IRSL dating of K-rich feldspars using the SAR protocol: Comparison with independent age control

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Abstract : *Infrared stimulated luminescence (IRSL) ages of four samples from three independently dated sites in the forelands of the Alps of Europe and New Zealand were determined using the single-aliquot regenerative-dose (SAR) methodology applied to K-rich feldspars. It is demonstrated that all IRSL ages agree well with the known age of the samples. Previously reported age underestimation of K-feldspar samples from the Rhine-Meuse delta was explained to result from increasing trapping probability caused by preheating of the natural sample. Experiments carried out for the samples investigated here, however, reveal no such increase. Hence, monitoring the trapping probability may be an appropriate test to identify K-feldspars suitable for SAR dating.*

Introduction

During recent years, the development of the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle 2000) significantly improved the precision of optically stimulated luminescence (OSL) dating of sediments. The SAR technique allows a more precise determination of the palaeodose than previously used multiple-aliquot additive-dose (MAAD) approaches. However, while the SAR method has most successfully been applied to quartz from different environmental settings (e.g. Banerjee et al. 2001, Colls et al. 2001, Fuchs and Lang 2001, Hilgers et al. 2001), when applied to K-feldspars from sediments of the Rhine-Meuse system the results systematically underestimated the expected palaeodose. In comparison with historical data and radiocarbon ages in the range 600 – 13,000 yr K-feldspars showed a systematic underestimation of up to 35 % (Wallinga et al. 2001). Experimental evidence suggested that this underestimation was caused by an increase in electron trapping probability as a consequence of preheating the natural sample (Wallinga et al. 2000a). Consequently, SAR methodology is rarely used for determination of the palaeodose of K-feldspars in luminescence dating. However, K-feldspars have some important advantages over quartz:

1) The saturation dose in feldspar is usually much higher than in quartz offering a potential to expand the range to be dated by luminescence methods.
2) The important contribution of ^{40}K within the grains to the total dose rate minimises the effect of uncertainties in the calculation of the external dose rate such as radioactive disequilibrium, water content, and cosmic dose.
3) Quartz originating from young orogenic systems such as the Alps often has poor luminescence properties, e.g. extremely low or no OSL signals. Frequently such quartz also shows large changes in sensitivity during repeated measurements that are not always corrected by the SAR protocol (Preusser 1999a, Stokes et al. 2000). Furthermore, it is difficult to deal with feldspar inclusions present in quartz from some geological provenances (Wallinga et al. 2002).

A reliable method to determine the palaeodose in K-rich feldspar would thus importantly improve the applicability of luminescence dating. It is important to notice that the investigations of Wallinga et al. (2000a, 2001) were all carried out on samples from the Rhine-Meuse system. This area lies down-stream of young volcanic rocks in the Eifel and Siebengebirge regions of western Germany. Thus, it is possible that the phenomenon causing the underestimation of K-feldspar ages is caused by the presence of volcanic feldspars, such as sanidine, in the sediment and might be absent in other geological regions. Feldspars of volcanic origin regularly show anomalous fading of the luminescence signal with time (Wintle 1973, Visocekas and Zink 1999 and references therein). However, in storage tests over periods up to six months, Wallinga et al. (2001) found evidence for only very limited fading of their samples (0.970 ± 0.007).

This study presents results for four samples from the forelands of the Alps of Europe and New Zealand for which independent age control exists. A modified SAR protocol was applied to K-rich feldspars using infrared stimulation.

