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Research paper

Construction of a 'global standardised growth curve' (gSGC) for infrared stimulated luminescence dating of K-feldspar

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ABSTRACT

We investigated the infrared stimulated luminescence (IRSL) and post-infrared IRSL (pIRIR) signals emitted by K-feldspars from sedimentary samples from Asia, Europe and Africa using a single-aliquot multiple elevated temperature (MET) stimulation procedure. For separate aliquots of the same sample, we show that variation among the dose response curves (DRCs), or growth curves, constructed from the regenerative dose signal (L_x), the test dose signal (T_x, an indicator of luminescence sensitivity) and the sensitivity-corrected signal (L_x/T_x) can be largely eliminated by normalising the DRCs using one of the regenerative dose signals; we call this procedure 'regenerative-dose normalisation' or re-normalisation. Furthermore, for the MET-pIRIR signals measured at 250 °C, we find that different samples have re-normalised DRCs that follow the same growth function, despite the samples differing significantly in terms of their geological provenance, sedimentary context, equivalent dose (D_e) and luminescence sensitivity. This common feature offers the potential to establish a 'global standardised growth curve' (gSGC) for different samples of K-feldspar, and thereby enable D_e values to be estimated for a large number of single aliquots by projecting the re-normalised natural signals on to the gSGC. For the 18 samples investigated in this study, we find that D_e estimates obtained from the gSGC are consistent with those obtained using full single-aliquot regenerative dose (SAR) procedures for doses of up to ~1600 Gy. The establishment of a gSGC would greatly reduce the time required to date older samples using K-feldspar, as regenerative doses of several hundreds to a few thousands of Gy are typically delivered to each aliquot in each SAR cycle.

1. Introduction

The single-aliquot regenerative dose (SAR) procedure (Galbraith et al., 1999; Murray and Wintle, 2000) has been widely adopted for optically stimulated luminescence (OSL) dating of quartz, and applied subsequently to infrared stimulated luminescence (IRSL) dating of potassium-rich feldspar (K-feldspar) (Wallinga et al., 2000). In the SAR procedure, a test dose is applied and the induced luminescence measured after stimulating the natural dose signal (L_n) and each of the regenerative dose signals (L_x). The corresponding test dose signals (T_n, T_x) are used to monitor and correct for any sensitivity changes induced by the laboratory treatments, which include irradiation, preheating and optical stimulation. The sensitivity-corrected signals for a series of regenerative doses (L_x/T_x) are used to construct a dose response curve (DRC), and the equivalent dose (D_e) is determined by projecting the sensitivity-corrected natural signal (L_n/T_n) on to the DRC.

Based on 6 samples of quartz from Australia and polymineral grains of loess from China, Roberts and Duller (2004) suggested that T_x not only corrected for sensitivity changes but could also be used for between-aliquot normalisation, thereby allowing a direct comparison among different aliquots of the same or different samples. This formed the basis for establishing a standardised growth curve (SGC). Roberts and Duller (2004) suggested that, once a reliable SGC had been established, D_e values may be estimated accurately based solely on measurements of L_n and T_n, thus offering a convenient means of saving on instrument time when D_e estimates are required for a large number of aliquots and/or for dating old samples.

The SGC approach was subsequently tested and applied successfully to quartz samples from a variety of regions, including agricultural and archaeological deposits in Scotland (Burbidge et al.,

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2006), loess from China, Germany and the Great Plains in the USA (Lai, 2006; Lai et al., 2007), sand dunes in South Africa and Florida in the USA (Telfer et al., 2008), lacustrine samples from the Qinghai–Tibetan Plateau in China (Long et al., 2010) and fluvial deposits from the Mississippi Valley in the USA (Shen and Mauz, 2011). The SGC method was further improved by Li et al. (2015), who suggested that normalisation of the DRCs using one of the regenerative doses—the so-called regenerative-dose normalisation (or re-normalisation) method—can further reduce the between-aliquot and between-sample variation among quartz DRCs. They tested the re-normalisation procedure on single aliquots of Asian, African, European and North American quartz and found that many of the samples followed a similar DRC function up to a dose of ~250 Gy, despite differing significantly in terms of their geological provenance, environmental setting and depositional age.

Compared to quartz OSL, however, there has been little study on the extension of the SGC method to the K-feldspar IRSL signal, except for the polymineral IRSL signals of Chinese loess examined by Roberts and Duller (2004). Recently, Li et al. (2014a) reported that K-feldspar samples from different regions of Eurasia exhibited a dose-dependency in their post-IR IRSL (pIRIR) signals and that these samples appeared to share a common DRC, hence suggesting the possibility of developing a 'global standardised growth curve' (gSGC) for K-feldspar. Potassium-rich feldspars have several advantages over quartz as a luminescence dosimeter, including a higher luminescence sensitivity and saturation dose limit, so there are benefits in exploring the potential to establish a gSGC for feldspars. It would prove especially useful for dating of older samples, as each SAR cycle involves the application of regenerative doses of up to several thousand Gy for each aliquot.

In this study, we examine the IRSL and pIRIR signals emitted by K-feldspar grains in sedimentary samples collected from sites in Asia, Europe and Africa. We combine a single-aliquot multiple elevated temperature (MET) stimulation procedure (Li and Li, 2011; Li et al., 2014a) with the re-normalisation method proposed for quartz by Li et al. (2015), and find that this greatly reduces between-aliquot variation in DRC shape for separate aliquots of K-feldspar from the same and different samples. By developing a gSGC for K-feldspar using the MET-pIRIR signal measured at 250 °C—which has previously been shown to fade negligibly (Li and Li, 2011)—we propose a practicable means of making rapid and reliable measurements of D_e for a large number of aliquots, which will be particularly advantageous for dating of Middle Pleistocene samples.

