the majority of the intensity depleted within 1 μs. The use of pulsed OSL (POSL) stimulation to separate a quartz OSL signal from a sample contaminated with feldspar has been proposed and tested by Denby et al. (2006) and Thomsen et al. (2006). With a pulsed LED setting of 50 μs on/off-time and recording the signal only during the off-time, they were able to obtain the accurate $D_e$ value of quartz in the presence of up to 40% of contaminating feldspar (by mass) using artificial mixtures of quartz and feldspar with different mixing ratios. Adopting the same on/off-time setting, Thomsen et al. (2008a) further tested the ability of POSL to minimise the feldspar OSL contamination using 11 natural unseparated quartz and feldspar samples. By measuring POSL after an IR stimulation, it was possible to measure $D_e$ values from unseparated samples which were indistinguishable from the separated quartz $D_e$. Furthermore, Ankjærgaard et al. (2010) investigated the quartz/feldspar signal ratio using various on- and off-time settings, and concluded that the ratio is optimised when equal on- and off-time pulses of 50 μs and only the initial part of POSL decay curve is used.

In these previous studies either artificial mixtures of quartz and feldspar or unseparated natural samples whose quartz have desirable luminescence characteristics have been used to test the performance of POSL. There are a number of applications of POSL for feldspar contaminated quartz samples from various origins (Dörschner et al., 2012; Feathers et al., 2012; Komatsu and Tsukamoto, 2015; Lauer et al., 2010, 2011; Munyikwa et al. 2011; Sohbati et al., 2012; Thiel et al., 2010; Tsukamoto et al., 2013). These studies also revealed that feldspar contaminated quartz samples often have other difficult luminescence properties (e.g. dim OSL signal, no clear fast component, and significant sensitivity change after heating). However, the performance of POSL, i.e. whether or not POSL provides a pure quartz OSL signal for such problematic quartz samples has not been tested thoroughly.

This paper aims to test the performance of POSL for nine sand-sized quartz samples from Italy, Tibet, Tajikistan, and Egypt which have severe feldspar contamination in the quartz OSL signals after the standard quartz preparation procedures. TR-OSL signals were analysed to investigate whether the off-time signal is dominated by quartz OSL. OSL decay curve shapes, equivalent doses ($D_e$) and anomalous fading rates (g-values) are then compared between pulsed and continuous wave (CW) stimulations. Finally CW-OSL and POSL ages from quartz and post-infrared infrared stimulated luminescence (pIRIR) ages from K-feldspar are compared.

## 2. Samples

Nine sand sized quartz samples from Italy (SC_1 and IA_1; Thiel et al., 2010), Egypt (LUM2347 and LUM2348), Tajikistan (Ur-3a and Ur-5; Komatsu and Tsukamoto, 2015) and Tibet (LUM1129, 11TYL1 and 11TYL13) were used in this study (Table 1). Three K-feldspar samples, SC_1 from Italy and 11TYL1 and 11TYL13 from Tibet and a calibration quartz provided by Technical University of Denmark were also used for comparison. The samples SC_1 and IA_1 originate from beach ridge sediments. Four samples (11TYL1, 11TYL13, Ur3a and Ur5) were deposited in a lacustrine environment, whereas LUM1129 is a dune sand. LUM2347 and LUM2348 were taken from an archaeological site. The samples were sieved between 150 and 200 μm (except for SC_1 and IA_1 between 100 and 200 μm), treated with hydrochloric acid, sodium oxalate, and hydrogen peroxide then density separated using a heavy liquid (sodium polytungstate) between 2.62 and 2.68 g/cm$^3$ to extract the quartz rich fraction and less than 2.58 to obtain the K-feldspar fraction. The quartz rich fraction was etched with 40% HF for 1 h and re-sieved either with a 100 μm sieve (SC_1 and IA_1) or 150 μm sieve (all the other samples) to remove any smaller grains. The quartz and K-feldspar grains were mounted on stainless steel discs using silicone oil spray.

## 3. Equipment and measurement settings

A Risø TL/OSL DA-20 reader equipped with pulsed stimulation and Photon Timer attachments (Lapp et al., 2009) was used for all luminescence measurements in this study. All POSL stimulations using blue LEDs (470 ± 30 nm) were carried out with 50 μs on and 50 μs off-time for 200 s at 125 °C. The gating period during the off-times started at 2.5 μs after the stimulation pulse was switched off (default setting of Sequence Editor). Pulsed IR stimulations with IRLEDs (870 ± 20 nm) were done in the “pseudo-CW” mode, with 1 ms on- and 10 μs off-time for 100 s at 125 °C. All CW-OSL and IRSL stimulations were made at 125 °C for 100 s, keeping the actual stimulation time same as in the pulsed measurements. TR-OSL measurements were performed using 50 μs on and 450 μs off-time at 125 °C for 1000 s and the data were analysed with a bin width of 410 ns. All CW-OSL and POSL stimulations were done following an IR stimulation at 125 °C (100 s) to monitor and to bleach the feldspar OSL signal except if otherwise stated. The OSL and IRSL signals were detected through a 7.5 mm HOYA U-340 filter. The settings and SAR protocols used in this study are summarised in Table 2. Generally a higher preheat temperature helps reduce the contaminating feldspar OSL signal in quartz samples. A preheat of 260 °C was used for SC_1 and IA_1 following Thiel et al. (2010) with a cut heat of 240 °C (0 s). However, for all the other samples a preheat of 160 °C for 10 s and a cut heat of 160 °C for 0 s were used. For Ur-3 and Ur-5a from Tajikistan Komatsu and Tsukamoto (2015) reported that any higher preheat temperatures induced severe sensitivity change and resulted in a failure in the dose recovery tests. This prevented us from using higher preheat and IR stimulation temperatures to further reduce the feldspar OSL signals.

**Table 1**
List of samples used in this study.

| Sample | Origin | Type of sediment | References |
|---|---|---|---|
| IA_1 | Sardinia, Italy | Beach ridge | Thiel et al. (2010) |
| SC_1 | Sardinia, Italy | Beach ridge | Thiel et al. (2010) |
| LUM2347 | Fawakhir, Egypt | Taillings from an old mine | |
| LUM2348 | Fawakhir, Egypt | Taillings from an old mine | |
| Ur-3a | Lake Karakul, Tajikistan | Lacustrine | Komatsu and Tsukamoto (2015) |
| Ur-5 | Lake Karakul, Tajikistan | Lacustrine | Komatsu and Tsukamoto (2015) |
| LUM1129 | Namco, China (Tibet) | Dune | |
| 11TYL1 | Tangra Yum Co, China (Tibet) | Lacustrine | |
| 11TYL13 | Tangra Yum Co, China (Tibet) | Lacustrine | |