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Luminescence dating of fine-grain lacustrine sediments from the Late Pleistocene Unterangerberg site (Tyrol, Austria)

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KEYWORDS

Pleistocene chronology
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anomalous fading
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Abstract

For age determination purposes, the luminescence signals from the fine-grained (4-11 μm) quartz and polymineral fractions of samples taken from a Pleistocene palaeolake site in the Eastern Alps of Austria were investigated. Optically stimulated luminescence (standard blue optical luminescence at 125°C (OSL), infrared stimulated luminescence at 50°C ($IRSL_{50/225}$) and post-infrared stimulated luminescence at 225°C ($pIRIR_{225}$)) were measured and analysed. In order to reveal the potential masking of anomalous fading of the natural signal by incomplete bleaching prior to deposition, anomalous fading and bleaching characteristics were addressed. Anomalous fading tests showed average g-values of <2%/decade for both the $IRSL_{50/225}$ and $pIRIR_{225}$ signals. Bleachability, i.e. the rate and extent of natural signal loss during direct exposure to sunlight was derived experimentally. The bleaching experiment showed rapid and thorough bleachability of the OSL and IRSL signals, and a distinctively slower bleaching of the $pIRIR_{225}$ signal. The comparison of ages calculated from the different measurements shows that both the fading of uncorrected and corrected $IRSL_{50/225}$ ages are consistent within uncertainties with the uncorrected quartz OSL ages. The corrected and uncorrected $pIRIR_{225}$ ages, however, show a large overestimation when compared with the OSL ages. Considering the small fading rates and the relatively poor bleachability of the $pIRIR_{225}$ signal, this may be due to incomplete bleaching. For the $IRSL_{50/225}$ ages the results suggests that the samples are well bleached and do not suffer from anomalous fading.

The results of this study allow to constrain the origin and duration of the Unterangerberg palaeolake to the Middle Würmian (ca. 55 to 40 ka) and hence provide the necessary geochronological framework for environmental and climatic proxy studies of this poorly known time interval prior to the Last Glacial Maximum.

Zur Altersbestimmung pleistozäner Paläoseesedimente aus den Ostalpen wurden die Lumineszenzsignale von polymineralischen sowie Quarz-Feinschluffproben (4-11 μm) untersucht. Dabei wurden blaulichtstimulierte Lumineszenz bei 125°C (OSL), infrarotstimulierte Lumineszenz bei 50°C ($IRSL_{50/225}$) und post-infrarotstimulierte Lumineszenz bei 225°C ($pIRIR_{225}$) gemessen und analysiert. Um eine mögliche Verschleierung des Anomalous Fading-Effekts durch eine unvollständige Bleichung des Materials vor der Ablagerung zu erkennen und zu vermeiden, wurden Anomalous Fading und Bleichungsverhalten untersucht. Fading-Tests sowohl für die $IRSL_{50/225}$ als auch für die $pIRIR_{225}$ ergaben g-Werte von durchschnittlich <2%/Dekade. Die Bleichbarkeit, d.h. Raten und Ausmaß des natürlichen Signalabfalls der Lumineszenzsignale während direkter Sonnenbestrahlung, wurde experimentell ermittelt. Das Bleichungsexperiment zeigt eine schnelle und vollständige Bleichung des Signals von OSL und IRSL, sowie eine vergleichsweise deutlich schlechtere Bleichung des pIRIR-Signals. Im Vergleich zeigen die $IRSL_{50/225}$-Alter mit und ohne Fading-Korrektur eine Übereinstimmung mit den OSL-Altern innerhalb der Fehlerbereiche. Im Gegensatz dazu weisen sowohl Fading-korrigierte als auch Fading-unkorrigierte $pIRIR_{225}$-Alter eine starke Altersüberschätzung im Vergleich mit den OSL-Altern auf. Während die geringen gemessenen Fading-Raten sowie die schlechten Bleichungseigenschaften dieses Signals auf eine unvollständige Bleichung des natürlichen Signals vor der letzten Ablagerung schließen lassen, führen die Ergebnisse des $IRSL_{50/225}$ zu dem Schluss, dass dieses Signal gut gebleicht und nicht von Anomalous Fading beeinträchtigt ist. Die Ergebnisse dieser Studie erlauben es, Alter und Existenzdauer des Paläosees von Unterangerberg in das Mittelwürm (ca. 55 bis 40 ka) zu stellen und bieten somit den geochronologischen Rahmen für Umwelt- und Klimaproxyuntersuchungen dieses noch wenig bekannten Zeitintervalls vor der letzten Maximalvereisung.

1. Introduction

In the European Alps, the Late Pleistocene (Würmian / Würm sensu Penck and Brückner, 1909) is relatively well documented for the Last Glacial Maximum (LGM) and the following Lateglacial (see e.g. reviews by van Husen, 2004; Preusser, 2004; Ivy-Ochs et al., 2008; Kerschner and Ivy-Ochs, 2008). For the time period directly before the LGM, however, climatic and environmental conditions are still poorly known, especially for the Middle Würmian, i.e. Marine Isotope Stage (MIS) 3 (~60 to 30 ka). While this period is characterised by loess-paleosol sequences in the alpine foreland (e.g. Brunnacker, 1983; Fink, 1979; Zöller et al., 1994; Thiel et al., 2011a,b) there is only a handful of fragmented records from alpine sites which offer both palaeoclimatic information and absolute chronologies (e.g. Fliri, 1973; Schlüchter et al., 1987; Preusser, 1999b;

Preusser, 2004; Spötl et al., 2006; Drescher-Schneider et al., 2007; Klasen et al., 2006; 2007; Preusser and Degering, 2007).

In this context, lake sediments represent an important archive as they provide access to a wide range of biotic and abiotic proxies (Birks and Birks, 2006), and are also suitable for luminescence dating (Preusser et al., 2008). In recent years, the method was successfully applied to lacustrine samples, with a regional focus on the Western Alps and their foreland (Preusser, 1999a,b, 2003; Preusser and Degering, 2007; Preusser et al., 2003, 2007; Klasen et al., 2007; Lowick et al., 2010; Lowick and Preusser, 2009, 2011; Lowick et al., 2012; Dehnert et al., 2012; Lukas et al., 2012). These studies highlighted the importance of careful analysis and checking of various luminescence characteristics whilst working in this region. In this context, the sensitivity of the quartz signal, the bleaching characteristics of different signal components, and the detection of anomalous fading are of particular importance for producing reliable depositional ages.

