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GEOCHRONOMETRIA 38(4) 2011: 432-440
DOI 10.2478/s13386-011-0047-0

Available online at
www.springerlink.com

IRSL AND POST-IR IRSL RESIDUAL DOSES RECORDED IN MODERN DUST SAMPLES FROM THE CHINESE LOESS PLATEAU

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Received 8 October 2010 Accepted 15 April 2011

Abstract: Using a set of modern/young (0 to about 200 years old) dust samples collected from the Chinese Loess Plateau the bleachability of IRSL measured at 50°C (IR_{50}) and post-IR_{50} elevated temperature IRSL (measured at 225°C and at 290°C) is investigated by measuring the apparent (residual) doses recorded by these signals. Doses recorded by quartz OSL are used as a reference. Allowing for differences in dose rates it seems that both IRSL and post-IR IRSL signals yield residual doses that are significantly larger than the doses measured in quartz. These residual doses can be largely explained by thermal transfer caused by preheating. Nevertheless, we advise against the use of a low temperature preheat (<200°C) with IR_{50} to date loess samples because, as has been reported before, the signal appears to be thermally unstable. In general, we conclude that it may not be advisable to apply post-IR IRSL dating to Chinese loess samples where residuals of up to ~20 Gy are a significant fraction of the total dose. However, these residuals quickly become unimportant when dating older samples, and this is the age range in which post-IR IRSL dating is likely to be most useful.

Keywords: modern dust, IRSL, polymineral fine-grains, post-IR IRSL, loess, China.

1. INTRODUCTION

Quartz is generally seen as the preferred dosimeter in luminescence dating studies (Murray and Olley, 2002), especially in the younger age range (Madsen and Murray, 2009). Many studies of Chinese loess have employed quartz optically stimulated luminescence (OSL) to establish a chronology both using coarse (e.g. Stevens *et al.*, 2006; Buylaert *et al.*, 2008; Lai, 2010) and fine (e.g. Watanuki *et al.*, 2003; Lu *et al.*, 2007) grains. Unfortunately Chinese loess (dust) may not contain sufficient coarse quartz grains (at least in the sand fraction), and the isolation of fine-grained quartz is time-consuming (Roberts and Wintle, 2001; Roberts, 2008). In addition, the quartz OSL signal has a limited dose range; because of the high dose rate in loess, quartz cannot usually be used to date older than ~50 ka (D_e >~150 Gy; Buylaert *et al.*, 2007; 2008). Polymineral fine-grains are very easily prepared and are commonly used with infrared (IR) stimulation and a blue detection window to date loessic sedi-

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ISSN 1897-1695 (online), 1733-8387 (print)

ments (e.g. Musson *et al*., 1994; Frechen, 1999; Tsukamoto *et al*., 2001). It is assumed that luminescence emissions from feldspars dominate the signal. It has also been shown that the loess infrared stimulated luminescence (IRSL) signal grows to much higher doses than does the OSL from quartz (e.g. Buylaert *et al*., 2007). However, the luminescence behaviour (such as bleachability, anomalous fading, and sensitivity change) of polymineral fine-grains is not yet well understood, and their reliability for dating remains to be established (Roberts, 2008; Roberts and Wintle, 2003). Other work on polymineral fine-grains has focused on trying to extract a quartz dominated OSL signal from this material with a so-called double-SAR (single aliquot regenerative) dose measurement procedure (Banerjee *et al*., 2001) and detection in the UV spectrum (e.g. Roberts and Wintle, 2001; Zhang and Zhou, 2007; Wang *et al*, 2006; Zhou *et al*., 2010).

Buylaert *et al*. (2007) demonstrated that the IRSL signal measured at 50°C (defined here as IR_{50}) from polymineral fine-grains extracted from the Luochuan type section shows anomalous fading (g_{2days} of ~3%/decade); this required corrections to the apparent age of ~35% using the model proposed by Huntley and Lamothe (2001). Recently, Thomsen *et al*. (2008) identified the potential of stimulating sand-sized grains of feldspar with IR while the sample is held at elevated temperature. They observed a substantially lower fading rate using a post-IR IRSL measurement protocol (in which an IR_{50} is immediately followed by IR stimulation at some elevated temperature) compared to that from a conventional protocol using only an IR_{50} signal. Buylaert *et al*. (2009) showed that these conclusions applied to the natural signal from sand-sized K-feldspar grains, and Thiel *et al*. (2011a) showed that the conclusions could be extended to polymineral fine-grains extracted from European loess.

The potential of SAR based IRSL and post-IR IRSL measurements on polymineral fine-grains extracted from Chinese loess has not yet been fully explored, and there are no studies of the bleachability of the IRSL and post-IR IRSL signals of polymineral fine-grains using modern dust. Feldspars bleach more slowly than quartz (Godfrey-Smith *et al*., 1988; Thomsen *et al*., 2008) and, although complete daylight bleaching of fine-grained dust material is usually assumed, there are suggestions that bleaching processes may be hampered by superficial coatings (as suggested in Prescott (1983) for TL signals of quartz extracted from aeolian sands in Australia). In this paper, we use polymineral fine-grains of modern dust samples collected across the Chinese Loess Plateau to investigate the completeness of bleaching of both the IRSL signal measured with the sample held at 50°C, and the post-IR IRSL signals measured at elevated temperature (225°C and 290°C). All feldspar signals are measured in the blue-violet part of the spectrum. The dose estimates are evaluated by comparison with the OSL results from quartz grains; these are known to be accurate in this age range (Madsen and Murray, 2009). Residual feldspar doses are discussed in terms of thermal transfer and incomplete bleaching. Finally, we address the question of whether a low temperature preheat can be used when dating loess using the IR_{50} signal.

