



EXAMINING QUARTZ OSL AGE UNDERESTIMATION FOR LOESS SAMPLES FROM LUOCHUAN IN THE CHINESE LOESS PLATEAU

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Received 29 January 2013

Accepted 22 August 2013

Abstract: When using quartz OSL to date loess samples from the Chinese Loess Plateau, it has been reported that the agreement between OSL ages and the independent ages is limited to the samples younger than ~70 ka with a corresponding D_e of ~230 Gy, and a sample with an expected age of 780 ka was dated to 107 ka, corresponding to 403 Gy. The growth curves of these samples do not saturate at doses of 700 Gy, and a linear growth part was observed for doses higher than 200 Gy. However, the maximum measured age of ~100 ka imply that the D_e determined using this linear part of a growth curve could be problematic, or that the quartz OSL signal is not as stable as previously thought and has a barrier age of ~100 ka. In the current study, we examine the reasons for the age underestimation. We examined the shape of growth curves, anomalous fading, thermal stability, etc. The results show that, for the loess samples examined, quartz OSL does not fade anomalously, and the barrier age of ~100 ka is due to the fact that the OSL signals are less thermally stable, the lifetime of 0.311 Ma at 20°C obtained is much smaller than those for quartz samples from other regions such as Australia (~100 Ma).

Keywords: quartz OSL, age underestimation, Chinese loess, signal stability.

1. INTRODUCTION

As a dosimeter in luminescence dating, quartz is much preferred over feldspars due to its non-anomalous-fading signal and the much simpler trap or recombination center mechanism. However, the quartz signal saturates at a lower dose (see review by Wintle and Murray, 2006, and references therein). Recently, a growing body of data shows that the quartz OSL SAR (Single Aliquot Regenerative dose protocol) growth curve has a linear growth part in the high dose range of *ca.* 200–1000 Gy, which

could allow the D_e determination up to >400 Gy (Watanuki *et al.*, 2003, 2005; Buylaert *et al.*, 2008; Lai, 2010; Lowick *et al.*, 2010; Lowick and Preusser, 2011). When using quartz OSL SAR protocol to date loess samples from Luochuan section, a standard loess section in the Chinese Loess Plateau (CLP) with a well-defined independent chronology based on astronomical tuning (Lu *et al.*, 1999), it has been reported that the agreement between OSL ages and the independent ages is limited to 70 ka with a corresponding D_e of *ca.* 230 Gy, and that the measured age was only 107 ka (with a corresponding age of 403 Gy) for a sample collected just below the B/M boundary with an expected age of 780 ka (Lai, 2010).

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Underestimation observations have also been made by Buylaert *et al.* (2008), Qin and Zhou (2009), and Chapot *et al.* (2012) for Chinese loess. These seem to imply that the D_e determined using this linear part of a growth curve could be problematic, or that the quartz OSL signal is not as stable as previously thought. Lu *et al.* (2007), however, reported the OSL dating of the loess samples <130 ka from the Luochuan section using a quartz OSL-MAR method. Their OSL ages obtained are broadly consistent with the expected ages, and the age underestimation is not apparent for samples from the L1 unit.

For the linear part of the growth curve at high doses, it is possible that the trap/luminescence recombination center, which is assumed to be responsible for the linear growth part, does not exist in nature (Lai, 2010), and is created by laboratory irradiation only, which cannot be removed by the preheat (Lai, 2010). When investigating quartz OSL dose-response curves at high doses, Lowick *et al.* (2010) proposed that a likely explanation is that for the change in OSL response at high doses is a change in competition for electrons between the UV recombination centers whose emission is seen through the detection windows and recombination centers that do not emit in this spectral region (or are non-radiative).