Wintle (1973) first described an athermal loss of the natural luminescence signal in feldspar which is considered to be the result of quantum-mechanical tunnelling (Visocekas, 1985). This so-called anomalous fading takes place during burial over geological times and leads to substantial age shortfall, usually expressed as signal loss (%) per decade (equivalent to the g-value of Aitken, 1998). Although there are a number of studies in which g-values have been successfully used to correct IRSL ages for fading (e.g. Huntley and Lamothe, 2001; Auclair et al., 2003; Buylaert et al., 2007, 2011), the inherent assumption regarding the strictly logarithmic nature of signal loss over geological timescales cannot be verified and thus

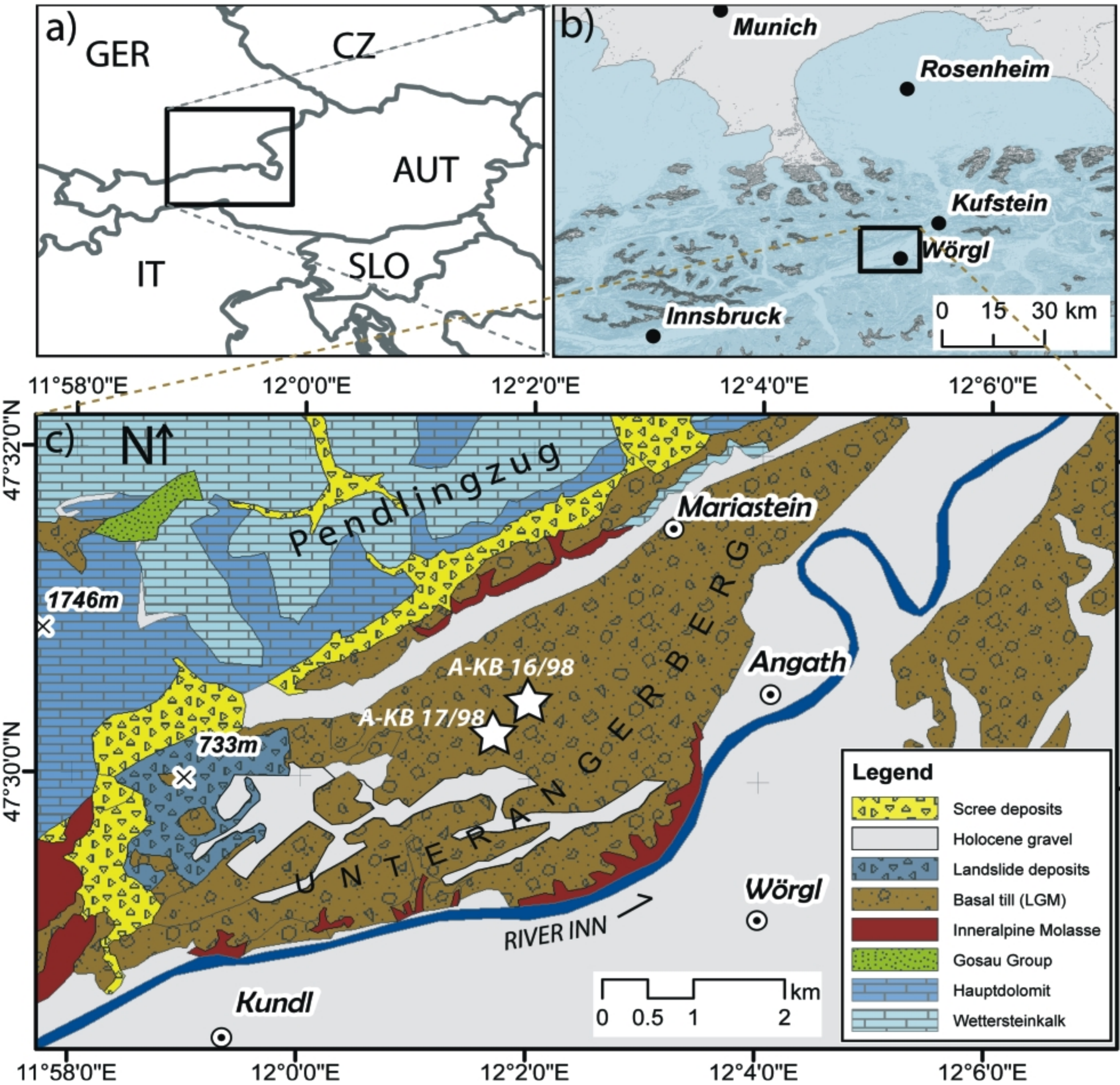

FIGURE 1: Overview of Austria (a), the Lower Inn valley during the LGM (reconstructed LGM ice cover following Ehlers and Gibbard (2004) (b) and simplified geological map of the Unterangerberg terrace (modified after Krenmayr et al., 2007; 2008) showing the location of the two studied drill cores (white asterisks) (c).

questions the reliability of laboratory fading experiments as representative of natural fading. Furthermore, Huntley and Lamothe (2001) stated that their fading correction cannot be used beyond the linear part of the growth curve, although Buylaert et al. (2011) could successfully correct beyond this point. However, Wallinga et al. (2007) found that, even on the linear part of the curve, fading-corrected ages were not always consistent with independent age control. A modelling approach for fading correction beyond the linear part was proposed by Kars et al. (2008). With these problems in mind, Thomsen et al. (2008) proposed an elevated temperature IRSL - the so-called post-IR IRSL or pIRIR - in order to circumvent anomalous fading. While some following studies showed that this pIRIR at 225°C or 290°C signal is not or only slightly affected by anomalous fading (e.g. Buylaert et al., 2009; Thiel et al., 2011a, b; Wacha and Frechen, 2011), the signal appears to be more susceptible to thermal transfer and incomplete bleaching, resulting in relatively large residuals (e.g. Buylaert et al., 2011; Stevens et al., 2011; Lowick et al., 2012).

In this study we investigate the suitability of different luminescence signals for dating Late Pleistocene lacustrine deposits from a site in the Eastern Alps of Austria. This is of particular interest as paleolake sediments are common in the Alps, but rarely contain organic material or are too old for radiocarbon dating. The results are part of a broader study of sediment cores from an inner-alpine site, comprising sedimentological, palaeoecological and geochronological analyses (Starnberger et al., 2013).

