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# Assessing the impact of IR stimulation at increasing temperatures to the OSL signal of contaminated quartz



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## HIGHLIGHTS

• The temperature effect of IRSL to the resolved OSL signal of quartz is identified.

• IRSL above 50 °C stimulates both fast and medium quartz OSL components.

• SAR equivalent doses with different stimulation modes, yield similar results.

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## ABSTRACT

The purpose of the present study is to identify the effect of the increasing temperature IR stimulation to the component-resolved OSL luminescence signal of mixed quartz-feldspars material. Post IR OSL signals measured at 110 °C were analysed via only general order kinetic terms, while IR signals obtained at increasing temperatures were de-convolved using the sum of general order kinetics plus a tunnelling component. By increasing stimulation temperature, it was demonstrated that IRSL at temperatures above 50 °C does not only stimulate feldspar but also stimulates both fast and medium quartz OSL components. In the temperature range between 175 and 250 °C, the IRSL initial intensity is dominated by the fast OSL component. Estimated equivalent doses using either Post-IR<sub>175</sub>.OSL<sub>110</sub> as well as IRSL<sub>175</sub> (with the indices indicating the measurement temperature) are in good agreement between each other, due to both stimulating quartz. Finally, the physical meaningfulness of the fitting parameters for the tunnelling component is also discussed.

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# 1. Introduction

Optically Stimulated Luminescence (OSL) has been established as a promising dosimetric technique in various fields, such as medical, environmental, personal, space and retrospective dosimetry (McKeever, 2001; Bøtter-Jensen et al., 2003; Yukihara and McKeever, 2011). OSL is based on the fact that naturally-occurring minerals like quartz and feldspar act as natural dosimeters and preserve a record of irradiation dose, i.e. energy per unit mass, received through time. This record (namely the dose) could be obtained by illuminating the sample with visible light inside the laboratory.

While applying dating protocols, OSL is usually measured during optical stimulation at steady stimulation power at a specific elevated temperature, resulting in a decaying, continuous wave OSL

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(CW-OSL) curve. The age is usually determined by using the initial part of the CW-OSL signal minus a background based on the signal level at the end of the stimulation period. Thus, this net initial signal includes contributions from all fast, slow and medium quartz OSL components, according to the terminology earlier adopted by Smith and Rhodes (1994) and Bailey et al. (1997).

Therefore, it is desirable to use a well-separated fast OSL component in equivalent dose routines. It has been shown that for the case of quartz, the signal of each component can be separated from others by analytical or instrumental procedures, such as curve fitting and/or linearly modulated OSL (LM-OSL) (Bulur, 1996; Jain et al., 2005; Polymeris et al., 2006; Kitis and Pagonis, 2008). The components of a CW-OSL from quartz can also be separated by using the temporal duration of each component where not only the intensity but also the duration of each component can be assessed (Chithambo and Galloway, 2001). Nevertheless, in some cases these procedures were proved to be time-consuming and model dependent. In the case of contaminated quartz samples or polymineral materials, the situation becomes even more complicated. Besides







separating the signal of each component, it is also important to separate the signals from the two ubiquitous natural luminescent dosimeters, namely quartz and feldspar, since they record different doses. This difference in estimated dose could be attributed to a number of reasons, such as (i) the internal dose rate, which is present in the case of feldspathic minerals (ii) different sensitivities, (iii) differences in response to irradiation and/or (iv) the residual dose due to incomplete zeroing of the luminescence signal prior to deposition (Ankjærgaard et al., 2010). Nevertheless, the most important reason lies behind the fact that the dose measured using luminescence is usually under-estimated in feldspars because of the well-known anomalous fading phenomenon (Wintle, 1973; Aitken, 1998; Huntley and Lamothe, 2001).

The first instrumental approach towards discriminating between the signals arising from quartz and feldspars was suggested by Hütt et al. (1988). These authors indicated the preferential depletion of the signal arising from feldspar by applying Infrared (IR) stimulation at ambient temperatures. Moreover, Godfrey-Smith et al. (1988) indicated this latter stimulation mode could not stimulate quartz. Another instrumental procedure towards the isolation of fast OSL component was suggested by Jain et al. (2005). These authors have demonstrated that in the temperature range between 120 and 190 °C it is possible to preferentially deplete the fast component only using Infrared stimulated luminescence (IRSL) at 880 nm (FWHM 45 nm). Following these results, Polymeris et al. (2008) have demonstrated that IR stimulation at temperatures above 50 °C does not deplete only the fast component in most sedimentary quartz samples studied. According to their approach which also included de-convolution, the net depletion of fast and medium components resulting from IR exposure is sampledependent and occurs faster as the stimulation temperature gets higher (Polymeris et al., 2008).

