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Sources of overdispersion in a K-rich feldspar sample from north-central India: Insights from D_e, K content and IRSL age distributions for individual grains

C.M. Neudorf*, R.G. Roberts, Z. Jacobs

Centre for Archaeological Science, School of Earth and Environmental Sciences, University of Wollongong, Northfields Avenue, Gwynneville, NSW 2522, Australia

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Luminescence dating of individual sand-sized grains of quartz is a well-established technique in Quaternary geochronology, but the most ubiquitous mineral on the surface of the Earth—feldspar—has received much less attention at the single-grain level. In this study, we estimated single-grain equivalent dose values and infrared stimulated luminescence (IRSL) ages for K-rich feldspar (KF) grains from a fluvial sample underlying Youngest Toba Tuff (YTT) deposits in north-central India, and compared these ages (corrected for anomalous fading) with those obtained from individual grains of quartz from the same sample. Both minerals have broadly similar single-grain age distributions, but both are greatly overdispersed and most grains have ages substantially younger than the expected age of the YTT deposit (~74 ka). Almost half (45%) of KF grains used for age calculation have fading rates statistically consistent with zero, but the age distribution of these grains is as dispersed as that of the entire population. We obtained a similar distribution of ages calculated for 51 grains using their individually measured internal K contents, which exhibited only minor grain-to-grain variation. Given the lack of dependency of single-grain ages on the measured fading rates and internal K contents, and the overall adequacy of bleaching of grains collected from a sandbar in the modern river channel, we consider the spread in ages is most likely due to mixing, at the time of deposition and after the YTT event, of potentially well-bleached fluvially-transported sediments with older grains derived from slumping of riverbank deposits. Some spread may also be due to natural variations in the IRSL properties of individual KF grains.

1. Introduction

Luminescence dating procedures that make use of multi-grain aliquots of quartz or feldspar, and in which more than one grain contributes significantly to the luminescence signal, implicitly assume that all contributing grains have suitable luminescence properties for dating and have been sufficiently bleached by sunlight before burial, and have not been mixed after burial. Single-grain optically stimulated luminescence (OSL) dating techniques have been available for quartz for more than a decade, enabling the identification of partially bleached grains or intrusive grains from overlying or underlying sedimentary units, as well as the rejection of grains with unsuitable characteristics for reliable dose determination using single-aliquot regenerative dose (SAR) procedures (Galbraith et al., 1999; Jacobs and Roberts, 2007). Similar single-grain dating techniques are rarely used for potassium (K)-rich feldspars but would also entail measurements of the internal dose rates (due principally to ^{40}K) and anomalous (athermal) fading rates of individual grains (Duller et al., 2003). Risø readers equipped with a 150 mW infrared (IR) (830 nm) laser allow for the direct stimulation of individual feldspar grains (Duller et al., 2003). In this paper, we describe the use of the IR laser to obtain single-grain equivalent dose (D_e) values and fading-corrected ages for K-rich feldspar (KF) grains from a fluvial sample collected from a sand unit underlying Youngest Toba Tuff (YTT) deposits in the Middle Son Valley, Madhya Pradesh, India. This study is part of a larger luminescence dating program to assess the time of deposition of the alluvial deposits and YTT ash in the Middle Son Valley. Experiments were conducted on two additional samples (one from the same geological section, and one from a modern sandbar in the Son River channel) to assess potential sources of overdispersion (OD) in the KF grain age distribution. The impact of single-grain fading rates and K contents on the KF single-grain age distribution is also examined.

2. Samples

IR stimulated luminescence (IRSL) measurements were made on three samples (GHO-2, GHO-3, and KHUT-10). Sample GHO-2 was

* Corresponding author. Tel.: +61 (0) 2 4221 4686; fax: +61 (0) 2 42214520.
E-mail address: cmn821@uowmail.edu.au (C.M. Neudorf).

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collected from a well-drained, medium-coarse fluvial sand unit that underlies YTT deposits in a cliff section on the north bank of the Son River (24° 30′ 7.608″ N, 82° 1′ 2.748″ E) (Jones, 2010). The YTT deposit at this location is thought to have been deposited ~74 ka ago (Jones, 2010; Gatti et al., 2011; Smith et al., 2011) but the ash here has not been dated directly. Sample GHO-3 was collected from a fine-medium sand unit immediately above the YTT deposit, and ~2 m above sample GHO-2. No carbonate concretions or organic matter was observed in the two sand units in the field. KHUT-10 was collected from a modern-day sandbar in the Son River channel, ~25 km upstream of the GHO section. See Supplementary Materials for further details on sample preparation, equipment and environmental dose rate calculations (Tables S1 and S2).