2. SAMPLES AND MEASUREMENT DETAILS

Three modern/young samples collected from the top few cm of apparently undisturbed loess sections in the Chinese Loess Plateau were used in this study, together with three superficial dust samples (roofs of buildings in Xi'an and Beijing, and a car bumper sample) (see **Table 1** for details). The Beijing dust sample (TPU) was collected in April 2007 from the top of air-conditioning units on the roof of the Geography Department of Peking University, and the car bumper sample (CB) was sampled after a ~7 days field trip in the Chinese Loess Plateau in April 2005. Both samples were taken from << 1 mm thick layer of dust. In this study we also make use of two old true loess samples to investigate the stability and preheat dependence of the (post-IR) IRSL signals (sections 3 and 6): sample A42 from the Luochuan section taken in loess layer L9 with an expected age of ~900 ka (Buylaert *et al*., 2007) and sample A7 from the Zhongjiacai section, dated with quartz OSL by Buylaert *et al*. (2008) to 29.0±1.4 ka.

Polymineral fine-grains (4-11 μm) were extracted from all the samples in the usual manner (HCl and H_2O_2 treatment and Stokes' settling) and deposited on aluminium discs (e.g. Frechen *et al*., 1996). About 1 mg of sample was used per disc. Sand-sized (63-90 μm) quartz grains were extracted using wet sieving, HCl and H_2O_2 treatment and etching for 40 min in concentrated HF to remove feldspars. For two superficial dust samples (TPU from the top of a building in Beijing and CB from the car bumper), no sand-sized quartz grains could be extracted. Instead, we extracted fine-grained (4-11 μm) quartz grains using a 3 days hydrofluorosilicic acid etch (Zhang and Zhou, 2007); for sample CB a coarse-silt (36-50 μm) quartz fraction was obtained by wet sieving and a 40 min concentrated HF digestion. After acid treatment all quartz fractions were checked for the presence of a significant IRSL signal. Some 63-90 μm fractions required another 40 min of concentrated HF to obtain clean quartz. Coarse-grained quartz was mounted on stainless steel discs using a 8 mm mask and silicon spray to produce large aliquots. Except for the 4-11 μm CB sample, all the quartz fractions were found to be pure after acid treatment. (The CB sample was measured with a double-SAR protocol (Banerjee *et al*., 2001) to obtain a quartz-dominated signal - see caption **Table 1** for details).

All luminescence measurements were made with a Risø TL/OSL DA-15 reader equipped with IR diodes emitting at 875 nm, blue diodes emitting at 470 nm, and $^{90}Sr/^{90}Y$ beta sources calibrated for fine-grains on aluminium and coarse-grains on stainless steel discs using quartz (Bøtter-Jensen *et al*., 2003). The quartz equivalent doses

(D_e) were measured using a UV filter (U-340) with a SAR protocol (Murray and Wintle, 2000) appropriate for young samples (preheat 180°C for 10 s; cut-heat of 160°C, 40 s stimulation at 280°C after every SAR cycle). The first 0.8 s of the signal less a background derived from the last 4 s was used for calculations. The IRSL signals from polymineral fine-grains, both at 50°C and elevated temperature, were detected through a blue filter combination (Schott BG39 + Corning 7-59, transmission 320-460 nm). In the SAR protocols used for IRSL measurements the thermal treatment (preheat) of the test dose was kept the same as for the natural and regenerative doses (Blair *et al*., 2005). Several preheating (and storage at room temperature) regimes for low (room temperature to ~60°C) temperature IRSL measurements on polymineral fine-grains exist (e.g. 1 month storage at room temperature + 5 min at 220°C, Lang *et al*., 2003; 160°C for 4 h, Lian and Huntley, 1999; 16 h at 140°C, Li and Wintle, 1992; one month room temperature storage +16 h at 150°C, Frechen and Dodonov, 1999; 250°C for 60s, Auclair *et al*., 2007). Because we measure the IR_{50} signal with a SAR protocol we have chosen practical short preheating times of 60 s. We measured IR_{50} both with a stringent 250°C preheat (Buylaert *et al*., 2007) and also with a low temperature preheat of 150°C. Further details of the thermal treatments and stimulation conditions for the individual SAR protocols are given in **Table 2**. The net IRSL signals were derived from the initial 0.8 s of the decay curve minus a background calculated using the last 9.6 s.

