Quaternary Geochronology 6 (2011) 71–81

Contents lists available at ScienceDirect

Quaternary Geochronology

journal homepage: www.elsevier.com/locate/quageo

Research Paper

Infrared stimulated luminescence measurements of single grains of K-rich feldspar for isochron dating

Bo Li [a], Sheng-Hua Li [a,*], Geoffrey A.T. Duller [b], Ann G. Wintle [b]

[a] Department of Earth Sciences, The University of Hong Kong, Pokfulam Road, Hong Kong, China
[b] Institute of Geography and Earth Sciences, Aberystwyth University, Aberystwyth SY23 3DB, UK

A R T I C L E I N F O

Article history:
Received 4 February 2009
Received in revised form
2 February 2010
Accepted 11 February 2010
Available online 23 February 2010

Keywords:
K-feldspar
IRSL
Single grain
Luminescence
Recuperation
Isochron
Dating

A B S T R A C T

This paper explores the use of single grain luminescence measurements in isochron dating of K-rich feldspars. The thermal stability of individual feldspar grains was investigated using pulse annealing methods, which appears to distinguish between K-rich and Na-rich feldspars. A good isochron fit was obtained using synthetic aliquots produced from the single grain data set and the age obtained based on an assumed K content of 13 ± 1% was in good agreement with that obtained using single aliquot measurements (and with other age control).

1. Introduction

A method of dating has been proposed recently in which the infrared stimulated luminescence (IRSL) of potassium-rich feldspar is measured (Li et al., 2007, 2008a,b), the isochron IRSL (iIRSL) method. In this method, the equivalent doses (D_e) for grains of different diameter were determined using the IRSL signal. Isochrons were constructed by plotting the D_e values as a function of the internal dose rates. The method is similar to the subtraction method proposed by Fleming and Stoneham (1973) for thermoluminescence signals from quartz and feldspar and to more recent isochron methods (e.g. Mejdahl, 1983; Clark, 1994; Zhao and Li, 2002).

Any isochron dating method is critically dependent on the accuracy and precision of the data, and the iIRSL method is no exception. The IRSL measurements were made on aliquots made up of several thousand grains that had been extracted by heavy liquid separation (selection of grains lighter than 2.58 g cm^{-3}) after sieving to obtain the relevant grain sizes; the age was determined from the slope of the best-fit line. It was assumed that this preparation procedure resulted in potassium-rich grains, enabling the internal dose rate to be estimated using an assumed content of 13 ± 1% (Huntley and Baril, 1997; Zhao and Li, 2005; Li et al., 2008a). The accuracy and precision of the D_e values obtained for a single grain size fraction will depend upon whether the IRSL signals are indeed mainly from KF or from some other feldspars (e.g. sodium-rich feldspar, NaF) that are present.

In the work reported here, single grain measurements have been used to investigate variations in thermal stability and other luminescence characteristics at a smaller observation scale and assess the potential for improving precision in isochron dating by the use of synthetic aliquots. These aspects are investigated by making IRSL measurements on single grains. The presence of NaF grains in the lighter than 2.58 g cm^{-3} fraction is determined by pulse annealing experiments, as previously used by Tso et al. (1996) to determine the relative thermal stability of KF and NaF. The impact of the presence of NaF grains on the D_e obtained using a multiple grain aliquot is ascertained by measuring the D_e values for several hundred individual grains. The potential of using single grain measurements and appropriate rejection criteria to isolate KF grains, and therefore improve the precision of D_e determination, is also explored.

2. Samples and experimental procedures

Two sedimentary sand samples from desert areas in Northern China were selected for this study. Sm1 was from below the

* Corresponding author.
E-mail address: shli@hku.hk (S.-H. Li).

1871-1014/$ – see front matter © 2010 Elsevier B.V. All rights reserved.
doi:10.1016/j.quageo.2010.02.003

Holocene soil at the Shimao section in the transition zone between the Mu Us Desert and the Loess Plateau. It was chosen since an optically stimulated luminescence (OSL) age of 9.1 ± 0.5 ka had been obtained on quartz grains from this sample (Li et al., 2008b). The sediment grains were thus considered to be well bleached at deposition. Support for the efficiency of light exposure at deposition was provided by an age of ~200 a being obtained for KF grains from a modern dune in the same area (unpublished data). The iIRSL method had been applied to multiple grain aliquots of the KF fraction for sample Sm1 (Li et al., 2008b). HLD3 was an aeolian sand from the He Er Hong De section in the Hulun Buir Desert and was selected to provide a second sample upon which to test the pulse annealing procedures. It had a quartz OSL age of 10.8 ± 1.2 ka (Li et al., 2002).

Heavy liquid separations were carried out at 2.58 and 2.62 g cm^{-3} on grains from which carbonates and organic material had been removed. The KF fractions (density <2.58 g cm^{-3}) of different grain sizes (90–125, 125–150, 150–180, 180–212 and 212–250 μm diameter) were used for single grain IRSL measurements which were carried out using a single grain system (Duller et al., 2003) attached to a Risø TL/OSL reader. The KF grains were etched 40 min with 10% hydrofluorite acid before IRSL measurements. The KF grains were stimulated using an IR laser (830Δ10 nm, 400 W cm^{-2}) (Bøtter-Jensen et al., 2003a) and the IRSL signals were detected using a photomultiplier tube with the IRSL passing through a filter pack containing a Schott BG-39 and a Corning 7-59 filter. Single aliquot D_e measurements were made on each grain size of the KF fraction, but using IR emitting diodes (880 ± 80 nm, 150 mW cm^{-2}) as the stimulation source (Bøtter-Jensen et al., 2003b). Irradiations were carried out within the Risø reader using the $^{90}Sr/^{90}Y$ beta source.