We analysed the characteristics of the quartz and feldspar signals recorded from these fine-grained samples, and included a post-IR IRSL measurement step in the IRSL-SAR protocol. The latter is considered to detect a signal which is less affected by anomalous fading than the conventional IRSL (Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2011a, Buylaert et al., 2011). To investigate this further, anomalous fading tests were carried out on all samples. In order to ensure that incomplete bleaching does not mask potential anomalous fading, the bleaching characteristics of the different luminescence signals were also investigated. Finally, the quartz-OSL ages were compared with both uncorrected and fading corrected IRSL and pIRIR ages.

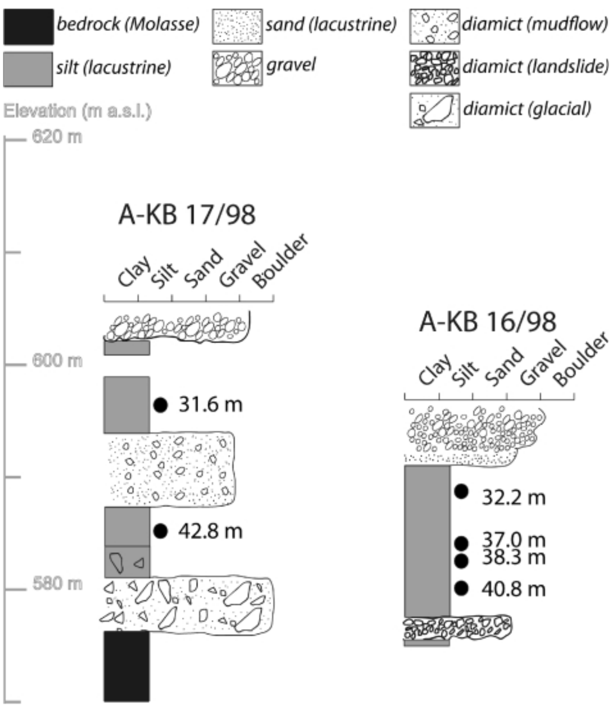

Figure 2: Simplified lithological sketch of the two cores from the Unterangerberg terrace illustrating the stratigraphical positions of the samples used for luminescence dating (black dots).

2. Study site

The Inn valley is one of the large longitudinal valleys in the Alps and is characterised by overdeepening due to repeated Pleistocene glaciations. A drilling at Kramsach, 40 km northeast of Innsbruck, reached the base of the Quaternary fill at 372 m a.s.l., i.e. ~180 m below today's valley floor (Preusser et al., 2010). Valley terraces are a characteristic morphological element of the central and lower Inn valley (e.g. Gnadenwald, Oberangerberg and Unterangerberg terraces). At least partly composed of Pleistocene sediments these terraces are all covered by till of the LGM (Fliri, 1976; Patzelt and Resch, 1986).

Our study site, the terrace of Unterangerberg, is located east of the city of Wörgl (47.49° N, 12.06° E), and stands approximately 150 m above the valley floor. Quaternary sediments overlie marls, calcareous sandstones and breccias of the Upper Oligocene Angerberg Formation (Ampferer, 1922; Ortner and Stingl, 2001; Ortner, 2003) (Fig. 1). The Quaternary sediments of the terrace, studied by Ampferer and Ohnesorge (1909) and Heissel (1951, 1955) remained poorly known until recently due to the lack of suitable outcrops. Between 1995 and 2006 geophysical investigations and a series of drillings under the supervision of the Brenner-Eisenbahn-Gesellschaft (BEG) were performed as part of a tunnel prospection project. The results show a complex subsurface topography of swells and troughs filled by Pleistocene sediments. The samples analysed in this study were obtained from two of these cores penetrating lacustrine sediments intercalated with diamict and gravel (Fig. 2).

3. Methods

3.1 Sample preparation and luminescence measurement facilities

Samples were collected from undisturbed sections of ~11 cm diameter cores which had been stored in a repository under normal daylight conditions. All further analyses were made under subdued red light conditions in the laboratory. The outermost, light-exposed parts of the samples were removed before the removal of carbonate (using 10% hydrochloric acid) and organic content (10% hydrogen peroxide). Samples were washed with distilled water after every treatment step. The 4-11 µm silt fraction was extracted by repeated washing and centrifuging following Frechen et al. (1996). Samples used for analysing the fine-grained quartz signal were treated with 30% hexafluorosilicic acid (H_2SiF_6) for one week in order to remove any feldspar grains (Roberts, 2007). Finally, measurement aliquots were produced by mounting the sediment on 9.7 mm diameter aluminium discs (polymineral: 1 mg/disc; quartz: 2 mg/disc) using an acetone suspension. Luminescence measurements were made using an automated Risø TL/OSL DA-15 Reader with a calibrated $^{90}Sr/^{90}Y$ beta source (~0.08 Gys^{-1}) and a Risø TL/OSL DA-20 Reader with a calibrated $^{90}Sr/^{90}Y$ beta source (~0.10 Gys^{-1}). The quartz-OSL signal was stimulated using blue light (470 nm) emitting diodes and luminescence was detected using a 7.5 mm Hoya U340 filter. The feldspar signal from fine-grained polymineral samples was stimulated using infrared light diodes transmitting at 870 nm and recorded in the blue-violet spectrum using a Schott BG39 / Corning 7-59 filter combination. For all measurements heating was at 5°Cs^{-1} in a nitrogen atmosphere.

3.2 Dose rate determination

Concentrations of the elements relevant to dose-rate calculation were determined using inductively coupled plasma mass spectrometry (ICP-MS; Preusser and Kasper, 2001) (Tab. 1). Alpha, beta and gamma dose rate conversion factors were taken from Adamiec and Aitken (1998). In order to account for the impact of alpha particles on the dose rate and in absence of an available calibrated alpha source, mean alpha efficiency values (a-values) of 0.03±0.02 (fine-grain quartz) and 0.07±0.2 (fine-grain polymineral) were taken from the literature (Rees-Jones, 1995; Lang et al., 2003; Preusser, 2003; Mauz et al., 2006). Cosmic dose rate values were calculated after Prescott and Stephan (1982). Due to the fact that the sediment in the cores was entirely desiccated, an estimated average water content value of 27% was calculated from sporadic measurements which were made during the drilling of the cores. To take past changes of moisture into account, an uncertainty of 10% was used for all samples. A potential effect of repeated freezing and melting of the sediment's pore water on the dose rate cannot be excluded.

