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Conventional IRSL dating of Romanian loess using single aliquots of polymineral fine grains

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HIGHLIGHTS

► SAR-IRSL investigations of polymineral fine grains.
► Dose recovery dependence on preheat temperature, given dose and bleaching procedure.
► Thermally unstable contributions in the absence of high preheats.
► Fading corrected ages obtained using a 250 °C preheat agree with quartz-OSL ages.
► Agreement is questionable due to poor dose recovery & fading correction limitations.

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ABSTRACT

Investigations of the luminescence characteristics of polymineral fine grains extracted from the loess-palaeosol sequence near Mircea Vodă (SE Romania) were performed using IR stimulation at 50 °C in a single-aliquot regenerative-dose (SAR) protocol. Initial sensitivity changes were observed to influence the ability to measure a given dose. They are shown to depend on preheat temperature, bleaching procedure and size of the given dose. An apparent influence of the preheat temperature on equivalent dose and fading corrected ages is documented through thermal stability experiments. These suggest that, in the absence of a sufficiently high preheat treatment, thermally unstable components contribute to the IRSL signal leading to significant age underestimation. When a high preheat temperature is used (250 °C for 60 s), the age results are in good agreement with previously obtained quartz-OSL ages for material deposited during the last glacial period. However, this agreement may be caused by initial sensitivity changes and limitations of the fading correction.

1. Introduction

This study documents the luminescence characteristics of polymineral fine grains extracted from the loess-palaeosol sequence near Mircea Vodă (SE Romania). This section was previously investigated by Timar et al. (2010) and Timar-Gabor et al. (2011) by applying optically stimulated luminescence (OSL) dating to silt (4–11 μm) and sand-sized (63–90 μm) quartz. Although for both grain-size fractions they obtained age results constraining the uppermost L1/S1 loess-palaeosol units to the last glacial/interglacial cycle, the coarse-grained quartz ages were between 20 and 70% higher than those obtained on the fine-grained quartz. As such, there is considerable uncertainty regarding the formation of a weakly developed palaeosol in the L1 unit (MIS 3 or 5). For samples collected from the lower loess units (L2, L3 and L4) the ages obtained using both grain-size fractions of quartz were interpreted as underestimates of the true age (Timar-Gabor et al., 2011). Interestingly, both quartz fractions passed the procedural tests of the single-aliquot regenerative-dose (SAR) protocol (i.e. recycling ratio, recuperation and dose recovery). Both the age-discrepancy and underestimation remain to be understood.

IRSL signals from silt-sized polymineral grains are investigated here in the hope that they may allow improvement in our understanding of the discrepancy between the two sets of quartz-based ages. Since IRSL signals from feldspars saturate at higher doses than OSL signals from quartz, feldspar-based ages could also extend

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the chronology of stratigraphic units for which quartz-OSL ages are interpreted as underestimates.

2. Materials and methods

This study uses archived material of samples from the previous studies by Timar et al. (2010) and Timar-Gabor et al. (2011). The reader is referred to their works for a complete description of the study site and sample collection. The samples are listed in order of depth in Table 1. Polymineral fine grains (4–11 μm) were separated using conventional sample preparation techniques (Lang et al., 1996; Frechen et al., 1996). Part of the material was further prepared for the extraction of fine-grained quartz (Timar et al., 2010) while the rest of the material was archived and subsequently used in this study. Aliquots were prepared by settling the polymineral fine grains in acetone on aluminium discs.

All luminescence measurements were performed using a Risø TL/OSL-DA-15 reader. Infrared stimulation used infrared (IR) LEDs emitting at 875 nm and IRSL signals were detected in the blue region through a BG39/Corning 7-59 filter combination (Bøtter-Jensen et al., 2003). A conventional IRSL approach is investigated using a single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) involving IR stimulation for 100 s at 50 °C. We used the same thermal treatment prior to measuring the response to both regenerative and test doses (Huot and Lamothe, 2003; Blair et al., 2005). The size of the test dose was 10 Gy in all experiments. Each measurement of the response to the test dose was followed by stimulation with the IR diodes for 40 s at 290 °C. The signals used

