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Correlation of basic TL, OSL and IRSL properties of ten K-feldspar samples of various origins

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ABSTRACT

Feldspars stand among the most widely used minerals in dosimetric methods of dating using thermoluminescence (TL), optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL). Having very good dosimetric properties, they can in principle contribute to the dating of every site of archaeological and geological interest. The present work studies basic properties of ten naturally occurring K-feldspar samples belonging to three feldspar species, namely sanidine, orthoclase and microcline. The basic properties studied are (a) the influence of blue light and infrared stimulation on the thermoluminescence glow-curves, (b) the growth of OSL, IRSL, residual TL and TL-loss as a function of OSL and IRSL bleaching time and (c) the correlation between the OSL and IRSL signals and the energy levels responsible for the TL glow-curve. All experimental data were fitted using analytical expressions derived from a recently developed tunneling recombination model. The results show that the analytical expressions provide excellent fits to all experimental results, thus verifying the tunneling recombination mechanism in these materials and providing valuable information about the concentrations of luminescence centers.

1. Introduction

Thermoluminescence (TL), optically stimulated luminescence (OSL) and infrared stimulated luminescence (IRSL) are well known dosimetric methods for dating (DMD). They are based on the dosimetric properties of naturally occurring inorganic materials. Feldspars stand among the most widely used minerals in DMD and can be found in all rock types, igneous, sedimentary and metamorphic. Having very good dosimetric properties, they can in principle contribute to the dating of every site of archaeological and geological interest. However, their general use in DMD is limited by the anomalous loss of their TL and OSL signal after irradiation, which contradicts the predictions of standard luminescence kinetic theory.

In a pioneer study Wintle [1] examined the possibility of using feldspars from volcanic rocks for dating, but found problems with anomalous signal loss after irradiation. Mejdahl [2] showed that although fading did exist in some samples, the phenomenon was not ubiquitous, and he pioneered the use of feldspars in archaeological, and subsequently geological, dating applications [3,4]. This anomalous signal loss is known as anomalous fading (AF), and has been reported for both IRSL and OSL emissions [5–8]. The AF phenomenon is attributed to quantum tunneling effects [1,9–17].

One of the great advantages of working with feldspars (as opposed to quartz) is that many feldspars exhibit a luminescence signal when stimulated in the infrared. Hutt et al. [18] showed the preferential depletion of this specific signal at ambient temperatures. Furthermore, due to their attractive dosimetric properties, feldspars are still extensively studied in order (a) to circumvent AF [19–22], (b) to explore the tunneling mechanism [13,17,23–28], (c) to find the appropriate corrections for the anomalous signal loss [23,24,29], and (d) to find new energy levels which do not suffer from anomalous fading [30–32].

The thermal, optical and infrared stimulation modes considered in the present work are entirely different. However, it is possible that all these stimulation modes access the same trap. It is assumed that the stimulated electrons escape from the traps and recombine with positive holes, giving rise to the emission of TL

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or OSL or IRSL light, which is a measure of the natural dose. These three stimulation modes have been recently described by the model of Jain et al. [26,27], which is based on tunneling recombinations within a random distribution of donor–acceptor pairs. Within this model, Kitis and Pagonis [28] developed analytical equations which can describe these luminescence signals.

Recently Polymeris et al. [33] investigated the possibility of using TL for structural characterization of ten K-feldspar samples. They found a good correlation between TL sensitivity and individual K-feldspar structure. The properties of the group of samples used by Polymeris et al. [33] make them ideal for investigating basic TL, OSL and IRSL properties, as well as their correlation.

The main goal of the present work is to use the analytical expressions of Kitis and Pagonis [28], to describe OSL, IRSL and residual TL (R-TL) signals from these ten K-feldspar samples.

The specific goals of this work are:

- To study the influence of optical and infrared bleaching on the TL glow-curves. Previous researchers have reported on these bleaching effects [34–39], but to the best of our knowledge this is the first quantitative description of these bleaching effects using analytical equations.
- To find a correlation between the OSL and IRSL signals and the TL lost after optical or infrared bleaching.
- To describe and fit the CW-OSL and CW-IRSL signals using analytical expressions
- To perform a component resolved analysis of CW-OSL and CW-IRSL decay curves.