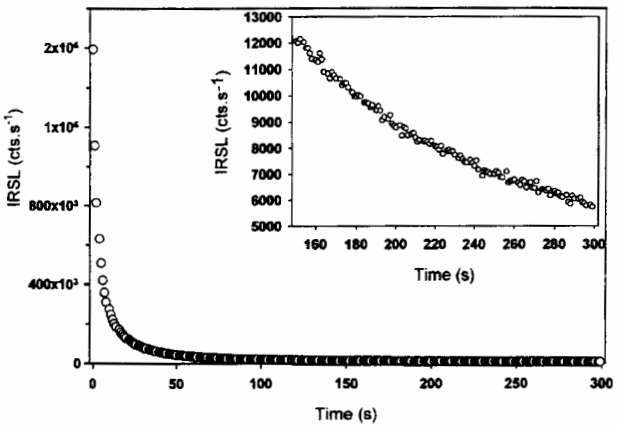

Figure 1.
IRSL decay curve for sample SHB1. The inset figure shows the latter part of the decay with an enlarged scale. Even after 150 s of IR exposure a further depletion of the IRSL signal is observed, but it is small in comparison to the initial signal.

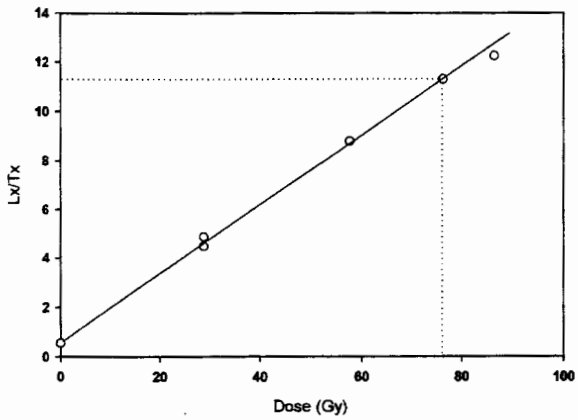

Figure 2.
Example of a dose response curve for an aliquot of sample GOS4.

Modified SAR protocol

Determination of the palaeodose was carried out using a modified SAR protocol following Murray and Wintle (2000) and Wallinga et al. (2000b). The SAR cycle included measuring the natural dose, three regenerative doses, and a zero dose. A replicate measurement of the lowest regenerative dose was carried out at the end of each SAR cycle. After each measurement a fixed test dose was given and the resultant IRSL intensity was used for normalisation. A preheat temperature of 290° C for 10 s was applied to all samples following Wallinga et al. (2001). A cut-heat temperature of 200° C was used for the test dose measurements. Stimulation by IR LEDs (maximum power 135 mW.cm^{-2} at the sample) for 300 s was applied to reset the latent infrared stimulated luminescence (IRSL) signal within the sample. The remaining IRSL signal after 300 s of IR exposure is 0.3 % of the original signal for the example given in Fig. 1. This is a typical value for most samples investigated so far. All measurements were carried out on a Risø TL/OSL 15 reader using 90 % IR diode power and carrying out the stimulation at a temperature of 125° C. A blue transmitting filter set (Schott BG 39, Schott GG 400, Corning 7-59) was used to isolate the main emission of K-rich feldspar at 410 nm (Krbetschek et al. 1997). This filter combination differs from the one used by Wallinga et al. (2000a) that transmitted IRSL in the near-blue UV, between 320 and 480 nm. A typical dose response curve is given in Fig. 2.

For all samples, three parameters are given to characterise the performance of the SAR protocol. Dose recovery indicates how accurately a known dose from a bleached (20 h by Osram Ultra Vitalux UV lamp) and irradiated (100 s beta = 11,96 Gy) but non-heated sample is recovered by the SAR protocol described above (given in Table 1 is the average of three aliquots). The recycling ratio represents how well the procedure corrects sensitivity changes that occur during repeated measuring, dosing, and preheating cycles. This performance is expressed as the ratio of the corrected luminescence intensity (L_x/T_x) of the lowest regenerative dose that is measured twice, at the beginning and at the end of the SAR cycle. The third parameter is a measure of the IRSL emitted by a bleached, non-irradiated but preheated sample (zero dose) in relation to the natural signal (L_x/T_x (zero dose) / (L_x/T_x (natural dose)). It is given as percent of the natural signal. An ideal sample will show a dose recovery of 100 %, a recycling ratio of 1.00 and 0 % recuperation.