2. Sample descriptions

Eighteen sediment samples were examined in this study to assess the variability in the properties of K-feldspars from different parts of Asia (China, Georgia, India and Indonesia), Europe (France and Italy) and Africa (Libya and Kenya). Sample locations are shown in Fig. 1, and the depositional contexts, experimental measurement conditions, and D_e ranges are summarised in Table 1. Loess samples LC-110, -230 and −270 were taken from the Luochuan section on the Chinese Loess Plateau, and have been used previously for testing the MET-pIRIR method (Li and Li, 2012a, 2012b). Aeolian sand samples Sm0404, 5, 6' and 8 were collected from the Shimao section on the southeastern margin of the Mu Us Desert in central China (Sun et al., 1999), and have been used previously to investigate dose-dependent sensitivity changes (Li et al., 2013b, 2014a). Samples MTL-OSL7 and DGT-OSL1 are lacustrine sediments from the Nihewan Basin in China; TUT-OSL1 and -OSL2 are alluvial sediments from Talepu in southwest Sulawesi, Indonesia; RUP-2 and -4 are samples of alluvium from Rusinga Island, Kenya (Tryon et al., 2010); and HF12-6023 is an aeolian–colluvial sample taken from the Haua Fteah cave in northeast Libya (Douka et al., 2014). DHB2-OSL4 and PIN-OSL2 consist of colluvial and alluvial sediments deposited at the open-air archaeological sites of Dhaba in north–central India and Pinavera in the southern Caucasus, Georgia, respectively. CEP-OSL1 was collected from a colluvial–alluvial palaeosol sequence at Ceprano, Italy, and LC10-16 from a collapsed cave at Les Cottés, France (Jacobs et al., 2015). The latter four samples have also been investigated in our previous MET-pIRIR studies (Li et al., 2013a, 2014a).

3. Experimental procedures and analytical facilities

Samples were prepared for IRSL analysis using routine procedures (Aitken, 1998). First, the samples were treated with HCl acid and H_2O_2 solution to remove carbonates and organic matter, respectively, and then dried and sieved to obtain grains of 63–90, 90–125, 125–180, 150–180 and 180–212 μm in diameter. K-feldspar grains were separated from quartz and heavy minerals using a solution of sodium polytungstate with a density of 2.58 g/cm^3. The separated K-feldspar grains were immersed in 10% HF acid for 40 min to etch the surfaces of the grains and remove the outer, alpha-irradiated portions, and then rinsed in HCl acid to remove any precipitated fluorides. After drying, the etched K-feldspar grains were mounted as a monolayer on stainless steel discs of 9.8 mm diameter using "Silkospray" silicone oil as an adhesive. Grains covered the central ~5 mm diameter portion of each disc, corresponding to several hundreds to thousands of grains per aliquot.

IRSL measurements were made on an automated Risø TL-DA-20 reader equipped with IR-emitting diodes for stimulation (870 Δ 40 nm). The total IR power delivered to the sample position was ~135 mW/cm^2 (Bøtter-Jensen et al., 2003) and laboratory irradiations were carried out on the reader using a calibrated ^{90}Sr/^{90}Y beta source. IRSL signals were detected by an Electron Tubes Ltd 9235B photomultiplier tube fitted with Schott BG-39 and Corning 7-59 filters to restrict transmission to 320–480 nm. Each IR stimulation was made for 100 s (Table 2), and the resulting signal was calculated as the sum of counts over the initial 5 s of measurement, with 'late light' subtraction (Aitken, 1998) of the background count rate over the final 5 s of stimulation. Prior to each IRSL measurement, an 'IR-off' period of up to 50 s was applied to minimise interference from the isothermal decay signal (Fu et al., 2012).

Samples were measured using the single-aliquot 'pre-dose' MET-pIRIR (pMET-pIRIR) procedure proposed by Li et al. (2014a) and outlined in Table 2. This procedure is similar to the standard MET-pIRIR procedure of Li and Li (2011), except that a 2 h solar simulator bleach is given at the end of each SAR cycle, instead of a high-temperature IR bleach. The purpose of the solar bleach is to reset the dose-dependent sensitivity of the MET-pIRIR signals, based on the observations by Li et al. (2013b) that the sensitivity of these signals is reduced to a stable level after a 2 h bleach using a UVACUBE 400 solar simulator. Li et al. (2014a) found that this bleach effectively reset the dose-dependent sensitivity between successive SAR cycles and that, consequently, both L_x and T_x signals could be used for D_e determination.

Table 1 lists the preheat temperatures, MET-pIRIR stimulation temperatures and sizes of test dose given to each of the study samples. Preheats were made at either 300 °C for 60 s or, for four of the samples, at 320 °C for 60 s, with the same preheat applied to the natural, regenerative and test doses. The MET-pIRIR signals were measured by stimulating with IR at successively higher temperatures, from 50 to 250 °C in steps of 50 °C. In addition, for the four samples preheated to 320 °C, an extra MET-pIRIR stimulation temperature of 280 °C was included. The size of the test dose ranged from 24 to 66 Gy. We show later that the shape of the DRCs