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The *a*-value of polymineral fine grain samples measured with the post-IR IRSL protocol

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HIGHLIGHTS

• Measurement of the *a*-value on polymineral fine grain using the pIRIR protocol.

• Mean *a*-value differs for the IR₅₀ and pIRIR₂₂₅ signal.

• We observe a dependency of the *a*-value on the method of signal resetting.

• *a*-values appear to be dose dependent.

• It seems not appropriate to use identical *a*-values for IR₅₀ and pIRIR₂₂₅.

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1. Introduction

Optically stimulated luminescence (OSL) dating is conventionally applied to natural mineral grains of quartz, feldspar or a mixture of minerals (polymineral). In most cases, quartz is a reliable dosimeter, but low luminescence sensitivity and mean saturation doses (D_0) of typically around 100–200 Gy (e.g., Yoshida et al., 2000; Roberts, 2008) limit its application. For (K-) feldspar a higher sensitivity and signal growth up to 1.5 kGy is reported (e.g.,



Recent post-IR IRSL (pIRIR) dating studies using polymineral fine grains assumed that the *a*-values obtained for the IRSL signal at 50 °C and the pIRIR signal at higher temperatures (e.g. 225 °C) are identical. However, the *a*-value of a sample depends on the stimulation method, and the assumption mentioned above remains to be tested. Using five polymineral fine grain samples, this study investigates whether a common *a*-value can be used for both the IR and the pIRIR signals. Applying the pIRIR protocol, the *a*values were measured with three different methods of signal resetting (optical bleaching, end of SAR cycle, heating). In addition, uncorrected α - and β -irradiation induced growth curves were determined for three samples and fitted with single saturating exponential functions. For the investigated samples we found significant mean differences, 0.023 ± 0.012 and higher, in the *a*-values determined for the IR₅₀ and pIRIR₂₂₅ signals. Synthetic *a*-values deduced from uncorrected multiple-aliquot dose response curves seem to confirm this observation. Although, in summary, our results indicate that the practice of using a common *a*-value should be carefully re-considered, the physical reasons remain to be determined. © 2014 Elsevier Ltd. All rights reserved.

> Hütt et al., 1988). However, the major disadvantage in using feldspar instead of quartz is an athermal signal reduction at ambient temperature with time (anomalous fading; Wintle, 1973; Visocekas, 1985) leading to an underestimation of the true burial dose. Much effort has been undertaken to circumvent this problem by using thermal pre-treatments (e.g., Li, 1991; Kadereit, 2000) or postmeasurement fading corrections (e.g., Huntley and Lamothe, 2001; Auclair et al., 2003). Based on the single-aliquot regenerative-dose (SAR) approach (Murray and Wintle, 2000), Thomsen et al. (2008) and Buylaert et al. (2009) presented a new protocol for equivalent dose (D_e) determination using the infrared stimulated luminescence (IRSL) signal at elevated temperatures measured after a prior IR bleach at low temperatures. The obtained



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post-IR IRSL (pIRIR) signal has been shown to be less affected by fading, and since then an increasing number of dating applications have been presented using the new protocol (e.g., Buylaert et al., 2011; Thiel et al., 2011; Lowick et al., 2012; Buylaert et al., 2012; Vasiliniuc et al., 2012; Murray et al., 2014; Thiel et al., 2014). Recent developments extended the post-IR IRSL approach by using multi-elevated temperature steps (Li and Li, 2011; Fu et al., 2012).

Originally tested for coarse grain (90-250 µm) feldspars (Thomsen et al., 2008), the application has been further extended to the polymineral fine grain fraction $(4-11 \mu m; e.g., Buylaert et al.,$ 2009, 2011; Lowick et al., 2012; Thiel et al., 2011; Schmidt et al., 2011; Vasiliniuc et al., 2012; Schatz et al., 2012; Murray et al., 2014). Using stimulation energies of about 1.4 eV, i.e. the approximate IR excitation resonance of feldspars, for the polymineral fine grain fraction it is assumed that the measured emission in the violet-blue band is predominantly caused by feldspar minerals. Nevertheless, the age calculation is more complex than for coarse grain feldspar separates, since the effects of α -radiation have to be considered additionally. In contrast, for the coarse grain fraction it is commonly presumed that the outer α -irradiated layer of the grains is removed by etching with HF (10%) for 40 min (e.g., Thomsen et al., 2008; Buylaert et al., 2009; Lauer et al., 2011). Therefore, the contribution of the α-radiation to the total dose rate is neglected.