A preheat test was carried out on a bleached (20 h by Osram Ultra Vitalux UV lamp) and dosed (11,96 Gy) sample (Fig. 3). The measured equivalent dose is almost constant with increasing preheat temperature, although, the recuperation signal increases with temperature. Fading tests for the samples investigated here have not yet been carried out, but they are available for samples from similar settings in both Switzerland (Preusser 1999a) and New Zealand (Hormes et al. 2003). None of these experiments revealed any loss of the luminescence signal over the period investigated (at least twelve months). Furthermore, Wallinga et al. (2000b, 2001) already demonstrated that fading does not explain the age short fall of their samples.

The concentration of dose rate relevant elements (K, Th, U) within the sediment was carried out by high-resolution gamma spectrometry (Preusser and Kasper 2001). For the calculation of internal dose rates potassium contents of 12.5 ± 0.5 % were used following Dütsch and Krbetschek (1997) and Huntley and Baril (1997). An alpha efficiency of 0.07 ± 0.02

was assumed. The dosimetric data is summarised in Table 2.

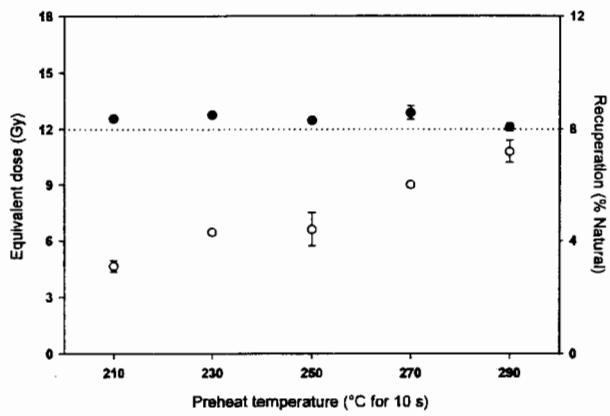

Figure 3.
Plot of equivalent dose (closed circles) and recuperation (open circles) as a function of preheat temperature (sample GOS4). The dotted line indicates the known beta dose given to the sample prior to the SAR cycle. While the equivalent dose is almost constant, recuperation slightly increases with rising preheat temperature. This does, however, apparently not effect the determination of the equivalent dose. A preheat of 290°C for 10s, giving a slightly lower but more accurate ED for sample GOS4, was applied in the dating procedure and the dose recovery test for all samples.

Dated sites and results

Four samples from three different sites were selected. In each case independent age control is available. One further sample was taken from sediment that is assumed to be older than the penultimate interglacial based on geological estimates. However, for this sample no verification of this age by other dating methods is available.

Sample GOS4 is a late Middle Würmian overbank deposit from a site near Gossau, Lake Zürich area, Switzerland. Three radiocarbon ages of 28,550 ± 310, 29,450 ± 1150 and 28,250 ± 350 ^{14}C yr have been determined for peat from just below the overbank deposit (Schlüchter et al. 1987). These radiocarbon dates represent an absolute age of *c.* 32,000 yr (Preusser et al. 2003). The U/Th age of that peat is 34,700 ± 4000 yr (Geyh and Schlüchter 1998). IRSL ages determined for the overbank deposit using the MAAD technique are 29,000 ± 3900 yr for polymineral fine-grains and 31,600 ± 3500 yr K-rich feldspars, respectively (Preusser 1999b, Preusser et al. 2003). The infrared radiofluorescence (IR-RF) age for the same sample is 32,900 ± 3400 yr (Erfurt et al. 2003).

The dose recovery experiment on sample GOS4 gave a value that was 101 ± 2 % of the known dose. Sixteen aliquots measured to determine the ED of the sample showed an average recycling ratio of 0.97 ± 0.07 and an average recuperation signal of 4.3 ± 0.7 %. The IRSL age of 28,400 ± 1900 yr determined by the SAR protocol fits well with previously calculated IRSL (MAAD) and IR-RF ages for the overbank deposit as well as with radiocarbon and U/Th dating results for the underlying peat.