3. WHY USE POST-IR IRSL SIGNALS?

Laboratory measurements have indicated that doses measured using post-IR IRSL are less affected by anomalous fading than those measured using IRSL at 50°C

Table 1. *Sample details and equivalent doses for the modern/young samples. (1) D_e was measured using a double SAR procedure (Banerjee et al., 2001). The required length of IR stimulation time in the double SAR procedure was determined using a plateau test (Wang et al., 2006); an IR bleach of 100 s at 60°C was found to be sufficient. (2) The D_e values of the 4-11 μm fractions are not included in the average.*

Code	Location	Sample type	Quartz		Polymineral fine-grains (4-11 μm), D_e (Gy)			
			OSL, D_e (Gy)	grain size (μm)	IR_{50} (Ph:150°C/60s)	IR_{50} (Ph:250°C/60s)	$pIRIR_{225}$ (Ph:250°C/60s)	$pIRIR_{290}$ (Ph:320°C/60s)
D6	Zhou he zhen bei (37°07.910'N, 108°40.887' E)	top few cm of loess section	0.65±0.10 (n=9)	63-90	1.01±0.03 (n=5)	2.79±0.09 (n=6)	6.9±0.5 (n=6)	19±5 (n=6)
D9	Shi mao (37°56.183'N, 109°58.930' E)	top few cm of loess section	0.13±0.02 (n=17)	63-90	0.41±0.07 (n=3)	2.07±0.11 (n=7)	3.8±0.2 (n=6)	9.1±0.6 (n=6)
D10	Yu lin cheng bei (38°21.610'N, 109°41.978' E)	top few cm of loess section	0.02±0.04 (n=16)	63-90	0.37±0.12 (n=4)	1.65±0.12 (n=4)	2.0±0.2 (n=4)	7.3±0.6 (n=4)
D13	Xi'an	superficial dust from top of building	0.17±0.07 (n=18)	63-90	0.19±0.02 (n=3)	1.88±0.06 (n=7)	1.6±0.4 (n=6)	6.5±1.3 (n=6)
CB	Collected during ~200 km transect from Xi'an to Luochuan	superficial dust from car bumper	0.36±0.04 (1) (n=11)	4-11	0.40±0.02 (n=4)	1.91±0.09 (n=4)	7.5±0.5 (n=6)	15.6±0.5 (n=6)
			0.109±0.004 (n=14)	36-50				
TPU	Beijing (building Peking University)	superficial dust from top of building	0.24±0.03 (n=15)	4-11	0.15±0.02 (n=3)	1.49±0.02 (n=3)	1.97±0.07 (n=6)	5.3±0.2 (n=3)
		Average ± s.e.	0.22±0.11 (2)		0.42±0.13	2.0±0.2	4.1±1.1	10.5±2.3

Table 2. *The different SAR-IRSL measurement protocols used in this study.*

Step	IR_{50}	$pIRIR_{225}$	$pIRIR_{290}$
1	Dose	Dose	Dose
2	Preheat (125°C to 325°C for 60s)	Preheat (250°C for 60s)	Preheat (320°C for 60s)
3	-	IRSL, 100s at 50°C	IRSL, 200s at 50°C
4	IRSL, 100 or 300s at 50°C → L_x	IRSL, 100s at 225°C → L_x	IRSL, 200s at 290°C → L_x
5	Test dose	Test dose	Test dose
6	Preheat (125°C to 325°C for 60s)	Preheat (250°C for 60s)	Preheat (320°C for 60s)
7	-	IRSL, 100s at 50°C	IRSL, 200s at 50°C
8	IRSL, 100 or 300s at 50°C → T_x	IRSL, 100s at 225°C → T_x	IRSL, 200s at 290°C → T_x
9	IRSL, 40s at 290°C	IRSL, 40s at 290°C	IRSL, 100s at 325°C
10	Return to step 1	Return to step 1	Return to step 1

(Thomsen *et al*., 2008). Consequently, the ages are less dependent on the assumptions included in the fading correction models. This has been confirmed by Buylaert *et al*. (2009) for sand-sized K-feldspar grains and for polymineral fine-grains extracted from European loess by Thiel *et al*. (2011a). Here we test this conclusion for the first time for polymineral fine-grains extracted from Chinese loess by comparing the observed natural IRSL signal with the dose response curve for the three signals discussed in this paper, IRSL at 50°C (IR_{50}), IRSL at 225°C following IR_{50} ($pIRIR_{225}$) and IRSL at 290°C following IR_{50} ($pIRIR_{290}$) using the protocols given in **Table 2** and sample A42 (~900 ka). This sample has a predicted burial dose for polymineral fine-grains (feldspar) of ~3 kGy taking into account the dose rate to polymineral fine-grains for this sample of 3.62±0.16 Gy/ka (Buylaert *et al*., 2007). This dose is well beyond the expected saturation dose for this material.

Fig. 1 shows the three natural signals interpolated onto their respective dose response curves. As expected the IR_{50} signal is only 56% of the laboratory saturation IRSL intensity and predicts a finite dose of ~400 Gy. The $pIRIR_{225}$ is 73% of the saturation value and the $pIRIR_{290}$ is indistinguishable from saturation with an intensity of 99%. These data clearly support the suggestion that pIRIR signals are less prone to fading than the IR_{50} and indeed suggest that the $pIRIR_{290}$ may not fade significantly. A similar observation has been reported by Thiel *et al*. (2011a) for IR_{50} and $pIRIR_{290}$ for European loess.