The lower thermal stability of luminescence signals could lead to age underestimation, which has been shown for feldspars (Wintle, 1973; Debenham, 1985; Lamothe and Auclair, 1999). Previous studies showed that the quartz OSL signal is dominated by a stable signal with a lifetime of 850 Ma at 20°C, confirming its suitability for dating (Murray and Wintle, 1999). However, it has been

reported that the OSL of some quartz showed much lower stability which could lead to age underestimation by more than a factor of 10 (Bonde *et al.*, 2001; Choi *et al.*, 2003; Li and Li, 2006; Tsukamoto *et al.*, 2007; Fan *et al.*, 2011). In particular, Fan *et al.* (2011) dated samples from Salawusu in the Mu Us desert in northern China, which was situated to the north of the CLP and regarded as one of the dust sources for the loess deposits in the CLP. Based on the single-grain pulse annealing results they demonstrated that, for some of the quartz grains, the fast component is thermally unstable. They tried to isolate those grains with stable OSL for age calculation and the resultant ages are consistent with independent control.

In this study, we investigated the luminescence properties of quartz grains from loess samples from the Luochuan section, and try to explain the quartz OSL age underestimation of loess samples.

2. SAMPLES USED

Four samples (LC12, LC22, LC30, and LM/9) were selected to represent the loess samples. The samples used are 45-63 μm fractions of quartz grains from Luochuan section in the CLP in northern China, and have been OSL dated by Lai (2010). Medium aliquots (~3 mm in diameter) were used in all measurements. The relevant sample information is summarized in Table 1, including the dose rate and expected age data. The section is a classical standard section due to its clear loess-palaeosol alternations reflecting the glacial-interglacial cycles (Kukla and An, 1989). A well-defined chronology based on astro-

Table 1. Sample information for Luochuan loess (data from Lai, 2010). A well-defined chronology based on astronomic tuning has been established for this section (Lu *et al.*, 1999), which is taken as an independent age control, and the expected ages for samples between loess/soil boundaries have been obtained using interpolation with an error of 10%.

Sample ID	Depth (m)	Dose Rate (Gy/ka)	OSL D_e (Gy)	OSL Age (ka)	Expected Age (ka)	Position in section
LC4	2.5	3.14 \pm 0.21	75 \pm 2	24.0 \pm 1.7	25.0	L1
LC5	2.8	3.13 \pm 0.21	83 \pm 4	26.5 \pm 2.2	27.0	L1
LC7	3.4	3.45 \pm 0.23	111 \pm 5	32.2 \pm 2.6	31.1	L1
LC9	4	3.20 \pm 0.22	121 \pm 3	37.8 \pm 2.8	35.2	L1
LC12	4.8	3.24 \pm 0.22	135 \pm 5	41.7 \pm 3.1	40.7	L1
LC14	5.4	3.35 \pm 0.23	138 \pm 5	41.2 \pm 3.2	44.7	L1
LC16	6	3.36 \pm 0.23	174 \pm 4	51.9 \pm 3.7	48.8	L1
LC35	6.9	3.24 \pm 0.22	182 \pm 5	56.2 \pm 4.2	54.9	L1
LC33	7.6	3.15 \pm 0.22	200 \pm 10	63.5 \pm 5.3	59.7	L1
LC30	8.5	3.17 \pm 0.25	195 \pm 6	61.5 \pm 5.2	65.8	L1
LC29	8.8	3.29 \pm 0.26	213 \pm 10	64.6 \pm 5.9	67.9	L1
LC28	9.1	3.27 \pm 0.26	216 \pm 13	66.0 \pm 6.6	73.0	L1
LC27	9.4	3.15 \pm 0.26	231 \pm 12	73.2 \pm 6.4	80.3	S1 (0.3 m below L1)
LC26	10	3.53 \pm 0.27	283 \pm 12	80.3 \pm 7.0	94.9	S1
LC25	10.3	3.49 \pm 0.27	289 \pm 10	82.7 \pm 7.0	102.2	S1
LC24	10.6	3.58 \pm 0.28	313 \pm 11	87.4 \pm 7.5	109.5	S1
LC23	10.9	3.45 \pm 0.27	308 \pm 13	89.5 \pm 8.0	116.8	S1
LC38	11.2	3.47 \pm 0.27	296 \pm 9	85.3 \pm 7.1	124.1	S1
LC22	11.5	3.32 \pm 0.27	315 \pm 11	94.9 \pm 8.3	131.4	S1 (0.3 m above L2)
L9/M	c. 60	3.77 \pm 0.28	403 \pm 25	107 \pm 10	780.0	L9 (just below B/M)

nomic tuning has been established for this section (Lu *et al.*, 1999), which is taken as independent age control, and the expected ages for samples between loess/soil boundaries have been obtained using interpolation with an error of 10%.