Table 1
Summary of calculated dose rates, equivalent doses (D_e), uncorrected ages, fading rates (g_{2days}), fading-corrected ages, and random (σ_r), systematic (σ_s) and total uncertainties (σ_{tot}). Dose rates were obtained using the radionuclide activity concentrations reported by Timar et al. (2010, their Table S3) and the conversion factors of Adamiec and Aitken (1998). Correction of alpha, beta and gamma contributions for the effect of moisture used a time-averaged water content of 20 ± 5% (Balescu et al., 2003). A mean *a*-value of 0.08 ± 0.02 was used (Rees-Jones, 1995). Fading measurements were performed following the procedure proposed by Auclair et al. (2003; see also Basarin et al., 2011). The response to a constant regenerative-dose (25 Gy) was repeatedly measured for various storage times (from ∼0.14 h to ∼21 h) after the preheating of the regenerative-dose. Fading rates were then calculated following Huntley and Lamothe (2001) and normalised to a measurement delay time of 2 days after irradiation. The OSL data for fine-grained quartz are taken from Timar et al. (2010). Uncertainties on the age results were obtained using the error assessment system proposed by Aitken and Alldred (1972) and Aitken (1976). The uncertainties mentioned with the dose rates, D_e's and g_{2days}- values are random; all uncertainties represent one sigma.

Sample	Measurement preheat	Dose rate (Gy/ka)	D_e (Gy)	$Age_{uncorr.}$ (ka)	g_{2days} (%/decade)	$Age_{corr.}$ (ka)	σ_r (%)	σ_{sys} (%)	σ_{tot}	
									(%)	(ka)
GLL-071801	IRSL-115	3.20 ± 0.05	25.2 ± 0.2	8	3.6 ± 0.1	11	3	14	14	2
	IRSL-250		41 ± 3	13	1.9 ± 0.4	15	8		16	3
	OSL	2.85 ± 0.05	24.9 ± 0.4	9			2	14	15	1
GLL-071802	IRSL-115	3.22 ± 0.06	33.1 ± 0.2	10	3.4 ± 0.1	14	3	14	14	2
	IRSL-250		49 ± 1	15	2.3 ± 0.3	19	5		15	3
	OSL	2.87 ± 0.05	32 ± 1	11			3	14	15	2
GLL-071803	IRSL-115	3.46 ± 0.05	44.6 ± 0.4	13	3.4 ± 0.2	18	3	14	15	3
	IRSL-250		67 ± 1	19	1.65 ± 0.04	23	2		15	3
	OSL	3.08 ± 0.05	64 ± 2	21			3	14	15	3
GLL-071804	IRSL-115	314 ± 0.05	62 ± 1	20	3.1 ± 0.1	26	2	14	15	4
	IRSL-250		105 ± 2	33	1.3 ± 0.3	38	4		15	6
	OSL	2.78 ± 0.04	114 ± 2	41			2	14	14	6
GLL-071805	IRSL-115	3.40 ± 0.07	82 ± 1	24	3.2 ± 0.1	33	3	14	15	5
	IRSL-250		148 ± 5	43	2.0 ± 0.6	52	7		16	9
	OSL	3.01 ± 0.06	156 ± 3	52			3	15	15	8
GLL-071806	IRSL-115	3.50 ± 0.05	94 ± 1	27	3.3 ± 0.1	37	2	15	15	5
	OSL	3.10 ± 0.04	190 ± 3	61			2	15	15	9
GLL-071807	IRSL-115	3.58 ± 0.05	99 ± 2	28	3.6 ± 0.1	39	3	15	15	6
	IRSL-250		174 ± 18	49	0.8 ± 0.5	52	12		19	10
	OSL	3.16 ± 0.05	197 ± 3	62			2	15	15	9
GLL-071808	IRSL-115	3.72 ± 0.05	102 ± 1	27	3.7 ± 0.1	39	2	15	15	6
	IRSL-250		191 ± 5	51	2.0 ± 0.5	62	6		16	10
	OSL	3.30 ± 0.05	209 ± 3	63			2	15	15	9
GLL-071809	IRSL-115	3.69 ± 0.04	111 ± 1	30	3.4 ± 0.1	42	2	15	15	6
	IRSL-250		205 ± 6	56	1.7 ± 0.6	65	7		16	10
	OSL	3.28 ± 0.03	221 ± 5	68			2	15	15	10
GLL-071810	IRSL-115	3.30 ± 0.05	160 ± 3	48	3.5 ± 0.1	68	3	15	15	10
	IRSL-250		382 ± 11	116	2.3 ± 0.3	144	5		15	22
	OSL	2.92 ± 0.04	310 ± 9	106			3	15	15	16
GLL-071812	IRSL-115	3.29 ± 0.06	185 ± 2	56	3.3 ± 0.2	78	3	15	15	12
	IRSL-250		738 ± 132	224	1.3 ± 0.1	254	18		23	59
	OSL	2.91 ± 0.05	430 ± 13	147			3	15	15	23
GLL-071813	IRSL-115	3.30 ± 0.04	194 ± 4	59	3.5 ± 0.1	83	3	15	15	13
	OSL	2.92 ± 0.03	467 ± 11	159			3	15	15	24