Of course, there is always the option for either physical or chemical isolation between these two, ubiquitously present minerals (Wintle, 1997). The standard protocol towards this aim is time consuming, including extended heavy liquid separation as well as pre-treatments with acids; however, in many cases, physical separation of the minerals is not always possible, due to a variety of reasons such as the presence of feldspar micro-inclusions inside quartz grains. Nevertheless, in many cases the chemical treatments affect also the quartz grain quantity, size and properties as well.

Consequently, major effort has been devoted in reducing feldspar contamination, by using mostly three different experimental approaches: (a) the instrumental method for isolating a quartz signal from a mixed quartz-feldspar sample based on pulsed optical stimulation (POSL). This approach relies on the fact that the shapes of the time-resolved OSL (TR-OSL) of quartz and feldspar are very different (Chithambo and Galloway, 2000; Denby et al., 2006), (b) an elevated temperature IR stimulation prior to CW-OSL measurement (Jain and Singhvi, 2001). This approach, which was referred to as IR bleaching, has been applied by a number of authors (Wallinga et al., 2002; Kiyak and Erturac, 2008), despite the fact that may present difficulties because at high temperatures both the fast and the medium components in quartz OSL are depleted significantly by IR exposure (Jain et al., 2003). Finally, Thomsen et al. (2008) and Ankjærgaard et al., 2010 reported the effective application of a combination of post-IR pulsed blue stimulation with previous IRSL stimulation at ambient temperatures and 175 °C, respectively.

The present work provides an analytical study of the impact of this IR stimulation at elevated temperatures prior to OSL measurement to the fast OSL component of the natural signal, for a wide range of stimulation temperatures. Our approach includes a combination of both instrumental as well as analytical procedures, since all OSL and IRSL decay curves were de-convolved into their individual components. Implications for dating will also be discussed in the framework of the existing models.

#### 2. Materials and method

#### 2.1. Sample origin

The sample subjected to the present study was natural quartz of sedimentary origin that was collected from a fault line in Kütahya-Simav, the Aegean Anatolia region, Turkey. The preparation of samples was formed under red light conditions. After sieving, grains of dimensions 90–180 µm were obtained. These grains were treated with HCI (10%), H2O2 (35%), HF (40%, 45-60 min of handling) and a final treatment with HCI (10%) in order to obtain a clean quartz extract. Aliquots with mass of 2 mg each were prepared by mounting the material on stainless-steel disks. All aliquots were checked with infrared (IR) stimulation (880 nm) at ambient temperature to ensure the absence of feldspars. The quartz, however, was proven to be contaminated with feldspars, even after the application of the chemical preparation procedure towards extraction a clean guartz extract. This result was yielded by comparison of the IRSL (at RT) and Blue OSL (at 110 °C) signals, which are plotted in Fig. 1. As this Fig. reveals, these two signals provide ratio of IRSL over Blue OSL for the corresponding initial intensities of the order of 1.5. Therefore the contribution of the feldspar contamination, even after chemical procedures is considerably adequate when compared to the blue signal.

#### 2.2. Apparatus and measurement conditions

All luminescence measurements were carried out using a Risø TL/OSL reader (model TL/OSL-DA-20), equipped with a  ${}^{90}$ Sr/ ${}^{90}$ Y beta particle source, delivering a nominal dose rate of 0.130 ± 0.004 Gy/s. A 9635QA photomultiplier tube was used for light detection. The stimulation wavelength is 470 (±20) nm for the case of blue stimulation, delivering at the sample position a maximum power of 40 mW cm<sup>2</sup>. For IRSL, the stimulation wavelength is 875 (±40) nm and the maximum power of ~135 mW cm<sup>2</sup> (Bøtter-Jensen et al., 1999a; Bøtter-Jensen et al., 1999b). The detection optics consisted of a 7.5 mm Hoya U-340 filter ( $\lambda_p \sim$  340 nm, FWHM ~ 80 nm). All TL measurements were performed in a nitrogen atmosphere with a low constant heating rate of 2 °C/s, in order to avoid significant temperature lag; for the case of TL the samples were heated up to the maximum temperature of



Fig. 1. IR check at room temperature after chemical procedures.

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