3. MEASUREMENT INSTRUMENTS

A Risø automated TL/OSL system was used for all measurements. The OSL signals were measured by a photomultiplier with U-340 filters under the stimulation of blue light-emitting diodes (470 ± 30 nm). Beta irradiation was performed using the $^{90}\text{Sr}/^{90}\text{Y}$ beta source within the Risø system.

4. EXPERIMENT RESULTS AND DISCUSSIONS

Growth curves

The quartz OSL growth curve of the Luochuan samples up to a dose of 700 Gy has been constructed by Lai (2010, in his Fig. 5) using a SAR protocol, with a preheat at 260°C for 10 s and with the test dose receiving a cut-heat of 220°C for 10 s, and OSL of the first 0.8 s stimulation (background of the last 5 s was subtracted) was integrated for growth curve construction. The growth curve was constructed using four aliquots of sample L9/Ma and could be well-fitted using a single exponential plus linear function:

$$Y = 8.55 \cdot (1 - \exp(-X/128.3)) + 0.0062X \quad (4.1)$$

where D_0 is 128.3 (Gy) (Lai, 2010). Assuming a maximum D_e value can be determined up to $2D_0$ (Wintle and Murray, 2006), D_e values up to 256 Gy should be obtainable. Fig. 1 shows the comparison of the expected ages with the OSL ages for samples younger than 150 ka, not

including the sample of L9/M. Samples older than 70 ka are underestimated. L9/M, which has an expected age of 780 ka, gave an age of 107 ka.

It will be interesting to see the shape of the growth curve in nature. Chapot *et al.* (2012) have constructed a natural dose response curve for known-age samples from Luochuan section, but with only 5 samples within a dose range of 500 Gy. Here we use 19 samples within a dose range of 450 Gy. For each sample listed in Table 1, the sensitivity corrected natural signals (L_N/T_N) have been measured for four aliquots. In Fig. 2 the X-axis is the

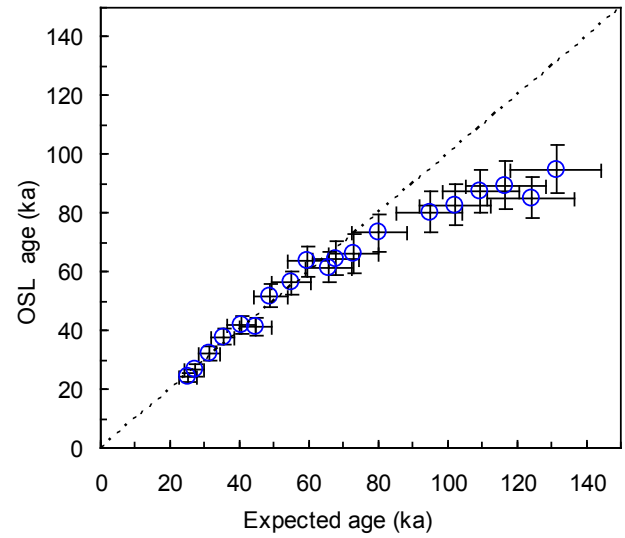


Fig. 1. Plot of OSL ages against expected ages for loess samples from the Chinese Loess Plateau. The OSL ages for the loess samples are from Lai (2010), and their expected ages are inferred from Lu *et al.* (1999).