3.3 Luminescence measurements

3.3.1 OSL-SAR protocol

For measuring the fine-grained (4-11 µm) quartz fractions, the OSL-SAR protocol as introduced by Murray and Wintle (2000, 2003) with the modifications suggested by Duller (2003) was applied. Following 100 s stimulation at 90% power, the equivalent dose (De) values were calculated using the first 0.3 s of the decay curve, with a background subtraction of the last 10 s. For equivalent dose (De) estimations, a single saturation exponential function was used. Preheat tests were performed on every sample and preheat temperature plateaus could be identified between ~220°C and ~280°C (Fig. 3). For all subsequent measurements preheat was held at 260°C for 10 s and cutheat was at 160°C. Recuperation values were below 5% and recycling ratios yielded values consistent with unity (±10%). The OSL IR depletion ratio was determined to identify any feldspar contamination in the quartz samples. This was done using a post-IR-OSL within the SAR-protocol by repeating the recycled point at the end of the sequence, but introducing an IRSL shine of 100 s at room temperature prior to the measurement of OSL (Duller, 2003). The resulting sensitivity corrected Lx/Tx signal intensity is compared with that from the proceeding regeneration dose point (equation 1) and, where no feldspar is present, results in values close to unity. OSL IR depletion ratio results (Tab. 2) yield values around unity for all samples, thus indicating that the signal is not affected by feldspar contamination.

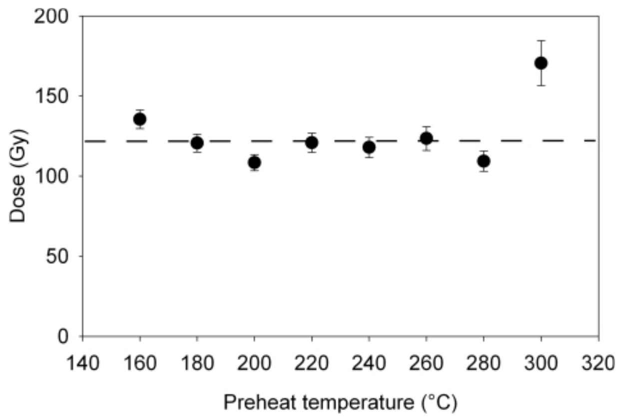

Figure 3: Representative results from a preheat plateau test performed on fine-grained quartz aliquots from sample A-KB 17/98 (31.6 m). Data points represent average values from three aliquots.

$$\frac{Lx(post-IR-OSL)/Tx(OSL)}{Lx(OSL)/Tx(OSL)} \quad (1)$$

3.3.2 pIRIR-SAR protocol

In a second experimental approach, the feldspar signal from

Sample	Depth (m)	U [ppm]	Th [ppm]	K [%]	Dose rate (Gy ka^{-1})	
					Quartz	Feldspar
A-KB16/98	32.2	3.0±0.3	7.9±0.8	2.7±0.3	3.37±0.25	3.70±0.25
	37.0	3.7±0.4	9.1±0.9	2.6±0.3	3.58±0.26	3.98±0.26
	38.3	4.6±0.5	12.0±1.2	2.9±0.3	4.17±0.29	4.67±0.29
	40.8	3.8±0.4	8.6±0.9	2.3±0.2	3.29±0.24	3.68±0.25
A-KB17/98	31.6	3.8±0.4	6.0±0.6	1.7±0.2	2.64±0.22	2.98±0.22
	42.8	2.5±0.3	9.2±0.3	1.9±0.5	2.77±0.21	3.10±0.21

Table 1: Summary of radionuclide concentrations of uranium (U), thorium (Th) and potassium (K) and resulting dose rates for quartz and feldspar fractions of the samples.

Core	Depth (m)	n	OSL IR depletion ratio
A-KB 16/98	32.2	4	0.93±0.02
	37.0	6	1.00±0.04
	38.8	6	0.95±0.02
	40.8	5	0.95±0.03
A-KB 17/98	31.6	6	0.98±0.04
	42.8	6	0.94±0.05

Table 2: OSL IR depletion ratio results of fine-grained quartz samples.

the fine-grained (4-11 µm) polymineral fraction was measured using an IRSL-SAR protocol. Firstly, preheat plateau tests were conducted for the conventional IRSL signal and plateaus were identified between 270 and 310°C. Next, an elevated temperature measurement step at 225°C ($pIRIR_{225}$) following the IRSL stimulation was inserted in the protocol ($IRSL_{50/225}$) (Tab. 3). A preheat temperature of 290°C was chosen and preheat of the test dose measurements was held at the same temperature as used for the natural and regeneration doses (Blair et al., 2005). Recycling ratio values yielded satisfactory results of around unity (±10%). The $IRSL_{50/225}$ and pIRIR225 signals were recorded for 300 s and integrated over the first 5 s, with a background calculated using the last 50 s. For De estimations, a single exponential plus linear function showed the best fit for the dose response curves.

3.4 Dose recovery and bleaching tests

In order to test the ability of the applied SAR-protocols to recover a known laboratory dose, dose recovery tests (Wallinga et al., 2000; Murray and Wintle, 2003) were performed on aliquots from all samples. For this, a known laboratory dose was given to previously bleached and unheated aliquots which were then measured using the SAR-protocol. For fine-grained quartz samples, laboratory bleaching was achieved by blue LED stimulation for 400 s under room temperature, followed by a ~2.8 h pause, then a second irradiation for 400 s (Murray and Wintle; 2003). For the $IRSL_{50/225}$ and $pIRIR_{225}$ measurements on the polymineral fraction, a similar laboratory bleaching procedure was applied, with two IR stimulations at room temperature for 400 s (J. Wallinga, pers. comm.). This, however, resulted in large residual doses for the $pIRIR_{225}$.

To investigate the bleaching behaviour of the OSL, $IRSL_{50/225}$, and $pIRIR_{225}$ signals under natural daylight conditions and to find a suitable bleaching procedure for the $IRSL50/_{225}$ and $pIRIR_{225}$ signals, the following experimental setup was used: a total of 63 (30 fine-grained polymineral and 33 fine-grained quartz) aliquots was exposed to direct sunlight on a cloudless August afternoon (with mean global radiation for August being ~160 Wm^{-2} at ~47° 16' N, 11° 24' E, ~580 m a.s.l.; Krismer, 2004) for different time durations between 60 s and 2 h. Following this, the residual natural signals were measured by obtaining a D_e using the protocols as outlined above.