The diameter of the fine grains is small compared to the mean natural α -particle range in sediments (>20 μ m for $\rho = 2.6$ g cm⁻³, e.g., Aitken, 1985a) and therefore fine grains are considered to be fully penetrated by α -particles (e.g., Zimmerman, 1971, 1972). However, in contrast to radiation with low ionisation density (or low Linear Energy Transfer, LET) such as β - and γ -radiation or highenergy X-rays, α -particles are comparatively heavy, thus providing high ionisation density (e.g., Zimmerman, 1972; Jain et al., 2007). This causes localised charge (over-) saturation of electron traps along the approximately linear tracks of the α -particles through the dosimeter (e.g., Zimmerman, 1972; Stolz, 2005). According to Fleming (1979), a simplified view of this mechanism is a competitive process between charge trapping and instantaneous recombination, while recombination is thought to be more intense than in case of incident β - or γ -radiation. Practically, it is observed that the luminescence response of α -irradiation and of β -irradiation following the same nominal dose, differs by an order of magnitude. The difference is termed as α -effectiveness or α -efficiency. Zimmerman (1972), who systematically investigated the thermoluminescence effects of α - and β -irradiation, expressed the numerical relationship by use of the so called k-value system (Zimmerman, 1971). It should be noted that he also briefly discussed other physical effects to explain the difference in the induced luminescence such as 'ionic displacement' or 'thermal release' (Zimmerman, 1972), but so far the above described 'energydensity' model seems to be broadly accepted. Along with the original k-value system from Zimmerman (1971), three other systems were introduced to describe the relationship of the induced luminescence by high and low LET radiation (Aitken and Bowman, 1975; Valladas and Valladas, 1982; Bowman and Huntley, 1984). An overview of the connection between the systems is given by Aitken (1985b). It was found by Aitken and Bowman (1975) that the luminescence output per generated unit α -track length is nearly independent of α -energy and so in this system generated track length ($\mu m^* \mu m^{-3} = \mu m^{-2}$) is used instead of absorbed dose (Gy) to quantify incident α -radiation (a-value system). For quartz and mono-energetic α -irradiation (mostly 3.7 MeV in the case of ²⁴¹Am), *a*-value = *k*-value. Other materials than quartz require an additional correction factor between both values, which depends, amongst others, on the density of the dosimeter. However, the difference in density of quartz and feldspar accounts for a ~2%

deviation from the uncorrected *a*-value only, thus it is considered as negligible within measurement uncertainty.

Accordingly, the *a*-value of feldspar samples can be simply written as the ratio of the α - to the β -dose induced luminescence per unit dose (Gy). In addition, α -efficiency determination using this system has been adopted for the commonly used SAR protocol (Murray and Wintle, 2000) by Mauz et al. (2006) and Lai et al. (2008). For the physical meaning of the system and the details of the application the reader is referred to the named literature.

For pIRIR dating of polymineral fine grains, dose rate calculation has usually been carried out using a fixed *a*-value taken from the literature (e.g., from Rees-Jones, 1995 as done by Buylaert et al., 2009, 2011; Thiel et al., 2011; Schmidt et al., 2011; Vasiliniuc et al., 2012; Schatz et al., 2012; Murray et al., 2014). Based on previous empirical findings and theoretical considerations, we propose that the use of a fixed *a*-value is not justified:

- (1) The most frequently used *a*-value is 0.08 ± 0.02 and is taken from Rees-Jones (1995). In this work, *a*-values of only three polymineral fine grain samples with a mean of 0.09 and a standard deviation of 0.02 are presented. Irrespective of methodological differences, other extensive work (e.g., Lang, 1996; Kadereit et al., 2006, 2010) has shown that the *a*-value of the polymineral fraction is likely to vary to a much greater extent.
- (2) Using the pIRIR protocol, first the unstable (fading affected) signal is removed using an infrared light depletion at low temperatures. The subsequent stimulation at higher temperatures is assumed to increase the probability of electrons to recombine at more distant centres (Thomsen et al., 2008; Jain and Ankjærgaard, 2011) so that different signals are measured in the same detection window by using varying stimulation energies. Nevertheless, there is a priori no obvious reason to believe that both signals are similarly affected by α -irradiation.
- (3) The second IR stimulation step is carried out at elevated temperatures of 225 °C or 290 °C (e.g., Thiel et al., 2011). In previous work it has been pointed out that the *a*-value of thermally stimulated luminescence is markedly higher than that of optically stimulated signals for the same sample (e.g., Zöller, 1995; Lang, 1996; Tribolo et al., 2001; Lai et al., 2008), and Zimmerman (1972) further reported a difference of *a*-values for different TL peaks in quartz due to different traps with specific saturation behaviour. Although the thermally assisted pIRIR at 225 °C is lower than the peak temperatures usually used in TL dating (e.g., 250–400 °C; e.g., Musson and Wintle, 1994), it is unclear how the elevated temperature affects the *a*-value when measuring a combination of TL and IRSL.
- (4) In addition, Tsukamoto et al. (2012) presented activation energies for the fast decaying IR signal of the fine grain polymineral fraction and derived substantially higher values for the pIRIR signal. From this, one may conclude that different populations of electron traps and recombination centres are involved in the processes giving rise to the IR and pIRIR luminescence signals which might therefore be affected by α -irradiation in a different way.
- (5) The work on volcanic ashes from Biswas et al. (2013) showed that the α -efficiencies of the pIRIR signals at higher temperatures (300 °C) are always higher compared to the IRSL signals at 50 °C and they suggest that the *a*-value should be determined using fine grains and the pIRIR protocol.

In this work we test the hypothesis that the *a*-value of polymineral fine grains for the IR signal at 50 $^{\circ}$ C (IR₅₀) and the pIRIR

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