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Isochron measurements of naturally irradiated K-feldspar grains

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Abstract

The equivalent doses of K-feldspar grains in a range of grain sizes from 90 to 250 μm diameter were measured using a single-aliquot regenerative-dose protocol for the infrared stimulated luminescence (IRSL) signals for two samples of desert sand. The equivalent doses for each sample were compared with that for the 125–150 μm grains of quartz from the same samples. The results suggested that the K-feldspar equivalent doses were underestimated because of anomalous fading. Measurements of the decay of the IRSL signals following laboratory irradiation for these two samples, and an additional one from a previously published isochron study, showed anomalous fading during the period of laboratory storage. The decay rate was about 3% per decade for all samples and was independent of the grain size used. Using plots of equivalent doses for K-feldspars as a function of their calculated internal dose rate, and the quartz equivalent dose as a function of grain size, it was concluded that the IRSL signal derived from the internal dose rate had not faded over the 13,000 years that had elapsed since the grains were deposited.

Keywords: K-feldspars; Infrared stimulated luminescence; Anomalous fading; Isochron

1. Introduction

During recent years, quartz has become the main mineral used for optical dating of sediments. The fast component of the optically stimulated luminescence (OSL) signal in quartz has been used for dating as it is rapidly bleached by sunlight and a method for measuring the dose using this signal from a single aliquot of sand-sized quartz grains has been developed (Murray and Wintle, 2000; Wintle and Murray, 2006). However, the fast component of the OSL signal has been shown to saturate at relatively low doses and this has made it difficult to use for dating beyond about 70,000 years (70 ka), unless the environmental dose rate is exceptionally low. Feldspars can also be used for optical dating, either using visible wavelengths for stimulation or using infrared stimulation to produce an infrared stimulated luminescence (IRSL) signal. The IRSL signal has been shown to continue to grow to higher dose levels than quartz OSL, and thus it would be advantageous to develop a method that is based on IRSL measurements of feldspars.

In the case of sand-sized potassium-rich feldspars grains (K-feldspars), a considerable portion of the dose will come from the decay of ^{40}K that makes up the lattice structure of the grain. Thus K-feldspars will receive proportionally less of their total annual dose from their environment than quartz grains of a similar size and the difference in dose rate can be used to advantage. Using thermoluminescence (TL) signals from K-feldspar grains of different sizes from a piece of pottery, as well as TL signals from quartz and plagioclase grains of the same size, Mejdahl (1983) constructed an isochron of measured equivalent dose as a function of the total beta dose rate. Using such a plot, the age can be derived from the slope of the line, and thus it is not necessary to know the environmental gamma dose rate. Such an approach would be advantageous when there is uncertainty in both the gamma and the external beta dose rate, for

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example, due to changing water contents during the burial period, disequilibrium in the uranium or thorium decay chains or when there has been a change in the cosmic dose rate due to build up of sediment above the sediment unit being dated. Eliminating the need to know the environmental dose rate was first suggested by Fleming and Stoneham (1973) when they obtained subtraction ages for pottery; they used the different equivalent doses for fine grained polyminerals and coarse grained quartz. However, this method is problematic if there is fading of the TL signal from feldspar grains in the polymineral fraction.

Zhao and Li (2002) proposed a similar method in which an isochron was constructed by plotting the measured equivalent dose (D_e) as a function of grain size. They measured both quartz and K-feldspar grains with different average grain sizes, ranging from 108 to 231 μm. In their study of a wind-blown sand, whose age had been determined as 13 ± 1 ka using the fast OSL signal from the quartz grains (Li et al., 2002), the feldspar IRSL ages were about 9 ka. This $\sim 30\%$ underestimation in age was ascribed to anomalous fading of the IRSL signals from the K-feldspars (e.g. Spooner, 1992, 1994; Huntley and Lamothe, 2001; Auclair et al., 2003; Huntley and Lian, 2006). In applying their isochron method, Zhao and Li (2002) assumed that the fading rate for the IRSL signals of their K-feldspars was the same for each of the different grain sizes and they calculated an isochron age of 29 ± 8 ka for this sample; this is too large when compared with the age of 13 ± 1 ka for the quartz grains. This age discrepancy suggests that their assumption concerning the rate of anomalous fading in nature may have been incorrect, or their measurements may have been in error.