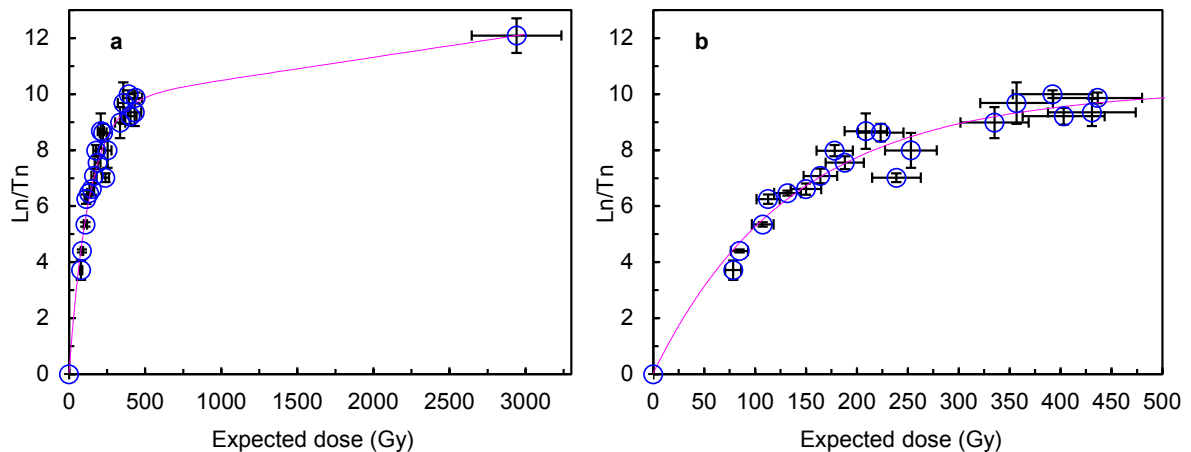


Fig. 2. A growth curve for the loess samples from Chinese Loess Plateau. The curve was constructed by plotting the sensitivity-corrected natural singles (L_N/T_N) of the loess samples from different depths of a section against their expected doses calculated from their expected ages and dose rates. (a) For all the samples being dated in Lai (2010), (b) for the samples with expected doses of <450 Gy.

expected dose (expected age multiplied by dose-rate, see **Table 1**) and the Y-axis the L_N/T_N (mean of four aliquots). The growth curve could be fitted using an exponential plus linear:

$$Y = 9.63 \cdot (1 - \exp(-X/131)) + 0.00082X \quad (4.2)$$

where D_0 is 131 Gy, so that the maximum value for dating should be 262 Gy ($2D_0$) Gy (corresponding to an OSL age of about 85 ka), similar to the artificial growth curve by Lai (2010) with a D_0 of 128.3 (Gy).

Anomalous fading test

To investigate whether anomalous fading is the cause of the age underestimation, three aliquots of each of the four samples (LC12, LC22, LC30, and LM/9) were measured using the procedure described by Auclair *et al.* (2003). The beta doses applied to those bleached aliquots were 100 and 25 Gy, for L_x and T_x , respectively. A pre-heat of 260°C for 10 s was performed immediately after each 100 Gy irradiation. Before measuring the signal corresponding to the test dose, a cut-heat to 220°C was used. The OSL measurements were repeated with five different time delays (0.1, 1, 10, 100, and 200 h) after irradiation. The normalized OSL signals were plotted versus the time delays (**Fig. 3**). The g-values are 0.001%, 0.004%, -0.004%, and -0.001% per decade for samples LC12, LC22, LC30, and LM/9, respectively. The results show that there is no detectable fading for the OSL signals of the four loess samples.

The OSL signals measured in the anomalous fading test were also analyzed using curve-fitting. The bulk OSL signal is dominated by the fast component (>90%). The fast component shows no evidence of anomalous fading. The results demonstrate that the OSL age underestimation (Lai, 2010) should not be caused by the anomalous fading of the OSL signals.

Thermal stability of OSL signal: kinetic parameters by pulse annealing

Pulse annealing experiments were employed to investigate the thermal stability of luminescence signals from quartz (Li and Chen, 2001). A previously bleached aliquot was irradiated with 100 Gy, and was heated to a certain temperature. Then the remaining OSL signals (L_x) were measured with 40 s blue stimulation. OSL response at 125°C to a test dose of 25 Gy after L_x measurement was used to monitor the sensitivity change. At the end of each run, the aliquot was heated to 500°C to clean all signals. This run was repeated several times, in which the temperature was increased from 200 to 360°C, with an increment of 20°C. The heating rates were 5°C/s for all cycles. At least six aliquots were measured for each of the four samples (LC12, LC22, LC30, and LM/9).