Step	Treatment	Observed
1	Give dose[a]	
2	Preheat (290°C for 60s)	
3	IR stimulation (50°C for 300s)	$L_x(IRSL_{50/225})$
4	IR stimulation (225°C for 300s)	$L_x(pIRIR_{225})$
5	Give test dose	
6	Preheat (290°C for 60s)	
7	IR stimulation (50°C for 300s)	$T_x(IRSL_{50/225})$
8	IR stimulation (225°C for 300s)	$T_x(pIRIR_{225})$
9	Return to 1	

Table 3: pIRIR-SAR protocol with an elevated temperature step at 225°C following IRSL stimulation.

3.5 Anomalous fading tests

Fading tests were carried out in order to detect any signal loss after different storage times. For the polymineral samples four aliquots from each sample were chosen which had already been sensitised during previous De measurements. The same SAR protocol as outlined in table 3 was applied and aliquots were repeatedly irradiated with a fixed regeneration dose between 115 and 151 Gy and a test dose of ~20 Gy. Between steps 2 and 3 of the protocol (Tab. 3), pauses of ~30 min to ~160 h (delayed measurements) were introduced (Auclair et al., 2003). Calculations of g-values, i.e. the loss of the initial laboratory-irradiated signal per decade during deposition (Aitken, 1998), and corrected equivalent doses were done following Huntley and Lamothe (2001).

4. Results and discussion

4.1 Signal intensities, growth curves, and De calculation

The OSL signal obtained from the fine-grained quartz samples has a rapid decay indicating that the signal is dominated by the fast component (Fig. 4). The signal obtained from the quartz samples is bright enough to be used for luminescence dating. Low quartz signal intensities of only a few hundred initial counts or less are often observed in glacial and glacier-associated sediments worldwide, especially from young orogens, and are explained by the small number of sediment reworking cycles they have undergone (Fuchs and Owen, 2008; Preusser et al., 2009; Thrasher et al., 2009). For alpine samples, low quartz sensitivities were reported e.g. by Klasen et al. (2007) and Lukas et al. (2012). As an alternative to quartz in the Alps, Preusser (1999b; 2003) verifies the suitability of the IRSL signal from K-rich feldspars, although in later work low sensitivities have also been encountered for samples from this region (Preusser et al., 2007). The $IRSL_{50/225}$ and $pIRIR_{225}$ signals observed from the Unterangerberg samples show generally high initial intensities (Fig. 5) and the $pIRIR_{225}$ signal is typically brighter than the $IRSL_{50/225}$ signal. This observation was also made by e.g. Thiel et al. (2011b) when working with alpine foreland loess.

To investigate the saturation behaviour of the OSL, $IRSL_{50/225}$ and $pIRIR_{225}$ signals, the dose response curves of each aliquot were fitted with a single saturating exponential function. To ensure that the signal used is below ~85% of saturation, Wintle and Murray (2006) recommend the use of only De values <$2D_0$ for dating. For the Unterangerberg quartz fraction, 2D0 values range between ~250 and 450 Gy (Fig. 4) and the De values derived for age calculation are clearly lower (Tab. 4), ranging between ~120 and 200 Gy.

For $IRSL_{50/225}$ and $pIRIR_{225}$, $2D_0$ values are generally higher than the respective values from OSL, with values of ~500 Gy ($IRSL_{50/225}$) and ~512 Gy ($pIRIR_{225}$) obtained by fitting the regeneration dose response curves to a single saturation exponential function (Fig. 5). As for the OSL, natural De values for the analysed samples remain below these values, ranging from ~130-210 Gy ($IRSL_{50/0225}$) and ~150-330 Gy ($pIRIR_{225}$). However, the abovementioned saturation doses are still relatively low when compared e.g. with values from the northern alpine foreland, where saturation beyond ~800 Gy (Dehnert et al., 2012) and up to ~2600 Gy (Thiel et al., 2011b) are reported.

Equivalent dose values for OSL, $IRSL_{50/225}$ and $pIRIR_{225}$ Lx/Tx values were used for age calculations and are reported in Table 3. Between six and eight aliquots were accepted per sample. For $2D_0$ calculations, growth curves were fitted to a single saturation exponential function.

4.2 Dose recovery and bleaching characteristics

Dose recovery tests performed on the fine-grain quartz samples yielded measured/given dose ratio values close to unity (±10%) after laboratory bleaching (Fig. 6), showing that this bleaching procedure was appropriate.

The bleaching experiment revealed a good bleachability of the natural quartz-OSL signal, especially when compared to the $IRSL_{50/225}$, and $pIRIR_{225}$ signals from the fine-grained polymineral fraction (Fig. 7). The OSL signal bleached rapidly and was reduced to ca. 5% after 60 s of sunlight exposure and fur-

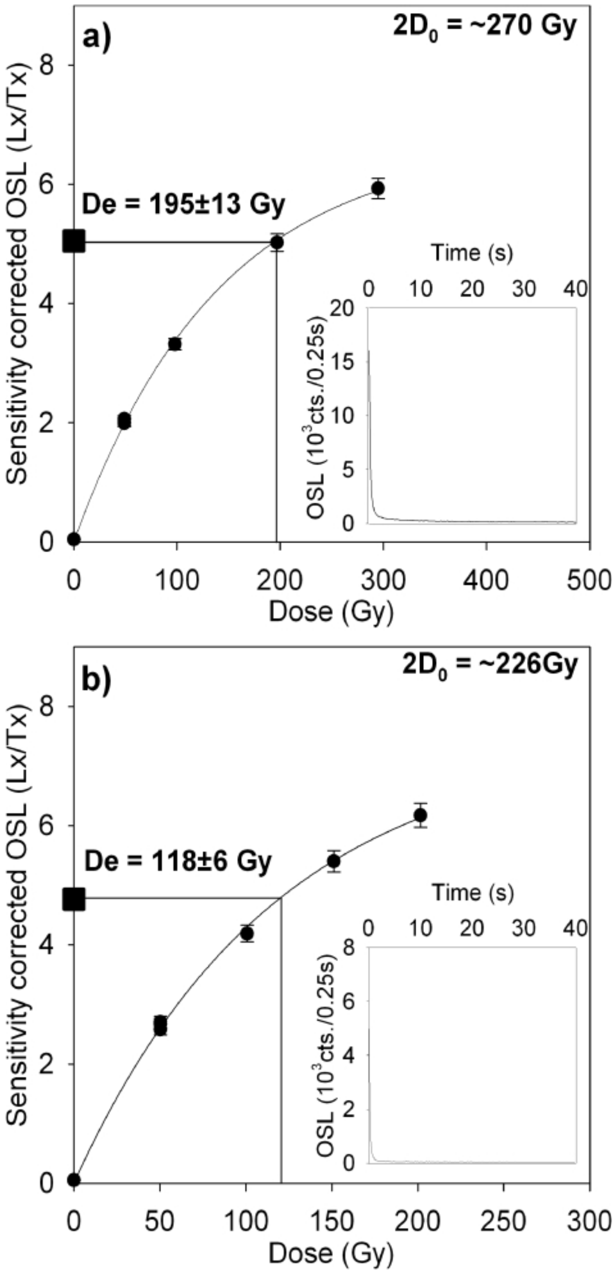

Figure 4: Representative OSL dose response curves and natural decay (insets) curves of samples (a) A-KB 16/98 38.3 m and (b) A-KB 17/98 31.6 m. $2D_0$-values were derived from fitting a single saturation exponential function to the regenerations dose points.