3. Testing a SAR protocol

3.1. Natural IRSL signals

Most KF grains (60–80%) from sample GHO-2 are characterized by bright initial signals (commonly greater than 40,000 counts in the first 0.134 s) that decay rapidly upon laser stimulation, but fail to reach a constant background (Fig. 1a). Approximately 20–40% of grains from sample GHO-2 exhibit dim and very slowly decaying, IRSL signals (Fig. 1a, inset). Elemental (microprobe) analyses (Section 6.4) show that the bright grains are most commonly orthoclase and some are plagioclase, and the dim IRSL signals are usually derived from holes occupied by quartz grains, both minerals identified on the basis of their elemental compositions. The IRSL signals detected from the holes containing quartz grains may be

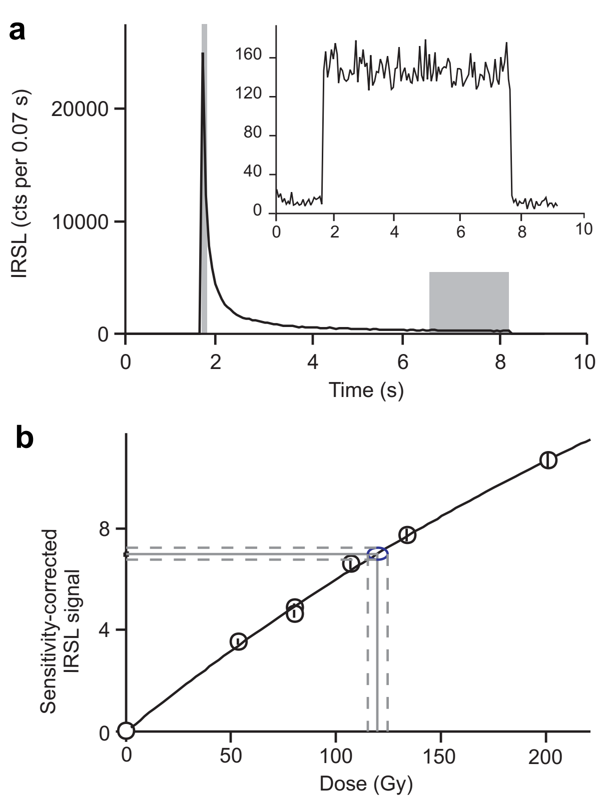

Fig. 1. a) Shine-down curves for a KF grain and a quartz grain (inset) from sample GHO-2. The integration limits for the initial (first 0.134 s) and background (last 1.742 s) signals are shaded in grey. b) Growth curve for the same KF grain. The KF grain has a K concentration of 12.2%, and the quartz grain is 99.5% SiO_2 (both measured using WDS; see text).

due to weak IRSL emissions from the quartz grains or feldspar inclusions inside them, or stray IRSL emissions from adjacent bright KF grains. Approximately 10% of measured feldspar grains have natural and/or regenerative signals greater than 268,000 counts per 0.134 s. These grains were excluded from further analyses as such intense signals induced partial saturation of the photomultiplier tube.

3.2. The SAR procedure and data rejection criteria

The D_e was measured using an IRSL SAR procedure similar to that described by Wallinga et al. (2007). The temperature and duration of the preheat (250 °C, 10 s) preceding the L_n and L_x measurements were identical to those preceding the T_n and T_x measurements, following Huot and Lamothe (2003) and all IRSL measurements were made while holding the sample at 50 °C (Table S3). At the end of each SAR cycle, all grains on each disc were bleached using IR LEDs for 40 s at 290 °C to reduce recuperation. A typical KF grain dose-response curve is shown in Fig. 1b.

KF grains were rejected if: 1) they exhibited an IRSL decay curve typical of holes occupied by quartz grains, 2) their signals failed to grow systematically with increasing regenerative dose (i.e., they had no dose-response curve curve), 3) the sensitivity-corrected zero-dose signal was greater than 5% of L_n/T_n, 4) the first 0.134 s of the test dose signal following the natural IRSL measurement was less than 3 times the background, 5) the recycling ratio differed from unity by more than 2σ (Jacobs and Roberts, 2007), and 6) their natural and/or regenerative signals exceeded 268,000 counts per 0.134 s (to prevent partial saturation of the photomultiplier tube). Signals were analysed using the 'Previous background (BG) subtraction' option in Analyst, but we note that this method results in D_e values that are negligibly smaller (by ~0.2%) than those obtained using the standard 'late light' subtraction procedure.

3.3. Dose recovery test

A single-grain dose recovery test was performed on KF grains from sample GHO-2 that were bleached in the sun for 2 days before measuring a laboratory beta dose of 68 Gy using the SAR procedure outlined in Table S3. Out of 590 measured grains, 232 (39%) passed all rejection criteria (Table S4). The distribution of measured dose/given dose ratios is shown in a radial plot in Fig. 2a. The weighted mean ratio is 0.97 ± 0.01 and the OD is 6.9 ± 0.5%. Both statistics were calculated using the central age model (CAM) of Galbraith et al. (1999). These dose recovery results suggest that the SAR protocol is appropriate for the majority of KF grains in this sample.

4. D_e determination and sources of overdispersion

The single-grain D_e distribution of sample GHO-2 is shown in Fig. 2b and the decay curves for two extreme values are plotted in Fig. 2c; the latter exhibit no obvious differences in shape. Of 1149 measured grains, 475 (41%) passed all rejection criteria (Table S4). The weighted mean (CAM) D_e of all accepted grains is 52.7 ± 1.6 Gy, with an OD of 46.5 ± 1.5% (Table 1). The OD and relative spread in values of this sample are much larger than those obtained in the dose recovery test, which we attribute to a number of factors, including:

1) natural variations in luminescence properties of KF grains additional to those observed in the laboratory (as bleaching and irradiation conditions in nature are not identical to those used in the dose recovery test),
2) heterogeneous external beta microdosimetry after burial,