Sample HURL1 originates from a sandy layer associated with sinter deposits from a site near Hurlach, Lech river valley, Bavaria, southern Germany. The Last Interglacial age of this sediment is indicated by pollen and malacological (snail) evidence as well as by an U/Th age of 120,300 ± 5800 yr (Jerz and Mangelsdorf 1989, Kovanda 1989). A previously published IRSL (MAAD) age of 134,800 ± 11,600 yr and an IR-RF age of 125,900 ± 12,200 yr (Erfurt et al. 2003) agree with both the U/Th age and biological evidence.
Dose recovery of sample HURL1 is 107 ± 2%. Eight aliquots measured for this sample show an average recycling ratio of 1.00 ± 0.15 and an average recuperation signal of 5.2 ± 0.2 %. The IRSL (SAR) age of 136,400 ± 10,500 yr agrees with independent age control.

Two samples (KMK5, KMK8) were taken from two different sandy layers within a sequence of fine-grained overbank deposits intercalating last glacial glaciofluvial out-wash gravel at a site near Kamaka, North Westland, South Island of New Zealand. A series of 14 radiocarbon ages determined for wood and plant fragments from this horizon range from 22,800 ± 200 ^{14}C yr to 21,990 ± 220 ^{14}C yr (Denton et al. 1999). This represents an absolute age of *c.* 25,000 yr when applying the calibration of Kitagawa and van der Plicht (1998).
Six and seven aliquots were measured for samples KMK5 and KMK8, respectively. Dose recovery for these two samples is 98 ± 2 % and 97 ± 0 %, respectively. The average recycling ratio is 1.10 ± 0.04 for sample KMK5 and 1.10 ± 0.08 for sample KMK8. The recuperation signal for the samples is 10.9 ± 1.3 % (KMK5) and 9.6 ± 3.1 % (KMK8). IRSL (SAR) ages of 24,300 ± 1500 yr (KMK5) and 24,400 ± 1500 yr (KMK8) agree very well with the calibrated radiocarbon age of *c.* 25,000 yr.

The estimated age of sample SHB1 from a quarry at Solenberg near Schaffhausen, northern Switzerland, is older than the penultimate interglacial (Graf, pers. com.). The sample was taken from a fluvial sediment

Sample	Origin	Dose recovery (%)	n	Recycling ratio	Recuperation. (%)	IRSL-Age (SAR) (yr)	Age control
GOS4	Gossau, CH Overbank deposit	101 ± 2	16	0.97 ± 0.07	4.3 ± 0.7	28,400 ± 1900	^{14}C: *c.* 32,000 cal. ^{14}C yr (2) U/Th: 34,700 ± 4000 yr (3) IRSL (MAAD): 29,000 ± 3900 yr (4) IRSL (MAAD): 31,600 ± 3500 yr (5) IR-RF: 32,900 ± 3400 yr (6)
HURL1	Hurlach, D Fluvial sand	107 ± 2	8	1.00 ± 0.15	5.2 ± 0.2	136,400 ± 10,500	U/Th: 120,300 ± 5800 yr (7) IRSL (MAAD): 134,800 ± 11,600 yr (6) IR-RF: 125,900 ± 12,200 (6)
KMK5 KMK8	Kamaka, NZ Overbank deposit	98 ± 2 97 ± 0	6 7	1.10 ± 0.04 1.10 ± 0.08	10.9 ± 1.3 9.6 ± 3.1	24,300 ± 1500 24,400 ± 1500	^{14}C: *c.* 25,000 cal. ^{14}C yr (1)
SHB1	Solenberg, CH Fluvial sand	106 ± 1	3	0.96 ± 0.04	2.8 ± 0.2	268,000 ± 18,000	none

Table 1.
Overview on relevant information of samples investigated in the present study. References: (1) Denton et al. (1999), (2) Schlüchter et al. (1987), (3) Geyh and Schlüchter (1987), (4) Preusser 1999b, (5) Preusser et al. (2003), (6) Erfurt et al. (2003), (7) Jerz and Mangelsdorf (1989).