In this paper, we investigate possible causes of this discrepancy for this sample and two others. We discuss the implications of the measured D_e values for the mechanism of anomalous fading in K-feldspars. The isochron measurements for three naturally irradiated K-feldspars are investigated alongside the results of laboratory measurements of anomalous fading.

2. The role of the potassium content

In their calculation of the internal dose rate, Zhao and Li (2002) used the potassium content of their K-feldspars as measured using atomic absorption spectroscopy for the grains that they had separated using a heavy liquid with a specific gravity of 2.58. The value of $9.63 \pm 0.2\%$ was low compared with the theoretical maximum of 14% for a pure K-feldspar with a structure of $KAlSi_3O_8$. This suggests that their separation was not perfect and that some sodium-rich and calcium-rich feldspars (plagioclase feldspars) would have been included in the density-separated grains used for IRSL measurement.

Huntley et al. (1991) concluded that the IRSL signal from imperfectly separated grains would be dominated by the IRSL signal from K-feldspar when using a transmission window composed of a Schott BG-39 and a Corning 7-59 filter (300–490 nm). This was supported by measurements of the TL spectra (Prescott and Fox, 1993; Sánchez-Muñoz et al., 2007), as well as IRSL spectra (Spooner, 1992; Baril and Huntley, 2003), in which higher sensitivity was found for feldspars with higher K content. Based on these observations, Huntley and Baril (1997) proposed that if any plagioclase feldspars were present, their lower luminescence sensitivity would cause them to have a negligible effect on the equivalent dose determined using IRSL signals. This led them to conclude that the use of a value of $12.5 \pm 0.5\%$ for the potassium content would be appropriate for dose rate calculations when IRSL measurements were made on grains separated using a heavy liquid with a specific gravity of 2.58.

In a subsequent study, Zhao and Li (2005) made measurements of the potassium content of 16 individual K-feldspar grains obtained by heavy liquid separation from two different sedimentary samples; using an electron micro-probe, they obtained a mean potassium content of $13.55 \pm 0.09\%$. The values were supported by ICP-MS measurements that also allowed the concentrations of rubidium to be determined. Zhao and Li (2005) concluded that for these grains, the measured K:Rb concentration ratio was 200:1 in accordance with the suggestion of Aitken (1985). For the K-feldspars with a potassium content of 13.55%, this implies a rubidium concentration of $\sim 600\ \mu g\, g^{-1}$, not dissimilar to the value of $400 \pm 100\ \mu g\, g^{-1}$ recommended by Huntley and Hancock (2001). Both studies imply that the internal dose rate (from K and Rb) used by Zhao and Li (2002) was too low. However, using these new values for K and Rb dose rates do not resolve the discrepancy between the ages of quartz (13.2 ± 0.8 ka) and K-feldspar (8.9 ± 0.5 ka) as re-calculated for this sample. The ICP-MS analyses also allowed the U and Th contents to be obtained on K-feldspar grains from the two samples. The values of ~ 2 ppm for Th and ~ 0.6 ppm for U resulted in an internal alpha dose rate of $\sim 0.30\ Gy\, ka^{-1}$ and an internal beta dose rate of $\sim 0.03\ Gy\, ka^{-1}$ for the 450–500 μm grains that were analysed (Zhao and Li, 2005). For the grain size range (90–250 μm) being used by Zhao and Li (2002) to construct their isochrons, the internal alpha dose rate remains effectively constant, since almost all alpha particles produced in a grain will deliver their dose inside the grain. The internal beta dose rate from U and Th will vary with grain size, but in concern with the internal beta dose rate from K and Rb; its effect on the slope of the isochron will be negligible as it contributes only $\sim 1\%$ to the internal beta dose rate.

To address whether the IRSL signals observed in the current study are from K-feldspar grains or plagioclase feldspar grains, we have made some single grain IRSL measurements. Since the IRSL signal of K-feldspar has been shown to be more thermally stable than that of Na-feldspar (Li and Wintle, 1992; Tso et al., 1996), single grain pulse annealing measurements have been applied to the samples in the current study. These experiments showed that more than 98% of the grains that give detectable IRSL signal from the K-feldspar fraction separated using a heavy liquid with a specific gravity of 2.58 are K-feldspars (in preparation).

3. The isochron plot of Zhao and Li

It is first necessary to summarize the isochron dating procedure proposed by Zhao and Li (2002) and consider the assumptions behind it. A simplified version of the isochron plot for a