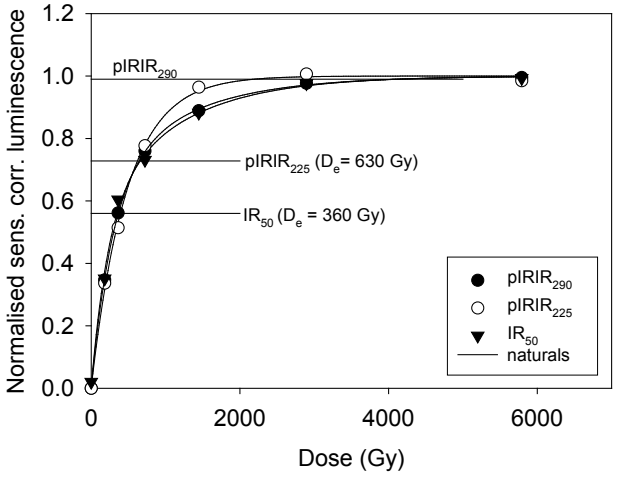

Fig. 1. *IR_{50}, $pIRIR_{225}$ and $pIRIR_{290}$ SAR dose response curves for polymineral fine-grains extracted from sample A42 collected in the L9 loess layer at the Luochuan section (well below the Brunhes/Matuyama boundary) (Buylaert* et al.*, 2007). This sample has an expected age of ~900 ka and is confidently expected to be in saturation. The $pIRIR_{290}$ and IR_{50} data were fitted with a double saturating exponential function of the form $y=a(1-exp(-bx))+c(1-exp(-dx))$ and the $pIRIR_{225}$ data with a single saturating exponential function $y=a(1-exp(-bx))$. The L_x/T_x values were normalised to the fitted saturation values ('a+c' for the double saturating exponentials and 'a' for the single exponential function). Taking a light level of 86% of saturation as the practical upper limit to give a resolvable dose (Wintle and Murray, 2006) we derive upper dose limits of 1300 Gy (IR_{50}), 950 Gy ($pIRIR_{225}$) and 1170 Gy ($pIRIR_{290}$). The sensitivity corrected natural luminescence levels are shown as horizontal lines and the corresponding D_e values are given between brackets. One aliquot was measured per protocol.*

Previous work has shown that the $pIRIR_{225}$ and $pIRIR_{290}$ residual doses (using modern coarse grained K-feldspars and laboratory bleached fine-grains respectively) tend to be greater than those observed using the IR_{50} signal (Thomsen *et al*., 2008; Buylaert *et al*., 2009; Thiel *et al*., 2011a). The next section examines whether these preliminary observations apply to polymineral fine-grains extracted from Chinese loess.

4. LUMINESCENCE CHARACTERISTICS AND D_e ESTIMATES

Fig. 2 illustrates some of the luminescence characteristics of Chinese loess using sample D9. As is to be expected for such a young sample, both the corrected natural OSL and IRSL signals lie on the linear part of the dose response curve. The natural signal from quartz is clearly much smaller than the regenerated signal compared to any of the three feldspar signals (i.e. IR_{50}, $pIRIR_{225}$ and $pIRIR_{290}$; see top left insets **Fig. 2**). The D_e's derived from the different signals are summarised for all the samples in **Table 1**.

To demonstrate that the SAR protocols used here (outlined in **Table 2**) are applicable to these samples several checks can be made. As is common for feldspar measurements, recycling ratios are very close to unity for all samples (0.988±0.009, n=9, $IR_{50,ph=150°C}$; 0.995±0.010, n=27, $IR_{50,ph=250°C}$; 1.00±0.01, n=35, $pIRIR_{225}$; 0.99±0.02, n=32, $pIRIR_{290}$; n denotes the number of aliquots that contribute to the quoted averages from different samples); recuperation is also acceptable (<7% for $IR_{50,ph=150°C}$, <5% for $IR_{50,ph=250°C}$, <7% for $pIRIR_{225}$ and <4% for $pIRIR_{290}$). The most complete laboratory test for any SAR protocol is to carry out a dose recovery test (Wallinga *et al*., 2000) to check whether a laboratory dose given before any heat treatment can be accurately measured. The advantage of working with modern dust samples is that we can add a large laboratory dose on top of the relatively small natural dose which allows us to avoid the bleaching step that would be necessary to remove the natural luminescence signal. In this experiment we have added a laboratory dose of 45 Gy to the natural samples and measured the dose with the SAR protocols outlined in **Table 2**. Between 2 and 8 aliquots were measured per measurement protocol for each of the 5 samples used. After subtraction of the natural D_e (**Table 1**) from the measured dose, dose recovery ratios are consistent with unity for all protocols used. The mean values are 1.020±0.003 (n=6, $IR_{50,\ ph=150°C}$), 0.999±0.008 (n=6, $IR_{50,\ ph=250°C}$), 1.01±0.03 (n=28, $pIRIR_{225}$) and 1.01±0.05 (n=23, $pIRIR_{290}$). It seems that all the SAR protocols