Sample		De [Gy]			g [%/decade]		Uncorrected age [ka]			Corrected age [ka]	
Core	Depth [m]	OSL	$IRSL_{50/225}$	$pIRIR_{225}$	$IRSL_{50/225}$	$pIRIR_{225}$	OSL	$IRSL_{50/225}$	$pIRIR_{225}$	$IRSL_{50/225}$	$pIRIR_{225}$
A-KB 16/98	32.2	169±3	179±9	317±22	1.7±0.8	0.3±0.8	50±4	48±4	86±6	59±7	87±7
	37.0	152±8	164±8	237±11	0.4±0.5	0.6±0.6	42±4	41±3	60±4	43±4	63±5
	38.3	201±8	211±16	332±18	1.3±0.5	0.7±0.6	48±4	45±4	65±4	51±3	71±6
	40.8	148±3	153±7	260±17	-2.1±0.6	1.1±0.7	45±4	42±3	71±7	42±3	79±9
A-KB 17/98	31.6	121±4	139±12	149±6	0.6±0.4	-0.2±0.6	46±4	47±4	50±4	49±4	51±5
	42.8	151±8	164±4	269±15	1.5±0.3	-0.4±0.3	54±5	53±4	87±8	61±6	87±8

Table 4: OSL, $IRSL_{50/225}$ and $pIRIR_{225}$ equivalent doses (De) and respective uncorrected ages, calculated g-values and fading-corrected $IRSL_{50/225}$ and $pIRIR_{225}$ ages.

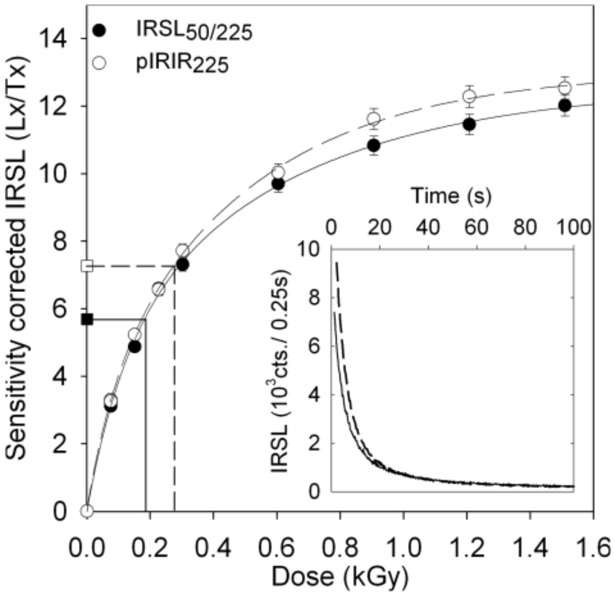

Figure 5: Representative dose response curves of the $IRSL_{50/225}$ (solid line) and $pIRIR_{225}$ (dotted line) signals and natural decay curves (inset) for sample A-KB 16/98, 38.3 m, with regeneration doses up to 1.5 kGy.

ther reduced to background level after ~10 min. The $IRSL_{50/225}$ and $pIRIR_{225}$ signals, however, lost only ca. 30% and ca. 15 % respectively, after the first minute and still contained ~10 and 30% of the signal after 10 min. Furthermore, whilst the $IRSL_{50/225}$ signal was bleached to only 4% of its initial level after 20 min, the $pIRIR_{225}$ signal needed 2 h of sunlight to be reduced to the same degree. The experiment demonstrates that the $pIRIR_{225}$ signal is harder to bleach than the $IRSL_{50/225}$ signal. However, it also demonstrates that for these samples, residual values are reduced to less than 5% of the natural signal after sunlight bleaching.

For the fine-grained polymineral samples, where laboratory bleaching was sufficient for the $IRSL_{50/225}$ signal but not for the $pIRIR_{225}$ signal (measured/given ratios were >1 for all samples),

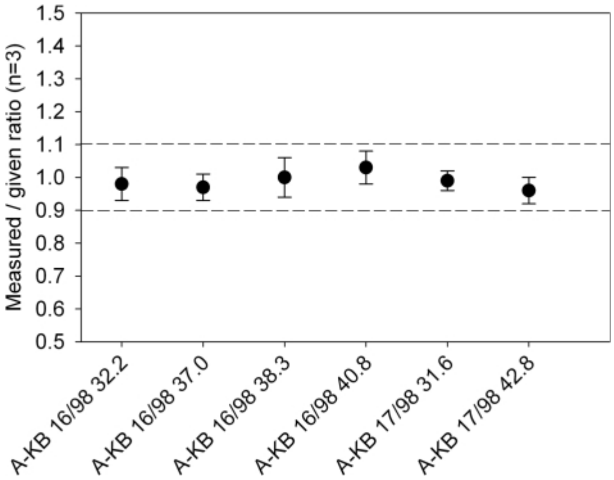

Figure 6: Results from dose recovery tests performed on fine-grained quartz samples taken from the Unterangerberg cores A-KB 16/98 and A-KB 17/98. The laboratory dose given to each sample was close to its natural dose.

it was decided to obtain bleaching by direct sunlight exposure of the aliquots for at least 2 h. Consequently, dose recovery tests performed on sun-bleached samples yielded measured/given ratio values close to unity (within 10% uncertainty) for the $IRSL_{50/225}$ and $pIRIR_{225}$ signals.

Different bleaching behaviour of quartz and feldspar luminescence signals when exposed to direct sunlight was also reported by Godfrey-Smith et al. (1988). They found that the quartz signal bleaches much faster (reduced to ~0.1 % after 6 min) than the feldspar signal (reduced to ~0.5 % only after 2 h). This was later supported e.g. by Thomsen et al. (2008) for samples from various Eurasian sites. Bleaching tests performed on alpine samples with results similar to those from Unterangerberg were reported by Preusser (1999a), Klasen et al. (2006, 2007) and Dehnert et al. (2012).