The remnant luminescence after being heated to a certain temperature was displayed as the ratio of the initial values in **Fig. 4**. The results show that: (1) The normalized OSL signals of sample L9/M showed an early decrease at a lower temperature of 240°C, comparing with

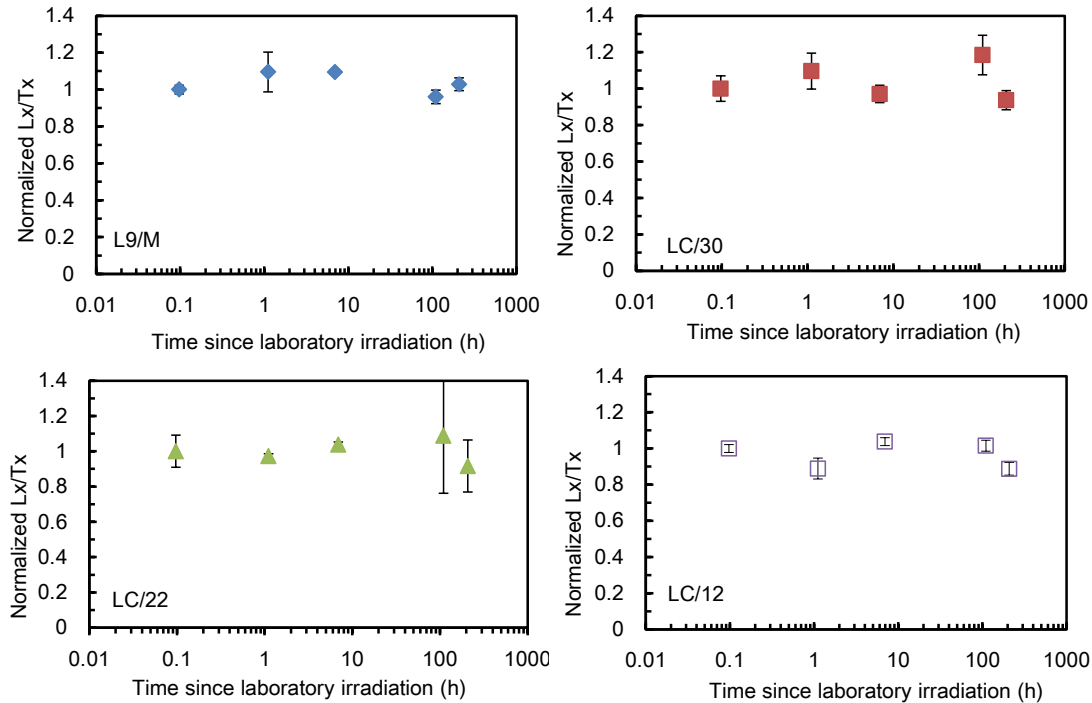


Fig. 3. Dependency of luminescence signals on delay times after irradiation. No anomalous fading was observed.

