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A comparative study of the luminescence characteristics of polymineral fine grains and coarse-grained K- and Na-rich feldspars

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ABSTRACT

The IRSL and post-IR IRSL (pIRIR) signal characteristics of polymineral fine grains are investigated and compared with those of K- and Na-rich feldspar extracts. TL signal loss after IR and pIRIR stimulations occurs mainly at around 320 °C for polymineral and Na-feldspar samples and around 410 °C for K-feldspar samples, when a preheat temperature of 250 °C for 60 s is used. After preheating to a higher temperature (320 °C for 60 s) all samples show a TL reduction around 410 °C in the blue detection window. Pulse annealing experiments for IRSL and pIRIR signals for preheats between 320 °C and 500 °C indicate that the signal stabilities are similar among the different feldspar types, when a higher preheat temperature (>320 °C) is used. Thermal activation energies for IRSL and pIRIR signals are largest in K-feldspar and smallest in polymineral fine grains, in both blue and UV detection windows for both fast time-resolved (TR) and continuous wave (CW) signals. These results suggest that IRSL and pIRIR signals in polymineral fine grains originate mainly from Na-feldspar grains; these signals are less thermally stable than those from K-feldspar, but a more stable signal (presumably from K-feldspar grains) can be obtained using a higher preheat temperature.

1. Introduction

Elevated temperature post-IR IRSL (pIRIR) measurements have proved to be very useful in preferentially isolating a stable luminescence signal from feldspars. In the original studies by Thomsen et al. (2008) and Buylaert et al. (2009) it was observed that a pIRIR stimulation at 225 °C (after stimulating with IR for 100 s) shows a significantly reduced fading rate compared to the IRSL at 50 °C, in both laboratory irradiated and geological samples. These authors used a preheat of 250 °C for 60 s as recommended by, for example, Huot and Lamothe (2003). Further investigations demonstrated that the IRSL signal at 50 °C from K-feldspar may be associated with a trap at ~410 °C (Murray et al., 2009); this observation made it possible to explore the effect of increasing the temperature of the preheat to >300 °C and measuring pIRIR at >225 °C, in the hope of sampling an effectively stable signal. Indeed, studies using the pIRIR signal at 290 °C after a preheat at 320 °C for 60 s have shown little or no anomalous fading in both sand-sized K-rich feldspar extracts and silt-sized polymineral fine grains (Thiel et al., 2011a,b,c; Thomsen et al., 2011; Buylaert et al., in press; Schmidt et al., submitted for publication a,b). Li and Li (2011) argue for a two (or several) trap model to explain the different thermal stabilities of IRSL and pIRIR signal, but Jain and Ankjærgaard (2011) show that when the role of the excited state and band tail states is considered in the process of IRSL generation, a single trap model is sufficient to explain these observations.

These studies have focused mainly on K-rich feldspar extracts; there have been no detailed investigations into the origins of the IRSL and pIRIR signals from Na-rich feldspars. This lack of knowledge restricts the routine application of the pIRIR method to polymineral fine grains (grain diameter 4–11 μm). Although such grains are a mixture of different kinds of minerals, feldspar is considered to be the primary phase that emits luminescence when stimulated with IR. The only published work on the stability of the IRSL signal from polymineral fine grains is Li and Wintle (1992),

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which compared the thermal stability of IRSL signals from polymineral fine grains with those from K- and Na-rich feldspars. They found that the thermal stability of the IRSL signals from fine grains from different areas around the world is similar, and that IRSL signals from fine grains have a similar stability to that of Na-feldspar, and are less stable than K-feldspar.

In this study the luminescence characteristics of six polymineral fine grain samples are compared with those of coarse-grained K- and Na-rich feldspar extracts, in order to identify the IR dosimetric trap in polymineral fine grains. Measurements of 1) TL signal loss after IR and pIRIR stimulations, 2) thermal stability, and 3) stimulation temperature dependence of both IRSL and pIRIR signals for continuous wave (CW) and pulsed time-solved (TR) IR stimulations, are used in this comparison.

2. Samples

Six aeolian, polymineral fine grain (4–11 μm) samples from Austria (Lum1217; Thiel et al., 2011b), Germany (Toe8; Schmidt et al., 2011), Croatia (Vuk1; Wacha and Frechen, 2011), Hungary (Lum1887), China (S1C; Tsukamoto et al., 2008), and USA (Kel1) were used in this study. Coarse grain K-rich feldspar extracts are from Italy (Lum1286, Thiel et al., 2010), Denmark (970203) and Russia (972503; Tsukamoto et al., 2006). Na-rich feldspar extracts are from Hungary (Lum2071) and from the same samples from Denmark and Russia as above (970203 and 972503). The coarse grain K- and Na-feldspar samples were separated after standard chemical treatment using heavy liquid (sodium polytungstate) of density <2.58 g cm^{-3} for K-feldspar and between 2.58 g cm^{-3} and 2.62 g cm^{-3} for Na-feldspar. Lum1286 and Lum2071 were not etched using HF, whereas other K- and Na-feldspar samples (970203 and 972503) were etched using 10% HF for 40 min. Because of the separation method used, it is possible that there are some contaminating K-feldspar grains in the Na-rich fraction and vice versa. The chemical composition of grains in these samples was measured with energy dispersive X-ray spectrometry with a scanning electron microscope (SEM–EDX; a FEI Quanta 600FEG). Between 50 and 97 grains from each sample were measured using SEM–EDX and the results were used to identify the mineralogy. Potassium content in K-feldspar grains and the orthoclase/albite/anorthite ratio for all six samples were calculated and are summarised in Table S1. The results confirm that the K-feldspar samples are dominated by K-feldspar grains. However, all Na-rich feldspar samples contain at least ~40% K-feldspar grains; the Na-feldspar samples used here are rather approximately 1:1 "Na–K–feldspar" mixtures.