In addition, 150–180 μm and 212–250 μm grains were separated using heavy liquids of densities 2.58 and 2.62 g cm^{-3}. The grains within this density range were assumed to be NaF. They were used together with the equivalent KF grains for studying the relative thermal stability of the IRSL signals from these two materials. The IRSL signals from each NaF grain were measured in the same way as for the individual KF grains. The efficiency of the heavy liquid separation of NaF and KF grains was tested by measuring the K content of the two fractions using the Risø GM-25-5 beta counter (Bøtter-Jensen and Mejdahl, 1988). The K content of the KF fraction was found to be in the range of 11.4–14.3% for different grain sizes of Sm1 and 10.2–12.2% for HLD3 (Li et al., 2008b), and the NaF fraction of HLD3 had a K content of 2.4 ± 0.2%. Given that the theoretical K content of a pure K-feldspar is ~14% (Huntley and Baril, 1997), the beta counting results indicated that the KF grains were concentrated in the fraction with a density that was less than 2.58 g cm^{-3}, while a range of feldspar compositions are likely to be present in the fraction with a density between 2.58 and 2.62 g cm^{-3}, but all sharing the common trait of a low K content.

3. Dose rate calibration for single grain measurement

There have been several studies on the uniformity of the dose rate from a $^{90}Sr/^{90}Y$ beta source for short source-to-target distances. Using single grains placed on a conventional aluminium disc, Spooner and Allsop (2000) showed that the dose rate decreased by as much as 10% from the centre of the target disc to 5 mm from the centre for their beta sources. For their readers, Veronese et al. (2007) and Ballarini et al. (2006) made a study of source uniformity using grains mounted in a single-grain disc. Therefore, it was essential for us to undertake calibration for individual holes in the single-grain disc in order to know the dose delivered to individual grains during the experimental procedures undertaken to obtain the equivalent dose. Only once this has been checked is it then possible to go on and obtain the dose rates for grains of different sizes. As in the case of measurement of aliquots containing several thousand grains, this calibration was carried out using quartz supplied by Risø National Laboratory, Denmark which they had extracted from a sediment collected from Jutland, annealed at 500 °C and then packed into a glass flat-pack prior to irradiating (5.10 ± 0.06 Gy) in the dark using a ^{137}Cs gamma source. The quartz was sieved to obtain fractions with diameters 90–125, 125–150, 150–180, 180–212 and 212–250 μm.

Two sets of measurements were undertaken. In the first, ten discs were measured using the single grain system to determine the dose rate specific to each grain position. This was undertaken to assess whether there was significant variability in the beta dose delivered to grains in different positions on the disc. A total of 1000 grains had their OSL signal (OSL detection through a U-340 filter) measured using the green-emitting laser (532 nm) in the same Risø TL/OSL reader. Of these, 981 passed the rejection criteria for the standard single aliquot regenerative dose (SAR) protocol (Murray and Wintle, 2000), giving between 7 and 10 calibration values for each position. The weighted mean was calculated for each position, and the standard error calculated as the uncertainty. These values were then used to calculate the central value of dose rates using the central age model (Galbraith et al., 1999). A small over-dispersion (OD) of 3.6% was obtained from the data set. This suggests that the spatial heterogeneity of the dose rate across the single-grain disc would bring a ~3.6% over-dispersion to all D_e distributions obtained using the same reader. To illustrate the spatial distribution of the dose rate across the single-grain disc, the grains with a dose rate inside and outside two sigma of the central value are shown in different colours in Fig. 1(a). It is shown that ~85% of the area has a similar dose rate, consistent within two sigma of the central value.

A second set of measurements was undertaken to precisely calculate the dose rate for the different grain sizes. To improve the precision, all grains on a single grain (SG) disc were measured simultaneously using blue LEDs (470 nm) as the stimulation light source, rather than the green (532 nm) emission from the laser. The standard SAR protocol (Murray and Wintle, 2000) was applied, and at least 10 discs for each grain size were measured to calculate the dose rate. Fig. 1(b) shows the experimentally determined dose rates as a function of the average grain diameter. The same dose rate was found for grain sizes from 90 to 250 μm when SG discs were used. Therefore, in this study of SG measurements, the same dose rate was applied to all grain sizes. It should be noted that the data in Fig. 1(b) differ from the results obtained previously using single aliquot (SA) discs; Li et al. (2007) found that the dose rate was slightly dependent on grain sizes in the range from 63 to 250 μm. This is probably due to the different irradiation geometry for grains lying on (SA) or within (SG) the two types of discs, e.g. different source-to-sample distance, different structure of discs and different grain position relative to the disc surface.

4. Thermal stability of IRSL signals

Previous studies have shown that IRSL signals from NaF are less thermally stable than those from KF (Li and Wintle, 1992; Tso et al., 1996) when the same detection window (blue-violet emission) is used for both NaF and KF. In comparison to the optically stimulated luminescence signals from quartz, the IRSL signals from feldspars do not come from a single trap. The IRSL signals are derived from a distribution of traps, each with a characteristic thermal stability; thus curve fitting cannot be used to obtain a thermal activation energy for the signal.

In this study, sample Sm1 gave a D_e value of 21.5 ± 1.5 Gy for single aliquot measurements on NaF (2.58 < ρ < 2.62 g cm^{-3}), a value which is significantly lower than that of 37.7 ± 0.5 Gy