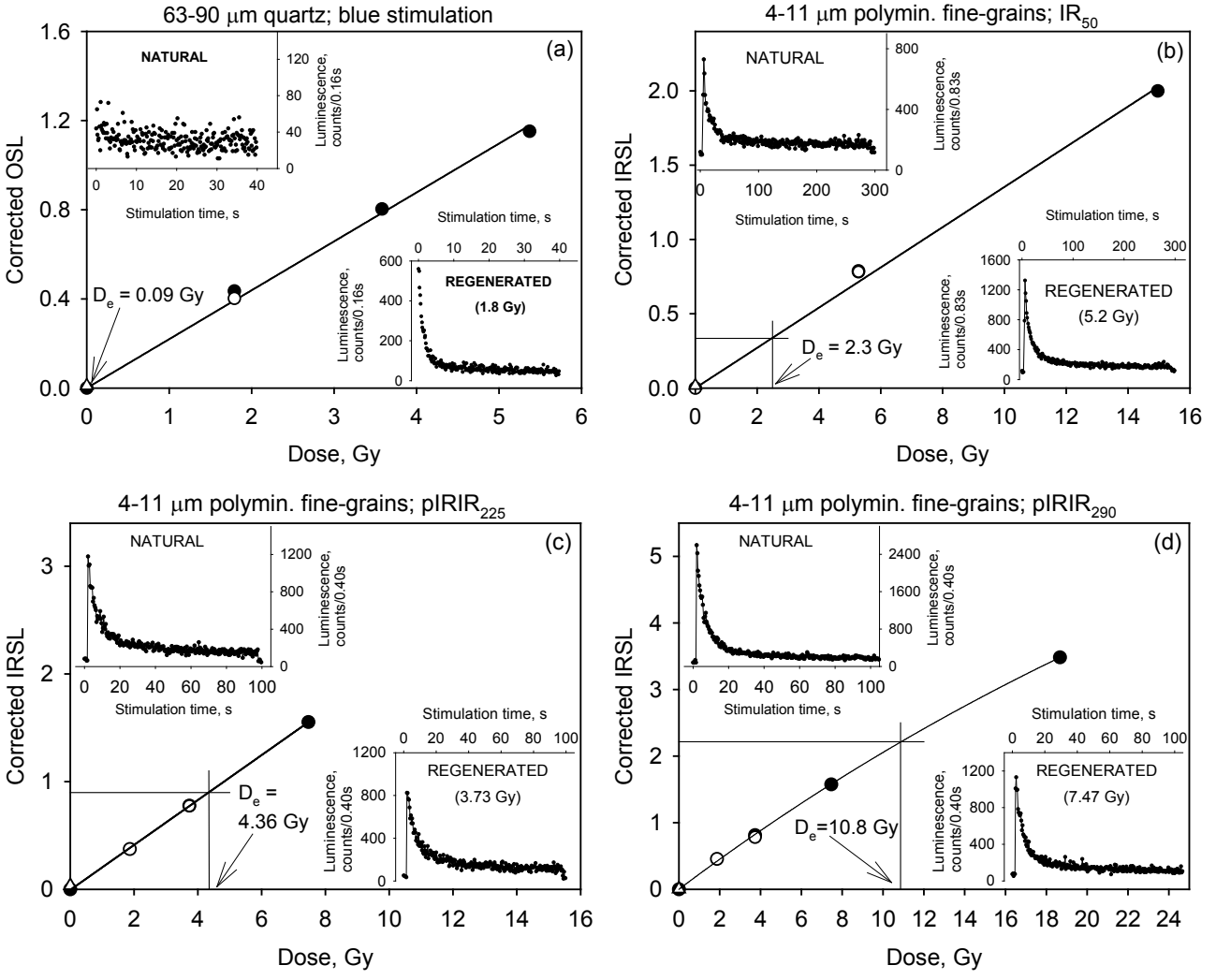

Fig. 2. *Representative dose response curves of sample D9 for the different signals under investigation. (a) quartz OSL, (b) IR_{50} (preheat=250°C/60s), (c) $pIRIR_{225}$, (d) $pIRIR_{290}$. Recycled points and zero dose (recuperation) points are shown as open circles and triangles respectively. The insets show natural (top left) and regenerated (bottom right) decay curves.*

outlined in **Table 2** are able to accurately measure a laboratory dose prior to any heat treatment. For completeness, the quartz OSL dose recovery ratio for sample D9 (dose of 34 Gy on top of natural aliquots) is 0.96±0.02 (n=3).

It is interesting to note that for the quartz OSL measurements only one sample (D10) yields a D_e indistinguishable from zero. This is surprising given the aeolian nature of deposition of these samples. The coarser grain fractions also seem to be better bleached than the 4-11 μm fractions. This is consistent with what has been reported for fluvial sediments (Olley *et al.*, 1998; Wallinga, 2002; Vandenberghe *et al.*, 2007). Nevertheless, for the purpose of comparison with the polymineral fine-grain feldspar doses the quartz doses are small enough (of the order of a few tens to hundreds of mGy) to represent well-bleached sediment.