Sample	Grain size (µm)	K (%)	Th (ppm)	U (ppm)	water (%)	Depth (m)	$D_{Internal}$ (Gy kyr^{-1})	$D_{External}$ (Gy kyr^{-1})	D (Gy kyr^{-1})	D_e (Gy)
GOS4	100-200	1.03 ± 0.05	8.21 ± 0.41	2.68 ± 0.13	20 ± 5	7	0.50 ± 0.16	2.01 ± 0.15	2.51 ± 0.16	71.3 ± 1.8
HURL1	100-200	1.00 ± 0.02	5.22 ± 0.24	2.67 ± 0.09	15 ± 5	1	0.50 ± 0.16	1.99 ± 0.15	2.49 ± 0.15	340.2 ± 15.7
KMK5	100-200	1.69 ± 0.05	9.14 ± 0.27	2.24 ± 0.07	25 ± 5	15	0.50 ± 0.16	2.34 ± 0.04	2.84 ± 0.16	68.9 ± 2.0
KMK8	100-200	1.49 ± 0.05	12.04 ± 0.36	2.90 ± 0.09	25 ± 5	15	0.50 ± 0.16	2.50 ± 0.06	3.00 ± 0.16	73.2 ± 2.4
SHB1	100-150	0.74 ± 0.02	3.73 ± 0.17	1.64 ± 0.06	15 ± 5	20	0.42 ± 0.08	1.64 ± 0.04	2.06 ± 0.11	550.9 ± 23.4

Table 2.
Dosimetric data, internal, external (including cosmic) and total dose rates and palaeodose for the investigated samples.

that is presumed to be deposited under cold climatic conditions. For this sample, the number of regenerative doses was increased to five allowing a better fitting of exponential dose response curves.
Dose recovery of sample SHB1 is 106 ± 1 %. Three aliquots measured for this sample show an average recycling ratio of 0.96 ± 0.04 and a recuperation signal of 2.8 ± 0.2 %. The calculated age of 268,000 ± 18,000 yr indicates a correlation with marine isotope stage (MIS) 8. From the geological point of view, this age agrees with a period of assumed cold climatic conditions.

Discussion

The dose recovery tests described here show that the modified SAR protocol works well for artificially dosed samples. Furthermore, none of the ages calculated shows any age shortfall in comparison with independent age control, in contrast to the experience of Wallinga et al. (2001) with sediments from the Rhine-Meuse system.
It may be possible that age shortfall in the different samples is masked by incomplete bleaching (Wallinga et al. 2001). However, previous experience with MAAD dating revealed that overbank deposits from different regions are usually well bleached prior to deposition (Fuller et al. 1994, Becker-Haumann et al. 2000, Fiebig and Preusser 2003, Hormes et al 2003). Similar experience is available for fluvial terrace sediments from the Alpine Foreland (Fiebig and Preusser 2001, 2003). Additionally, all samples investigated show a constant ED with stimulation time and the reproducibility of small aliquots (*c.* 50 grains) measured for the same samples is rather good (about 10 %). However, both approaches may not always allow identification of incompletely bleached samples (Aitken 1998, Wallinga 2002). For sample GOS4, complete bleaching of the IRSL signal is indicated by consistent ages determined for the silt and sand fraction (Preusser et al. 2003). Similar evidence is available for the Kamaka section by fine-grain dating of six others samples from the same site (Preusser, unpubl. data). IRSL (MAAD) and IR-RF dating confirm the IRSL (SAR) age of sample HURL1 (Erfurt et al. 2003). For the Solenberg site, five additional IRSL (SAR) ages for different samples from the same horizon agree within errors with the age of sample SHB1 (Preusser, unpubl. data). In summary, there exists different evidence that the samples were completely bleached. Thus it appears rather unlikely for the samples investigated here that age underestimation by K-feldspars is masked by the presence of a residual IRSL signal prior to deposition.