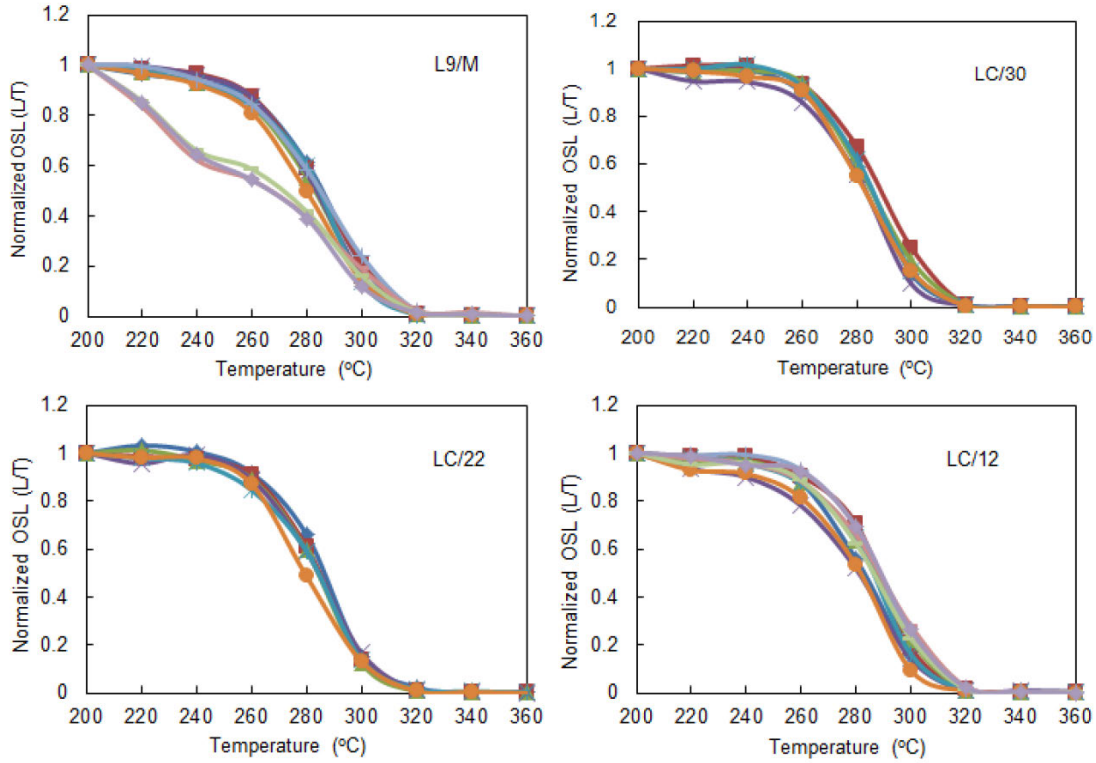


Fig. 4. Pulse annealing curves of the four loess samples (LC12, LC22, LC30, and LM/9).

the other three samples (LC/12, LC/22, and LC/30). (2) The most thermally-unstable signal is observed in only three aliquots of sample L9/M. The curve fitting analysis (as described in Section 4.4) shows that this unstable signal may be related to the ultra-fast component, which could be removed by a preheat at 260°C (Jain *et al.*, 2003). (3) Pulse annealing curves of all four samples showed aliquot-to-aliquot variations between temperatures ranging from 200°C to 300°C. (4) For all four samples, OSL signals decreased to less than 3% after heating to 320°C, suggesting that the OSL signals are mainly from the TL peak of 325°C (Wintle and Murray, 1998).

For dating purposes, the lifetime of OSL signal should be at least 5-10 times the age of the sample (Aitken, 1985). Previous studies showed that the quartz OSL signal of sample WIDG8 from Australia is dominated by a stable signal with a lifetime of 850 Ma at 20°C (Wintle and Murray, 1998), which is stable enough for dating samples as old as 1 Ma. The comparison of the pulse annealing curves for our loess samples and sample WIDG8 shows that the OSL signals from the former is less stable (Fig. 5). The beginning and ending of the OSL signals of the loess samples are 10-20°C earlier than those of the OSL signal measured from sample WIDG8. The TL peak corresponding to such signal is thus thermally less stable by 10-20°C.

To evaluate the effects of the unstable signal on the underestimation of quartz OSL ages, trap parameters,

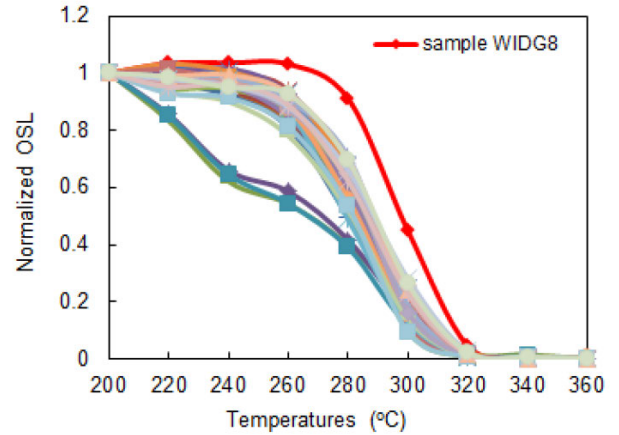


Fig. 5. Comparison of thermal stability between loess samples and a typical quartz sample WIDG8 from Australia used by Murray and Wintle (1999).