The doses recorded by IR_{50} (both ph=150°C and ph=250°C), $pIRIR_{225}$ and $pIRIR_{290}$ are plotted for all samples against their quartz OSL D_e in **Fig. 3**. On average the apparent dose follows the pattern: IR_{50} (ph=150°C) < IR_{50} (ph=250°C) < $pIRIR_{225}$ < $pIRIR_{290}$. Only two samples measured with a low 150°C preheat and with IR stimulation at 50°C are consistent with the slope predicted from the difference in dose rate between coarse quartz grains and polymineral fine-grains, the rest show apparent overestimation of feldspar doses (see caption to **Fig. 3**). In the next section we address the question whether this apparent overestimation arises because of incomplete bleaching or transfer of charge from optically-insensitive thermally-shallow traps to the optically-sensitive traps during preheating.

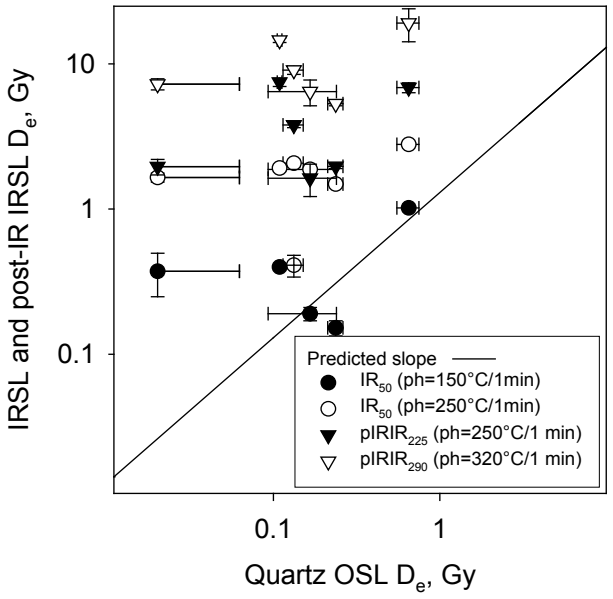

Fig. 3. *Equivalent dose estimates derived from the IR_{50} (both for preheats of 150°C and 250°C), $pIRIR_{225}$ and $pIRIR_{290}$ protocols plotted against those from quartz OSL for all modern samples used. A minimum of three aliquots was used to determine the average D_e values and error bars represent 1 standard error. The solid line represents the predicted slope of this relationship for well-bleached material; it is based on the difference in dose rate between coarse quartz grains and polymineral fine-grains. The average ratio of the polymineral fine-grain to coarse grained quartz dose rates for the loess samples in Buylaert* et al. *(2007; their Table S1) is 1.290±0.003 (n=9). This value was used to plot the predicted slope.*

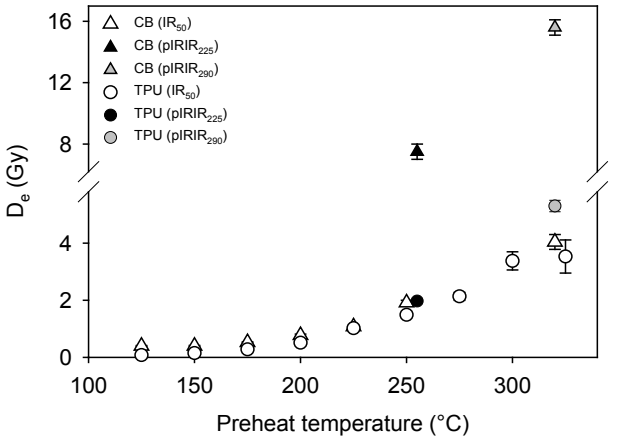

Fig. 4. *Apparent IR_{50}, $pIRIR_{225}$ and $pIRIR_{290}$ D_e values as a function of preheat temperature (storage 60 s) for two superficial Chinese dust samples. Three aliquots were measured per preheat temperature and error bars represent one standard error. Due to a lack of material the plot for the IR_{50} signal of sample CB ends at 250°C.*

5. INCOMPLETE BLEACHING OR THERMAL TRANSFER?

A contribution from charge thermally transferred to the IRSL trap(s) during preheating can cause an apparent dose to be measured in a sample expected to contain a "zero dose" or at least a very small dose (Aitken, 1998). The presence of thermal transfer in polymineral fine-grains has already been reported by Rees-Jones and Tite (1994). Whereas in principle it would be possible to keep the preheat low for the IR_{50} signal, a difficulty in post-IR IRSL dating is that it is necessary to use a preheat that is equal to or higher than the stimulation temperature (to avoid the influence of a thermoluminescence signal on the post-IR IRSL signal of interest). These preheats are typically 20-30°C higher than the post-IR IR stimulation temperature (**Table 2**).

Two superficial dust samples (TPU and CB) were used to check the contribution of thermal transfer in our data (**Fig. 3** and **Table 1**). For the IR_{50} signal the D_e values measured using polymineral fine-grains from these two samples are shown in **Fig. 4** as a function of storage for 60 s at the given preheat temperatures. At low temperatures the D_e is very small (although still finite), but it increases to ≥ 1.5 Gy for a preheat temperature of 250°C. This smooth increase with temperature suggests a contribution from transfer from thermally-shallow light-insensitive traps to the IR-sensitive dosimetry trap(s) for preheats ≥ 150°C. From the difference in dose estimates at low temperatures, it also appears that the Beijing dust sample (TPU) is better bleached than the car bumper (CB) sample. For the TPU sample the $pIRIR_{225}$ (preheat of 250°C) and $pIRIR_{290}$ (preheat of 320°C) signal seem to closely follow the trend of the IR_{50} thermal transfer curve. Both signals also appear to be nearly as well-bleached as the IR_{50} signal. For the CB sample the $pIRIR_{225}$ and $pIRIR_{290}$ signals seem to be less well-bleached than the IR_{50} signal; the dose difference for $pIRIR_{225}$ is ~6 Gy and for $pIRIR_{290}$ of ~10 Gy respectively.