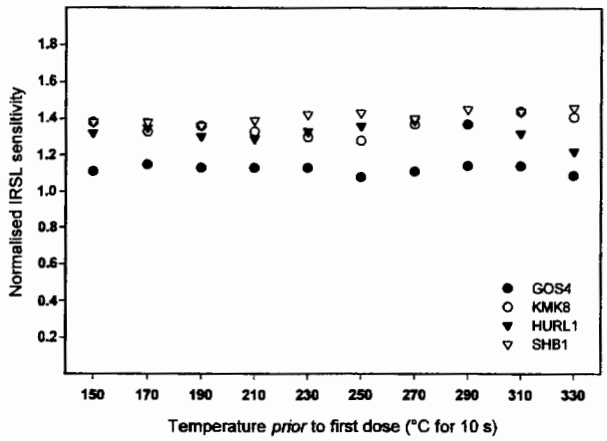

Figure 4.
IRSL response to a fixed dose as a function of the maximum temperature prior to dosing for samples from the four different sites investigated. Results are normalised to the luminescence response to the same dose in a second cycle, where all samples already experienced a heating to 290°C. In contrast to previous experience (Wallinga et al. 2000a), none of the samples showed any increase in normalised IRSL.

All the samples investigated, especially the two from Kamaka, show relatively high recuperation signals of up to 10 %. Recuperation probably results from thermal transfer of electrons (Rees-Jones and Tite 1994) and Wallinga et al. (2000b) demonstrated that the amount of recuperation is a function of preheat temperature. This is confirmed for sample GOS4 by the slight increase of recuperation with preheat temperature (Fig. 1). Anyhow, the influence on the palaeodose determination is small or even negligible as indicated for the samples investigated (Fig. 1).

Wallinga et al. (2000a) gave experimental evidence that the age underestimation of their samples from the Rhine-Meuse delta is probably caused by an increase in the trapping probability due to preheating. They undertook an experiment in which aliquots were bleached, heated to various temperatures between 25 and 275°C, given a fixed beta dose and then preheated to 290°C. The IRSL signal was then measured and this signal was normalised by giving the same beta dose and remeasuring the IRSL signal. In this experiment the normalised IRSL sensitivity increased when the temperature prior to the first dose was above 200° C, implying that the electron trapping probability had risen (Fig 5 of Wallinga et al. 2000a). Repeating this experiment for the samples investigated here reveals, however, that the IRSL sensitivity does not change with temperature prior to the first dose over the range from 150 to 330°C (Fig.

4). Thus the phenomenon that is likely to explain the previously observed age shortfall of K-feldspars from the Rhine-Meuse system, a significant change of trapping probability induced by preheating the natural sample, is not present in the samples from the forelands of the European and New Zealand Alps.

Conclusions

The case studies presented here imply that the modified SAR protocol for feldspars allows reliable dating of samples from both investigated areas. The discrepancy with previous experience (Wallinga et al. 2001) is explained by the different IRSL properties of the samples – in particular the fact that no change in the electron trapping probability is observed. It is, however, impossible to judge if this dating approach can be used for samples from other geological areas as well. There appear to be three criteria for identifying K-feldspars that are suitable for SAR dating:

1) Absence of anomalous fading. From present experience, fading is apparently linked to the presence of volcanic feldspars.
2) The ability to accurately recover the value of a given radiation dose.
3) Absence of any change in trapping probability as a function of temperature prior to dosing.

If these criteria are fulfilled one should expect to get a reliable palaeodose determination using the SAR protocol presented here. It is, however, necessary to apply the presented methodology to samples from other environments where independent age control is available.

Acknowledgements

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Reviewer

G. Duller

Comments

The reliability of luminescence ages based on feldspars continues to be a source of debate. The paper by Wallinga et al. (2001) contains compelling evidence to suggest that, at least for the sites that they studied, feldspars are not a reliable chronometer. However, experience elsewhere suggests that some feldspar ages are accurate. The dilemma we then face is how to determine whether a feldspar age is reliable when no independent chronology is available. This paper by Preusser is important firstly in providing further evidence that some feldspar ages are reliable, and secondly in giving a number of suggestions for tests that can be undertaken to assess the quality of the ages produced.