2. Experimental procedure

2.1. Sample details and preparation

The samples used in this study are those used by Polymeris et al. [33] and are listed in Table 1. As it is seen from Table 1 the samples were ten naturally occurring K-feldspars from igneous rocks of Northern Greece. The K-feldspars were separated from mafic and felsic minerals with the use of Franz (model L-1) magnetic separator and Sodium Polytungstate (SPT) heavy liquid, respectively. The purity of K-feldspars was identified and classified based on XRPD measurements. The XRPD patterns were obtained on a PHILIPS PW 1820/00 X-ray diffractometer. The operating condition for all samples were 35 kV and 25 mA using Ni-filtered Cu $K_{\alpha ave}$ radiation. The 2theta (2θ) scanning range was between 3° and 63° min^{-1}. The identification of the samples was made using the JCPDS-ICDD 2003 database. The unit cell parameters, as well as, refinements were calculated with CHEKCELL software [40]. The probability of an Al-cation occupying one of the T1 sites ($\Sigma T1 + T10 + T1m$) was calculated using the Kroll and Ribbe equations [41]. According to the XRPD patterns of the examined samples, they are divided in three species: sanidine, orthoclase and microcline. It should be noted that the probability $\Sigma T1$ has the lowest values for sanidine and the highest for microcline samples. Therefore, the examined samples were selected to cover the entire region of the potential $\Sigma T1$ values. Structural classification of K-feldspars are summarized by Deer et al. [42], while spectral information from feldspars relevant for luminescence dating were previously presented by Krbetschek et al. [43].

Table 1
K-Feldspar samples studied in the present work.

Sample	Kfs species	Unit cell volume (A^3)	$\Sigma T1$
bal2	Sanidine	699.5000 ± 0,3700	0.53 ± 0,023
sam2	Sanidine	704.8630 ± 0.4340	0.55 ± 0.024
sam3	Sanidine	708.4800 ± 0.2830	0.63 ± 0.019
mrk4	Orthoclase	713.5640 ± 0.4580	0.66 ± 0.028
xan8	Orthoclase	718.9580 ± 0.3550	0.79 ± 0.023
vrs4	Orthoclase	717.6500 ± 0.4100	0.86 ± 0.028
eld1	Microcline	717.4400 ± 0.2870	0.85 ± 0.019
vrs3	Microcline	717.2540 ± 0.5120	0.89 ± 0.042
vrs8	Microcline	713.9430 ± 0.3650	0.90 ± 0.022
kst4	Microcline	718.8690 ± 0.4020	0.98 ± 0.029

2.2. Apparatus and measurement conditions

Luminescence measurements were carried out using a Risø TL/OSL reader (modelTL/OSL?DA?15), equipped with a 90Sr/90Y beta particle source, delivering a nominal dose rate of 0.075 Gy/s, a 9635QA photomultiplier tube and a 7.5 mm Hoya U-340 filter (340 nm, FWHM 80 nm). Even though it is well established that IRSL normally consists of photons in violet-blue range, the Hoya U-340 filter was applied in all luminescence measurements in order to perform correlations. Therefore, in present work the UV part of the IRSL has been studied. All measurements were performed in a nitrogen atmosphere with a low constant heating rate of 2 °C/s, in order to avoid significant temperature lag, and the samples were heated up to the maximum temperature of 500 °C.

The OSL stimulation wavelength is 470 (±20) nm for the case of blue stimulation, delivering at the sample position a maximum power of 40 mW/cm^2. For IRSL, the stimulation wavelength is 875 (±40) nm and the maximum power of ~135 mW/cm^2. Conventional OSL and IRSL measurements were performed at RT.

2.3. Experimental protocol

The experimental procedure used is according to the following protocol.

- Step 0: Test dose and TL measurement up to a temperature $T = 500$ °C at 2 °C/s.
- Step 1: Test dose .
- Step 2: Continuous-wave OSL (CW-OSL) at room temperature for time t_i.
- Step 3: TL measurement up to a temperature $T = 500$ °C at 2 °C/s.
- Step 4: Repeat steps 1–3 for a new stimulation time t_i.
- Step 5: Test dose and TL measurement up to a temperature $T = 500$ °C at 2 °C/s.

The stimulation times used were t_i = 0, 1, 2, 4, 8, 16, 32, 50, 75, 100, 200, 400, 600, 800, 1000, 1500 and 2000 s. The case of $t_i = 0$ s corresponds to unbleached TL. In order to avoid any pre-conditioning of the samples between irradiation and measurement, room temperature was selected as the stimulation temperature for all measurements.

The same protocol was also applied using CW-IRSL stimulation in step 2, instead of CW-OSL stimulation.

Both protocols (CW-OSL and CW-IRSL) run in single aliquot mode. The sensitivity tests in steps 0 and 5 were also applied two more times after the bleaching times of 32 and 400 s. In all cases the results verified the already known excellent stability of these materials to repeated irradiation-TL readout cycles. Negligible sensitivity changes were observed for all samples.

3. Results and discussion

3.1. Influence of the bleaching on the shapes of TL glow-curves

The glow-curve shapes of K-feldspar samples listed in Table 1 were reported by Polymeris et al. [33]. A characteristic example of the way the optical and infrared stimulations bleach the electron