The question now arises whether one can, in order to avoid thermal transfer, use a low temperature (<200°C) preheat when dating loess with IRSL measured at 50°C.

6. CAN WE USE A LOW TEMPERATURE PREHEAT TO DATE LOESS USING IRSL AT 50°C?

A Chinese loess sample (A7) with a quartz OSL age of 29.0±1.0 ka (Buylaert *et al.*, 2008) is used to check whether low temperature preheats can be used for dating with the IRSL measured at 50°C. In this age range quartz OSL dating provides suitable age control (the quartz D_e is 92±2 Gy). This sample was also dated using $pIRIR_{225}$ and $pIRIR_{290}$. Anomalous fading rates (*g*-values; Aitken, 1985) were measured using SAR on the same aliquots that were used for D_e determinations (Auclair *et al.*, 2003). The results are shown in **Fig. 5** and **Table 3** gives the individual D_e values, fading rates and ages for preheat temperatures of 150°C, 250°C and 320°C.

The fading corrected IR_{50} age plateau, measured between preheats of 125°C and 275°C, shows a weak but

clear overall rising trend. The low IR_{50} ages at lower (<200°C) preheat temperatures are almost certainly caused by a contribution from a thermally unstable signal induced during laboratory dosing that is not removed during lower temperature preheating; the increase in D_e with increasing temperature is ~25 Gy (~22% of the average high temperature dose), much larger than the thermally transferred dose for IR_{50} in **Fig. 4** (which only reaches ~2 Gy at 275°C). For preheat temperatures >200°C the fading corrected IR_{50} results are in agreement with the quartz age. It is worth noting that the rising trend in the fading corrected IR_{50} age with temperature is somewhat less pronounced than the trend in dose because of a lower fading rate at higher temperatures; the difference between fading corrected IR_{50} ages at 150°C and at 250°C is ~15% (still too large to be explained entirely by thermal transfer).

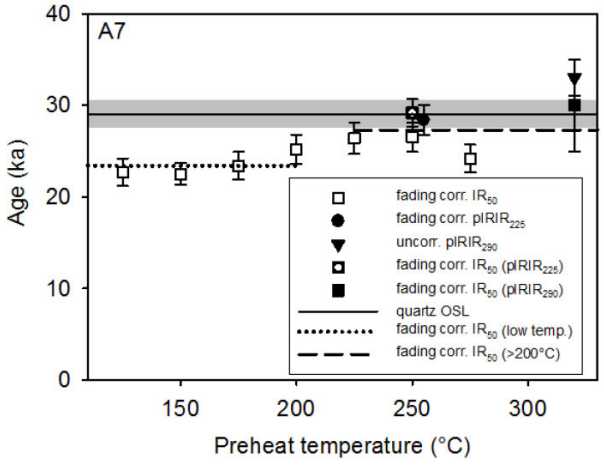

Fig. 5. *Age-preheat plot of the IRSL and post-IR IRSL measurements for an older Chinese loess sample (A7) previously dated using quartz OSL by Buylaert* et al. *(2008). The solid line represent the quartz OSL age (± 1 s.e. shown as a grey band). The residual doses of the CB sample were subtracted from the D_e values (**Table 1**). From the IR_{50} D_e values the residual doses were subtracted based on the thermal transfer curve of the CB sample in **Fig. 4**. The IR_{50} and $pIRIR_{225}$ ages were corrected using the model of Huntley and Lamothe (2001). As fading rates (g_{2days}-values) had only been measured using preheats of 150°C and 250°C for the IR_{50} signal, the fading rates for intermediate temperatures of the IR_{50} measurements were interpolated (175°C, 200°C and 225°C) or extrapolated (125°C and 275°C). The average fading corrected IR_{50} ages for low (125-200°C) and high temperatures (225-320°C) are shown as dotted and dashed lines respectively. Three aliquots were measured at each temperature and error bars represent one standard error.*

The fading rates of both pIRIR signals are lower than the fading rates measured at 50°C; this is consistent with previous observations (e.g. Buylaert *et al.*, 2009). It is interesting to note that the $pIRIR_{225}$ and $pIRIR_{290}$ fading rates are not significantly different from each other. There is some published evidence that $pIRIR_{225}$ ages do indeed need fading correction (Buylaert *et al.*, 2009, Thiel *et al.*, 2011b) and that $pIRIR_{290}$ ages do not need fading correction (Thiel *et al.*, 2011a; Thiel *et al.*, 2011c; Thomsen *et al.*, 2011); the latter observation is further supported by the data of **Fig. 1**, in which the natural IR signal from a $pIRIR_{225}$ protocol is only ~73% of the laboratory saturation value, whereas the $pIRIR_{290}$ signal is 99% of laboratory saturation. Both corrected $pIRIR_{225}$ and uncorrected $pIRIR_{290}$ (within 2 sigma) ages are in agreement with the quartz age and with the fading corrected IR_{50} ages measured as part of the post-IR IRSL measurement protocols (i.e. with 250°C and 320°C preheats) . For completeness we have also fading corrected the $pIRIR_{290}$ age; the resulting 36±3 ka seems to overestimate the expected quartz age (**Table 3**). This further supports the suggestion above that the $pIRIR_{290}$ signal should not be corrected for any apparent laboratory fading, and indicates that the measured (very low) fading rate for $pIRIR_{290}$ signal may be a laboratory artefact.