The mineralogical composition of the fine grain samples were analysed by X-ray diffractometry (XRD) using 200–300 mg of the samples. Semi-quantitative analysis was performed using the Rietveld software AutoQuan® and an approximate percentage of each mineral was calculated (Table S2). The results suggest that all polymineral fine grains contain quartz and muscovite as the major constituent minerals but with additional Na-rich plagioclase, chlorite, and K-feldspar (microcline). Since it is difficult to identify the exact types of feldspars by XRD analysis alone, SEM–EDX analysis was also applied to the fine grain samples. Between 162 and 194 fine grains from each sample were analysed and the data were compared with the expected chemical compositions for the mineral assembly obtained from the XRD (quartz, Na-feldspar, K-feldspar, mica, chlorite and dolomite/ankerite). The percentage of each mineral is summarised in Table S3. Results of both methods (Tables S2 and S3) are consistent, but for mica and chlorite only the sum of both minerals correlate. Four samples (Lum1217, Lum1887, S1C, and Vuk1) are clearly more dominated by Na-feldspar grains with a minor content of K-feldspar. The other two samples contain similar numbers of Na- and K-feldspar grains (Toe8) or more K-feldspar grains than Na-feldspar (Kel1).

3. Experimental details

An automated Risø TL/OSL reader (TL/OSL DA-20) equipped with a pulsed stimulation attachment and a Photon Timer (Lapp et al., 2009) was used for TL and IRSL measurements. All luminescence measurements described below were done both in a UV detection window using a Hoya U-340 (280–380 nm) filter and in a blue window using a combination of Schott BG-39 and Corning 7-59 (320–460 nm) filters. IR stimulation used IR diode arrays delivering ~130 mW/cm^2 at the sample position. One aliquot per sample was mounted as loose grains in stainless steel cups. All IRSL and pIRIR signals were measured at 50 °C for 100 s for IRSL and at 225 °C or at 290 °C for 200 s for pIRIR unless stated otherwise. The integrated intensity of the initial 5 s minus a background from the last 15 s was used to calculate the intensity for both IRSL and pIRIR signals.

4. TL signal loss after IR and pIRIR stimulations

A measurement of TL signal loss after IR stimulation is commonly used to investigate the relationship between IRSL and TL signals (e.g. Duller and Wintle, 1991; Duller, 1995; Murray et al., 2009; Tsukamoto et al., 2011) although Murray et al. (2009) also point out the ambiguities of such analyses. TL signal loss after IR and pIRIR stimulations was examined for all samples after giving a beta dose of 120 Gy. TL glow curves up to 500 °C were measured at a ramping rate of 5 °C/s after each of the following treatments:

TL1: dose, preheat, TL
TL2: dose, preheat, IRSL, TL
TL3: dose, preheat, IRSL, hold temperature at T°C for 200 s, TL
TL4: dose, preheat, IRSL, pIRIR at T°C for 200 s, TL

The measurements were made for all 12 samples with the two different preheat and pIRIR temperature combinations that have been commonly used: preheat at 250 °C for 60 s and pIRIR at 225 °C ($pIRIR_{225}$), and preheat at 320 °C for 60 s and pIRIR at 290 °C ($pIRIR_{290}$). The same sets of experiments using these two preheat and pIRIR combinations were undertaken using both the blue and the UV detection windows.

Fig. S1a shows the four TL glow curves from a K-feldspar sample (970203) using the preheat of 250 °C and $pIRIR_{225}$. The same sets of TL glow curves for 320 °C preheat and $pIRIR_{290}$ are also shown in Fig. S1b. Two TL peaks are observed at around 320 °C and 410 °C after preheating at 250 °C. The use of a 320 °C preheat in the second data set leads to the erosion of the lower temperature peak. The loss of TL due to IR and pIRIR stimulations was obtained by TL1–TL2 and TL3–TL4, respectively, and is plotted in Fig. 1 for the blue detection for polymineral (Lum1887), Na-feldspar (Lum2071) and K-feldspar (970203) samples.

In the blue detection window, TL reduction after IRSL for both Na-feldspar and K-feldspar shows a double peak structure at 320 °C and 410 °C, with the shoulder on either the higher or the lower temperature side (Fig. 1b, c) respectively, but this structure is not reflected in the polymineral data. In fact, the polymineral sample shows a narrower TL loss peak than both Na- and K-feldspar samples, with an almost complete absence of the higher temperature shoulder. In K-feldspar, the low temperature peak at 320 °C is more or less completely removed in the $pIRIR_{225}$ data; indeed there is actually some increase in the TL peak at the lower temperature after $pIRIR_{225}$. The main depletion in TL due to pIRIR is obvious at 410 °C (Fig. 1c). After preheating at 320 °C, the TL reduction in all feldspars occurs at 410 °C after both IR and $pIRIR_{290}$ stimulations