including trap depth (E), frequency factor (s) are obtained based on the fitting of the pulse annealing curves (Singarayer and Bailey, 2003). Assuming first-order kinetics during thermal erosion in the pulse annealing experiments, trap lifetime is derived by equation:

$$\tau = s^{-1} \cdot \exp(E/k_B T) \quad (4.3)$$

where T is temperature given in Kelvin and k_B is Boltzmann's constant. The remnant trapped charge n after annealing to temperature T , is

$$n = n_0 \exp \left[\left(\frac{-sk_B T^2}{BE} \exp \left(-\frac{E}{kT} \right) \right) + \left(\frac{-sk_B T_0^2}{BE} \exp \left(-\frac{E}{kT_0} \right) \right) \right] \quad (4.4)$$

where n_0 is the initial trapped charge concentration, T_0 is the ambient room temperature (~ 293 K) (Singarayer, 2002). The obtained trap depth (E), frequency factor (s) for the loess samples are listed in **Table 2**, and the results of the same parameters by Singarayer and Bailey (2003) and Wintle and Murray (1998) are also listed in **Table 2** for comparison. While the lifetime at 20°C are 850 Ma by Wintle and Murray (1998) and 310 Ma by Singarayer and Bailey (2003), the lifetime for the loess samples under the study is only 0.311 Ma. A calibration quartz sample, provided by Risoe, for calibrating the beta source was also measured and the lifetime is 393 Ma. The trap depth (E) of loess samples is also much lower than that of WIDG 8 (**Table 2**). The calculated value is consistent with the OSL ages measured for loess samples. Considering the fact that the measured OSL age is only 107 ka for sample L9/M with an expected age of ~ 800 ka (**Table 2**), the lifetime of the OSL signal from loess quartz should be shorter than 800 ka and probably is around 100 ka. The thermal instability might be the reason causing the age underestimation in those loess samples when the quartz OSL signal is used for dating.

Optical properties of the OSL signal

Curve fitting was then used to 1) determine whether the optical properties of the signals from the loess quartz grains are different from previously reported quartz samples, as well as the thermal stability; 2) to determine the relative contribution of components in the initial bulk OSL signal used for dating (Bailey *et al.*, 1997). At least two exponentially decaying components plus a constant background were needed to fit the CW-OSL curves, described by the equation

$$L = A \cdot \exp(-b_1 \cdot t) + B \cdot \exp(-b_2 \cdot t) + C \quad (4.5)$$

The average value of detrapping probability b_1 of the fastest component is $3.13 \pm 0.1 \text{ s}^{-1}$ for all OSL curves measured, including both natural signals and regenerative ones from four loess samples. There is no significant change in the component contribution between the natural and regenerative OSL signals. A photoionization cross-section α of $(2.93 \pm 0.10) \cdot 10^{-17} \text{ cm}^2$ was obtained. This is consistent with the value for the fast component in previous studies (Jain *et al.*, 2003; Singarayer and Bailey, 2003; Lowick and Preusser, 2011).

Based on the curve fitting, relative contribution of each component in the OSL signals are plotted in **Fig. 6**. The fast component contributes to over 90% of the OSL signals measured in the first 0.6 second from the aliquot. The OSL signal from the loess sample is fast-component dominated, confirming the results of Lai (2010).

For three aliquots of sample L9/M, an extra component is required (**Fig. 7**). The calculated photoionization cross-section α is $2.35 \cdot 10^{-16} \text{ cm}^2$, which is consistent with the value of $2.9 \cdot 10^{-16} \text{ cm}^2$ obtained for the ultra-fast component (Jain *et al.*, 2003). The ultra-fast component decreases to a negligible level ($<5\%$) after being heated to a temperature of 260°C . Thus, this component would not affect the SAR dating results by Lai (2010), in which a pre-heat step (260°C for 10 s) was used before measuring the OSL signals.

