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Research paper

Exploring fading in single grain feldspar IRSL measurements

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ABSTRACT

IR laser stimulated luminescence was used to determine the equivalent dose (D_e) for about 150 luminescent K-feldspar grains of two well-bleached aeolian sand samples, previously dated using quartz optically stimulated luminescence (OSL) and thermally-transferred OSL (TT-OSL). Fading tests were performed both on the entire single grain disc, using stimulation by IR LEDs, and on each grain individually, by stimulation with an IR laser. The single grain D_e distributions are highly over dispersed, even after applying rejection criteria, such as recycling ratio and recuperation. Fading rates determined with the IR LED stimulation do not represent the fading behavior of the IR laser stimulated signal and are therefore considered inappropriate for the fading correction of single grain D_e values. Fading rates of individual grains exhibit a wide range of values (0%–10% per decade) and plotting the single grain fading rates against the single grain D_e values reveals a negative relationship. Different approaches were taken to correct for fading, but the resulting ages tend to underestimate the reference ages.

1. Introduction

Using single grains of quartz for optically stimulated luminescence (OSL) dating is an important tool for dealing with partial bleaching in sediments (e.g., Roberts et al., 1998; Olley et al., 2004; Duller, 2006). However, for some geological regions, the quartz OSL signal shows anomalies that make it unsuitable for dating, for example, by the presence of unstable signal components (e.g. Steffen et al., 2009). Quartz has also been shown to underestimate the true deposition age of samples older than ca. 100 ka (Timar et al., 2010; Lowick et al., 2010). For the cases where partially bleached sediments have to be dated and the quartz OSL signal appears to be problematic (e.g. terrace sediments in Peru, Steffen et al., 2009), the infrared stimulated luminescence (IRSL) signal of single K-feldspar grains could offer an alternative. However, the IRSL signal of feldspar is often affected by fading, an athermal signal loss over time which leads to age underestimations (Wintle, 1973; Spooner, 1994). For single aliquot measurements two approaches are available to overcome this problem. One is to quantify the loss of the laboratory-induced IRSL signal per time (g-value) and apply a correction to the determined ages (Huntley and Lamothe, 2001; Lamothe et al., 2003). A more recent strategy focuses on isolating a luminescence signal that displays less fading, such as the elevated temperature IRSL signal following IR stimulation (post-IR IRSL; Thomsen et al., 2008; Buylaert et al., 2009; Murray et al., 2009). These papers found that the level of fading decreased with increasing stimulation temperature and so reduces the age correction factor required. While Buylaert et al. (2009) measured residual doses of only a few Gy, other studies have recorded residuals of between 40 and 100 Gy, and this severely limits the applicability of post-IR IRSL dating (Stevens et al., 2011; Lowick et al., 2012). Low temperature IRSL is expected to be the more appropriate approach for the dating of fluvial and glacial sediments, where partial bleaching is a common problem.

The use of single grain K-feldspar dating was first introduced by Lamothe et al. (1994). These authors were able to detect partial bleaching in their investigated sample, but the well-bleached grains lead to an age underestimation, possibly due to fading. Lamothe and Auclair (1999) developed the “fadia” protocol, which takes advantage of different fading rates of single feldspar grains and allows for extrapolation to zero fading. Nonetheless, the applicability of this protocol is complicated by high measurement uncertainties for dim grains and is limited to well-bleached grains (Lamothe and Auclair, 2000). Duller et al. (2003) were the first to test single grain feldspar measurements with the Risø single grain attachment equipped with an IR laser. They showed that K-feldspar grains are more uniform in their luminescence intensity and in their dose–response characteristics than single grains of quartz, but that there was a need for routine procedures for measuring anomalous fading, and the potassium content of individual grains.

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In this study, we explored the potential to measure and correct for fading when carrying out single grain IRSL measurements. The motivation behind this is to work towards a robust method to date partially bleached sediments containing quartz that is not suitable for dating and therefore K-feldspar has to be used. In this study, as a first step, test measurements were performed on well-bleached samples. Single grain equivalent dose (D_e) values for two aeolian samples previously dated by quartz OSL and thermally-transferred OSL (TT-OSL) were determined, and the results of fading tests performed on the entire single grain disc (IR LED stimulation) as well as on each grain individually (IR laser stimulation), are compared. Additionally, g-values were determined for synthetic aliquots produced from the single grain dataset. The obtained single grain D_e values were corrected using the different g-values and are compared to the reference ages.

2. Samples and measurement setup

Two aeolian sand samples from beneath palaeolake deposits in the south-western Rub' al Khali (Saudi Arabia) were selected for this study (Rosenberg et al., 2011a). Sample 22.4 has a minimum quartz OSL age of 89 ± 15 ka and a quartz TT-OSL age of 99 ± 11 ka; for sample 26.3 a minimum quartz OSL age of 123 ± 12 ka and a quartz TT-OSL age of 136 ± 14 ka were determined. Details of measurement conditions are given in Rosenberg et al. (2011b). All K-rich feldspar extracts were prepared by sieving the desired grain size (200–250 μm), followed by HCl and H_2O_2 treatment, and subsequent density separation of grains <2.58 g cm^{-3}. For the single grain measurements, K-rich feldspar grains were mounted on aluminum discs that can hold 100 grains each in 300 μm diameter holes (Bøtter-Jensen et al., 2003). The discs were then checked using a binocular, to ensure that every hole contained only one grain. Luminescence measurements were carried out on a Risø DA-20 TL/OSL single grain reader fitted with an internal $^{90}Sr/^{90}Y$ beta-source. To account for possible spatial non-uniformity of the dose rate each position on the single grain disc was individually calibrated (Thomsen et al., 2005; Ballarini et al., 2006) (Fig. S1). IRSL of single grains was stimulated using a 140 mW 830 nm IR laser with a RG-780 filter to attenuate the second harmonic frequency (415 nm). For all measurements a laser power of 90% was used. IRSL of the entire single grain discs was stimulated using light emitting diodes (LEDs) at 870 nm. Detection of IRSL was in the blue region of the spectrum, through an L.O.T.-Oriel D410/30 nm interference filter and a Schott BG-39.