The experimental setup applied in this study simulated bleaching under ideal conditions, i.e. direct summer-sunlight exposure for several hours. However, a more realistic fluvial transport and lacustrine deposition scenario may include factors such as turbulent water transport, cloudiness, particle cohesion and low incoming radiation. Consequently, longer bleaching times and potential incomplete bleaching, especially of the $pIRIR_{225}$, under natural conditions should be considered.

4.3 Anomalous fading and dating results

Fading tests gave average g-values (n=4 per sample) ranging from -2 to 1.7 % for $IRSL_{50/225}$ and from ~-0.4 to 1.1 % for $pIRIR_{225}$ measurements (Fig. 8; Tab. 4). The negative g-values are the result of a scatter in the fading measurements and treated as a zero value. Thiel et al. (2011c), for example, also reported negative g-values of up to ~-6 %/decade for almost all of their $IRSL_{50/225}$ values from Japanese loess which they could not explain. In their study, they did not calculate fading-corrected ages for these cases.

At Unterangerberg, the low g-values and the general consistency of the uncorrected and corrected $IRSL_{50/225}$ ages with the quartz-OSL ages (Fig. 9a) altogether indicate that anomalous fading does not significantly affect the $IRSL_{50/225}$ ages. Both uncorrected and fading-corrected pIRIR225 ages, however, tend to overestimate OSL ages (Tab. 4; Figs. 9a, b).

The results are similar to recent studies from the Western Alps: in the Swiss Jura Mountains, g-values between 0.9 and 1.9% for the conventional IRSL were reported by Gaar and Preusser (2012), with a mean of 1.3%. In this study, uncorrected ages fitted best to independently obtained quartz-OSL ages and the authors concluded that anomalous fading is absent in the feldspar signal from that site. Lowick et al. (2012) reported g-values between 1 and 4%/decade from different sites in the Swiss Alps and alpine foreland, using pIRIR signals at 225°C and 290°C. For these samples they could not find any differences in g-values for the $IRSL_{50/225}$, $pIRIR_{225}$ and $pIRIR_{290}$ signals Corrected $IRSL_{50/225}$, and both uncorrected and corrected $pIRIR_{225}$ ages were reported to overestimate independent ages, whilst the uncorrected $IRSL_{50/225}$ ages agreed with independent ages. Further, relatively large residual doses, especially of the $pIRIR_{290}$ signals were detected following 24 h exposure to a daylight lamp, and explained by the hardto-bleach nature of the elevated temperature traps. As a consequence, Lowick et al. (2012) stressed the need to identify and separate the influences of both anomalous fading and residual doses / incomplete bleaching of waterlain samples when working on alpine samples.

4.4 Geological and paleoenvironmental implications

OSL and $IRSL_{50/225}$ dating of the two cores allows constraining of the duration of lacustrine sedimentation at the Unterangerberg terrace to ~55 to 40 ka, i.e. MIS 3. During this time, sedimentation in the Lower Inn valley took place under distinctively different conditions compared to today. This lacustrine basin owes its formation and long persistence to the pronounced pre-Quaternary basement topography and its separation from the main valley by two landslides (Gruber, 2008; Gruber et al., 2009; Starnberger et al., 2013). The latter are among the oldest of such events dated in the Alps.

MIS 3 is known for its high-amplitude climatic variability as prominently shown by the oxygen isotope record from Greenland ice cores (e.g. Wolff et al., 2010). This climatic pattern has also been identified in fragmentary proxy records from the foreland of the Western Alps reflecting severe cold (stadial) and mild (interstadial) intervals, e.g. in Gossau (Jost-Stauffer, 2001, 2005; Preusser et al., 2003) and Niederweningen (Drescher-Schneider et al., 2007; Anselmetti et al., 2010; Dehnert et al., 2012). Unterangerberg is not only the first site in the Eastern Alps to provide detailed insights into the paleoenvironment and climate during MIS 3 using well-dated, high-quality (core) samples, but also one of the first of such sites inside the Alps. Further details are provided by Starnberger et al. 2013.

5. Conclusions

The investigations carried out at Unterangerberg have important implications for future luminescence dating in the Eastern Alps. They show that the quartz signal detected from purified fine-grained samples is bright enough for dating and that the standard OSL-SAR protocol can be successfully applied. The quartz-OSL signal was found to saturate at rather high

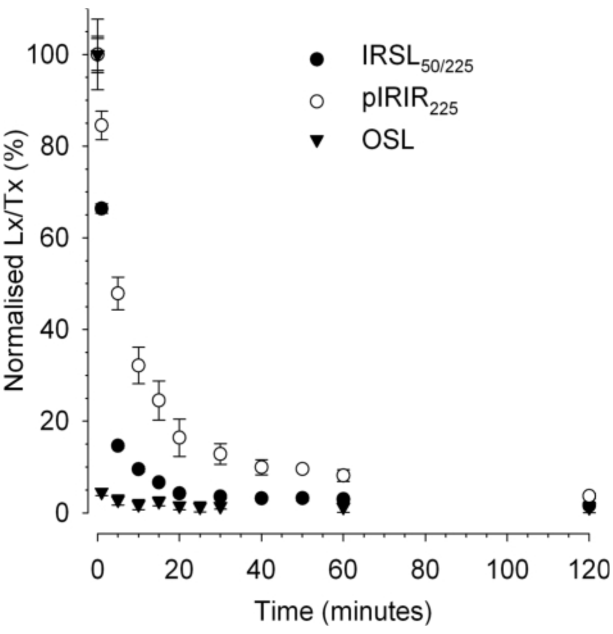

Figure 7: Results from a bleaching experiment performed on fine-grained (4-11 µm) quartz and polymineral fractions from sample A-KB 17/98 (31.3 m depth). Aliquots were exposed to sunlight between 60 s and 2 h before measuring the residual OSL, $IRSL_{50/225}$ and $pIRIR_{225}$ signals. The luminescence signal on the y-axis is normalised to N (natural signal).