5. CONCLUSIONS

This study investigated the possible factors affecting the age underestimation, when using the quartz OSL-SAR protocol on loess samples from Luochuan section in the Chinese Loess plateau. The growth curves in nature, fading property, thermal stability and optical properties of the quartz OSL have been investigated. The results show

Table 2. Trap parameters for loess samples in this study, as well as those from Wintle and Murray (1998) and Singarayer and Bailey (2003) for comparison. The calibration quartz sample was provided by Risoe for the purpose to calibrate the beta source of the OSL machine.

References	Signals measured	E (eV)	s (10^{11} s^{-1})	Lifetime at 20°C (Ma)
Singarayer and Bailey (2003)	Fast component	1.74	$8.9 \cdot 10^2$	310
Wintle and Murray (1998)	Bulk OSL	1.88	$7.9 \cdot 10^4$	850
Calibration quartz (this study)	Bulk OSL	1.79	$1.4 \cdot 10^4$	393
Loess (this study)	Bulk OSL	1.48	$5.0 \cdot 10$	0.311

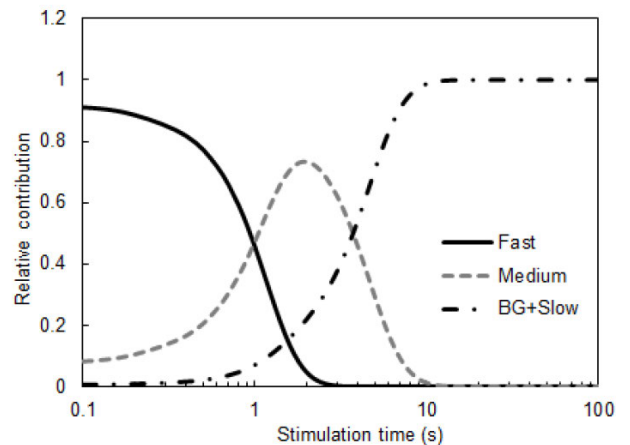


Fig. 6. Relative contribution of components from the OSL signal. The values are based on fitting of one regenerative OSL decaying curve of sample L12. The results are representative for most signals measured except some from sample L9/M.

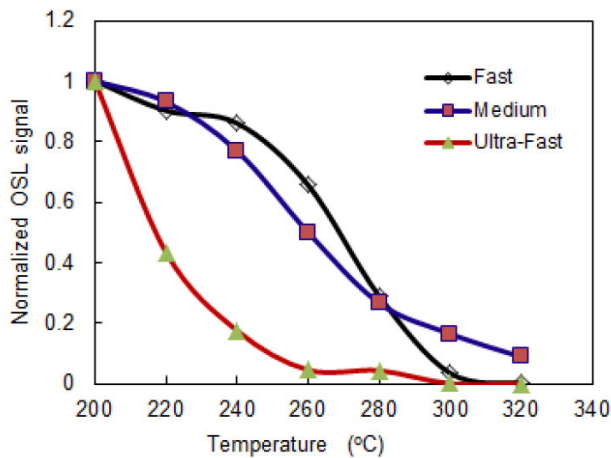


Fig. 7. Pulse annealing curve for the fast, medium and ultra-fast component from Sample L9/M. Signals of all three components are extracted from the CW-OSL curves and are normalized to their initial value.

that (1) the growth curve in nature (via L_N/T_N vs. expected dose) has a D_0 of 131 Gy, allowing the determination of up to ~262 Gy ($2D_0$) (with an OSL age of about 85 ka for loess), and it is similar to the artificial growth curve constructed in the laboratory using the SAR protocol. (2) The OSL is fast component dominated based on curve fitting analysis, and does not fade anomalously through laboratory detection, which means that anomalous fading is not a reason to explain age underestimation. (3) The barrier age of ~100 ka reported by Lai (2010) is due to the lower than expected signal thermal stability with a life-time of only 0.311 Ma at 20°C, much lower than previously reported life-time of >100 Ma for Australian quartz sample WIDG8.

ACKNOWLEDGEMENTS

This work is supported by China NSF (41172168, 41290252). We thank three anonymous reviewers for constructive comments which improved the paper.

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