A modified SAR protocol was used to determine the D_e, applying the same preheat before regeneration dose and test dose measurement (Blair et al., 2005). The chosen preheat was 60 s at 260 °C and IRSL measurements were carried out at 50 °C for 5 s. After 1 s of stimulation the signal is reduced to a background level, and the signal integral over the 1st second minus a background averaged over the last 2 s was chosen for D_e determination. This is appropriate as we assume that the IRSL signal of feldspar is not composed of different components with different bleaching properties, as is the case for the OSL signal of quartz (Hütt et al., 1988; Trautmann et al., 2000; Poolton et al., 2002). Due to a shortage of material, dose recovery tests were performed on K-feldspar grains of a sample from the same environment (sample 25.3 from the Rub' al-Kali, TT-OSL age of 144 ± 9 ka). This sample was bleached for 48 h using a Sunlux Ambience UV lamp, given a dose of 114 Gy in the luminescence reader with the internal beta-source, and measured using the SAR protocol with a test dose of 44 Gy.

Fading tests were carried out on the entire single grain discs (IR LED stimulation for 300 s) and on individual grains (IR laser stimulation for 5 s). The fading tests were based on repeated L_x/T_x measurements (114 Gy for L_x and 44 Gy for T_x) with different storage times between irradiation and the IRSL measurements, and with preheats performed prior to storage (Auclair et al., 2003). Measurement parameters were the same as for D_e determination, and storage times between 20 min and 30 h were chosen. The sensitivity corrected signals measured after different delays were plotted against the time delay between irradiation and IRSL measurement on a log scale, and the g-value was determined calculating the % of signal loss per decade (Huntley and Lamothe, 2001). Single grain D_e values were corrected for fading following Huntley and Lamothe (2001). Mean D_e values were calculated using the Central Age Model (Galbraith et al., 1999). Dose rates were calculated using the dosimetric data given in Rosenberg et al. (2011a) and assuming a K-content of the feldspars of 12.5 ± 5% (Dütsch and Krbetschek, 1997; Huntley and Baril, 1997).

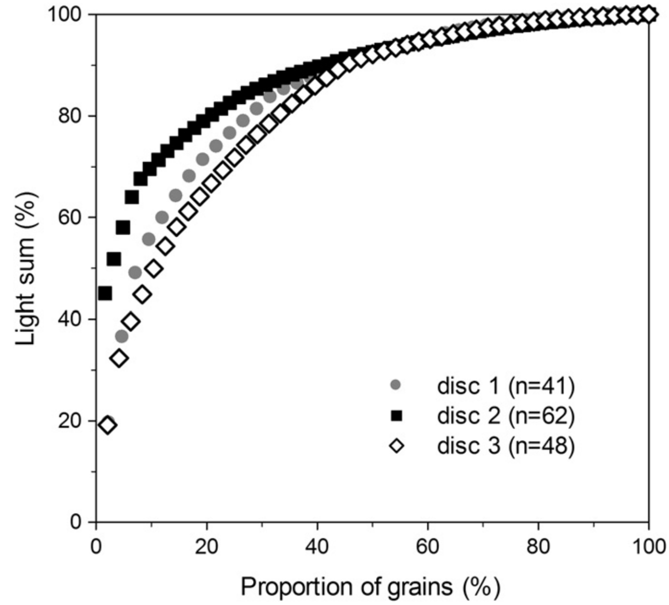

Fig. 1. Luminescent grains of three single grain discs of sample 26.3 ranked in order of descending brightness, and their cumulative light sum plotted as a function of the proportion of the brightest grains involved (following Duller et al., 2000). For all three discs the brightest 20% of the grains contribute to >60% of the light sum.

3. Results and discussion

3.1. D_e distribution and rejection of grains

Three single grain discs per sample were measured (300 grains) of which about 50% gave a signal >3 sigma above background, and further analyses are restricted to these grains. The signal intensity of the grains is highly variable. In Fig. 1, the cumulative IRSL light sum is plotted as a function of the proportion of the brightest grains for the three different discs of sample 26.3. Although the contribution is more uniform than in quartz (Duller et al., 2000), on each disc measured for sample 26.3, the three brightest grains are responsible for about 50% of the total light sum. The D_e distribution before rejecting any grains is very broad (Fig. 2a). After rejecting all the grains with recycling ratios >10% outside unity, the number of grains and the scatter of the dataset is only slightly reduced (Fig. 2b). A large number of grains exhibit high recuperation values, which are given by the ratio of the sensitivity corrected signal observed for zero dose to the natural sensitivity corrected signal. Fig. 3 explores the relationship between recuperation and D_e values. Where recuperation is above 10%, small D_e values seem to be related to high recuperation values, but there is no clear trend