The observation that the pIRIR ages for this sample are in agreement with the quartz age is encouraging. However, more samples, with independent age control are needed before we can confidently state that this dating

Table 3. *Summary of IRSL and post-IR IRSL measurements for sample A7 of the Zhongjiacai site previously dated using quartz OSL by Buylaert* et al. *(2008). The g-values were normalised to a measurement delay time of 2 days after irradiation (i.e. g_{2days}, Huntley and Lamothe, 2001). Three aliquots were measured per treatment.*

Code		Dose rate (2) (Gy/ka)	D_e (3) (Gy)	g_{2days} (%/decade)	Uncorrected age (ka)	Corrected age (4) (ka)	Quartz age (1) (ka)
A7 (1)	IR_{50} (Ph:150°C/60s)	4.08±0.21	61.9±0.1	4.13±0.10	15.2±0.8	22.5±1.2	29.0±1.4
	IR_{50} (Ph:250°C/60s)	"	87±2	2.4±0.4	21.3±1.2	27±2	-
	$pIRIR_{225}$ (Ph:250°C/60s)	"	103±2	1.33±0.14	25.2±1.4	28±2	-
	$pIRIR_{290}$ (Ph:320°C/60s)	"	135±3	1.0±0.8	33±2	36±3	-
	IR_{50} from $pIRIR_{225}$	"	86.1±0.4	3.44±0.03	21.1±1.1	29±2	-
	IR_{50} from $pIRIR_{290}$	"	87±5	3.9±1.1	21±2	30±5	-

(1) See Buylaert et al. *(2008)*

(2) Dose rate to polymineral fine-grains was calculated using the radionuclide concentrations and water content given in Buylaert et al. *(2008). An a-value of 0.08 to allow for the lower luminescence efficiency of alpha compared to beta and gamma radiation was used in the calculation (Rees-Jones, 1995).*

(3) Residual doses equal to those measured for the CB sample at the relevant preheat temperatures (Fig. 4) were subtracted from the D_e

(4) The fading correction was carried out following the model of Huntley and Lamothe (2001)

approach always yields accurate results in Chinese loess. Nevertheless, we can conclude that although low preheat temperatures may avoid or minimise thermal transfer, this is probably at the expense of an increasingly significant contribution from a thermally-shallow unstable signal which leads to a significant underestimate of dose. Similar conclusions were reported using a different (multiple aliquot) protocol by Li and Wintle (1992).

7. CONCLUSIONS

Using a set of modern/young dust samples from China the residual doses measured with IR_{50} and two post-IR IRSL protocols ($pIRIR_{225}$ and $pIRIR_{290}$) have been measured; these give doses in the range up to ~20 Gy. Quartz OSL doses (all <650 mGy) are used to confirm that our samples are indeed modern/very young. As has been shown by others, there is clear evidence for thermal transfer in IRSL signals from polymineral fine-grains; this results from the preheating of the sample prior to IRSL measurement. From our data it seems that thermal transfer is present in IR_{50} signals but also, and to a larger degree, in the post-IR IRSL signals. On average the residual doses recorded in modern/very young samples, presumably mainly arising from thermal transfer effects, amount to 2.0±0.2 Gy for IR_{50}, 4.1±1.1 Gy for $pIRIR_{225}$ and 10±2 Gy for $pIRIR_{290}$. Whereas in principle a low temperature preheat could be used to avoid thermal transfer during IR_{50} measurements this seems unadvisable for the post-IR IRSL measurements because of the then unavoidable TL contribution arising from the high temperature second IR stimulation. Based on a measurement of the dependence of age on preheat for a relatively old (~30 ka) loess sample, we show that it is not actually advisable to use a low preheat temperature for the IR_{50} signal either; for preheat temperatures <200°C at least part of the IR_{50} signal appears to be made up of a thermally unstable component. The apparent residual doses measured on modern material using post-IR IRSL suggests that it seems prudent not to apply post-IR IRSL dating to Chinese loess samples for which residual doses up to ~20 Gy are likely to be significant.

ACKNOWLEDGEMENTS

Financial support from the Nordic Centre of Excellence programme of the Joint Committee of Nordic Natural Science Research Councils is gratefully acknowledged. DV is a postdoctoral fellow of the Research Foundation – Flanders (FWO – Vlaanderen). Liping Zhou, Jintang Qin, and Yali Zhou are thanked for help during sample collection. The technical assistance of Jochem Temmerman, Nicole Selen and Gilles Velghe (Ghent University) is very much appreciated. Saiko Sugisaki is thanked for help with the luminescence measurements, and Ann Wintle for comments on an earlier draft of this paper.

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