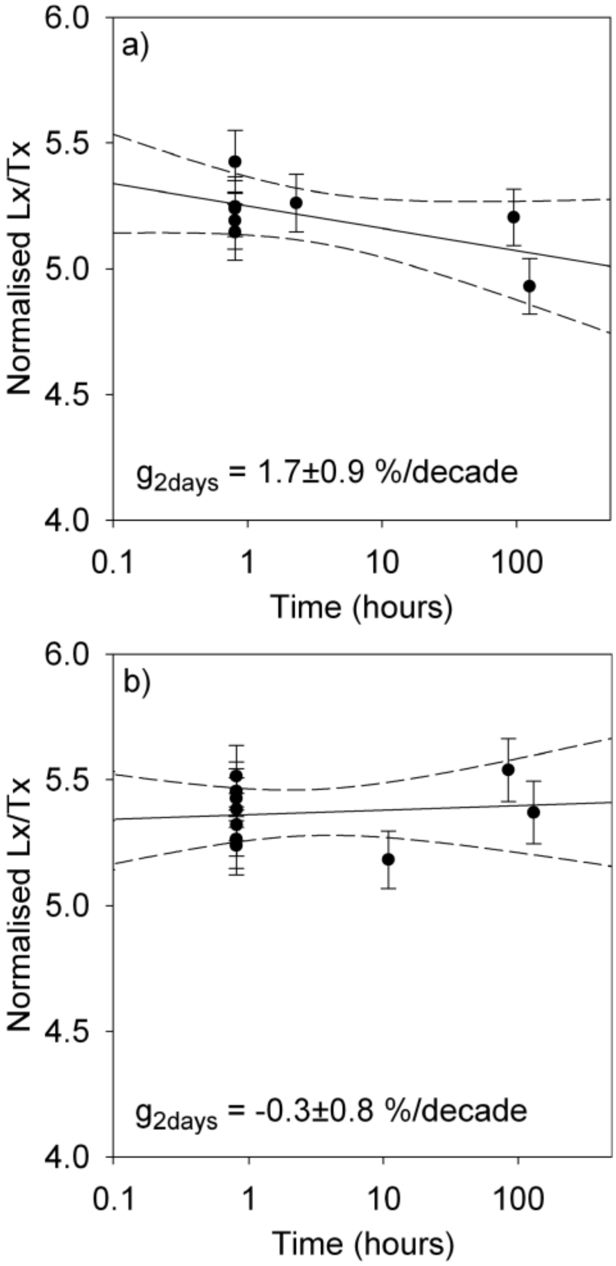

Figure 8: Fading test results for one fine-grained polymineral aliquot of sample A-KB 16/98 (32.2 m). Normalised (a) $IRSL_{50/225}$ and (b) $pIRIR_{225}$ Lx/Tx signal values are plotted against storage time. Dotted lines represent 2 sigma confidence intervals. Maximum storage times are (a) 125 h and (b) 130 h.

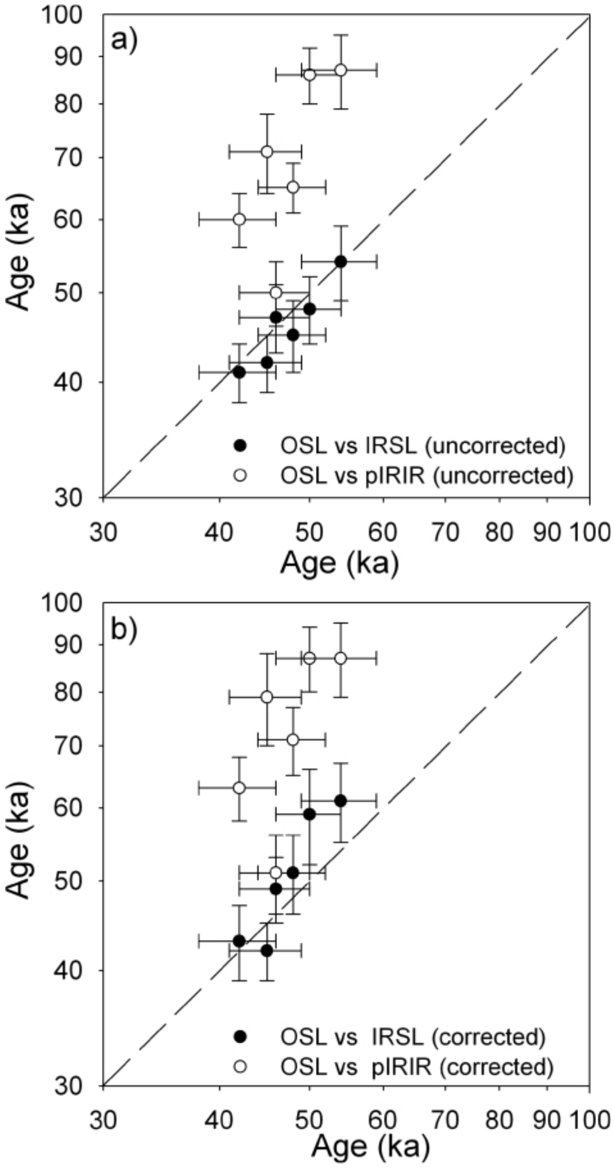

Figure 9: OSL ages compared to (a) uncorrected and (b) fading-corrected $IRSL_{50/225}$ and $pIRIR_{225}$ ages from the same samples. Note double-logarithmic axes.

doses of ~250 Gy which, considering the relatively high environmental dose rates at Unterangerberg (~2.6 to 4.2 Gy / ka), inhibits OSL dating beyond ~60 to 80 ka at this site. For the feldspar signal, saturation levels as well as dose rates indicate a higher theoretical upper dating limit of ~100 to 170 ka. The bleaching experiment revealed good bleaching characteristics for the OSL and $IRSL_{50/225}$ signals, and the comparison of OSL and $IRSL_{50/225}$ ages suggests that the latter signal was completely bleached before the most recent burial. Further, the generally good agreement between OSL and uncorrected $IRSL_{50/225}$ ages indicates that no significant anomalous fading of this signal is present. This is underpinned by laboratory storage tests yielding low fading rates. Corrected $IRSL_{50/225}$ ages result in small age overestimations when compared with the OSL ages. Large overestimations, however, arise when the $pIRIR_{225}$ signal is used for analysis. The bleaching experiment showed that the $pIRIR_{225}$ bleaches slower than the $IRSL_{50/225}$ signal following 2 h exposure to direct sunlight. After that time, however, the residual dose is reduced to <5 % and subsequent dose recovery tests yield good results. This suggests that the large overestimations in the De values calculated using the $pIRIR_{225}$ signal can be attributed to incomplete bleaching prior to the last deposition.

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