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Maximum age limitation in luminescence dating of Chinese loess using the multiple-aliquot MET-pIRIR signals from K-feldspar

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Numerical dating of loess is important for Quaternary studies. Recent progress in post-infrared infrared-stimulated luminescence (pIRIR) signals from potassium-rich feldspar has allowed successful dating of Chinese loess beyond the conventional dating limit based on quartz optically stimulated luminescence (OSL) signals. In this study we tested the multiple-aliquot regenerative-dose (MAR) pre-dose multiple-elevated-temperature post-IR IRSL (pMET-pIRIR) procedure on samples from the palaeosol S5 (~480 ka) and S8 (~780 ka) layers from the Luochuan and Jingbian sections, respectively. The results show that (1) compared to sensitivity-corrected signal (L_x/T_x), a higher saturation dose is observed for the sensitivity-uncorrected MET-pIRIR signals (L_x), suggesting that MAR is advantageous for dating old samples; (2) negligible fading component can be achieved using the pMET-pIRIR procedure; (3) for the sample from the top of palaeosol S5, D_e values (1360 + 226/-167 Gy) broadly consistent with expected D_e (1550 ± 72 Gy) can be obtained using the sensitivity-uncorrected 300 °C MET-pIRIR signal. Our study suggests that a D_e value of about 1800 Gy may be the maximum dating limit of Chinese loess using the MAR pMET-pIRIR procedure.

1. Introduction

The long-term, continuously accumulated Chinese loess maintains key record for Quaternary climate and, hence, detailed numerical dating of the loess deposits is vitally important for palaeoclimate and palaeoenvironment studies. The chronology for Chinese loess was established mainly from correlating its stratigraphy and climatic proxies to marine oxygen isotopic stages (e.g. Liu, 1985). Hence, Chinese loess not only provides ideal materials for testing new luminescence dating techniques but also provides a great challenge for extending the dating limit of luminescence dating.

The fast component of optically stimulated luminescence (OSL) signals from quartz has been widely used for optical dating of sediment since the single aliquot regenerative-dose (SAR) protocol was proposed (Galbraith et al., 1999; Murray and Wintle, 2000; Wintle and Murray, 2006). However, it usually saturates at relatively low doses (~200–400 Gy), restricting its application to date Chinese loess younger than ~130 ka only (e.g. Lu et al., 2007; Lai, 2010). Infrared stimulated luminescence (IRSL) signals from K-feldspar have long been known to have higher saturation doses and thermal stability, making it a promising candidate to date older sediments (e.g. Li et al., 1997; Aitken, 1998). Compared to quartz OSL, it also has advantages such as brighter intensity and higher reproducibility and precision (e.g. Li et al., 2007). The major difficulty for using IRSL to date sediment is anomalous fading, an effect of leaking trapped electrons at a much faster rate than would be expected with regard to kinetic consideration (Wintle, 1973).

Recent progress shows that, by using prior-IR stimulations, a less-fading component could be isolated from the post-IR IRSL (pIRIR) signals at elevated temperatures (Thomsen et al., 2008). This has led to the development of two-step pIRIR procedures (e.g. Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2011) and the multiple-elevated-temperature (MET) pIRIR procedure (e.g. Li and Li, 2011a, 2012). These two procedures have been successfully tested using different types of sediment and D_e values consistent with expected D_e have been reported (e.g. Buylaert et al., 2012; Li et al., 2014b). Li and Li (2012) have shown that D_e values of

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up to ~1000 Gy can be reliably measured using the MET-pIRIR method, corresponding to a maximum age of ~300 ka for typical Chinese loess samples. More recently, further extension of the age range has been made by Li et al. (2013, 2014a), based on the observation of a pre-dose dependency of the sensitivity for the MET-pIRIR signals. They developed a pre-dose MET-pIRIR (pMET-pIRIR) method, which can reliably measure doses of up to ~1600 Gy. This method provides the potential for dating older Chinese loess samples.

In previous studies, a SAR procedure was most commonly adopted and the dose response curves (DRCs) were obtained from sensitivity-corrected signal (L_x/T_x). The validity of a SAR procedure requires reliable sensitivity correction to be met. However, potential problems of sensitivity correction, especially for the measurement of the natural signal, have been noticed recently (e.g. Vasiliniuc et al., 2012; Chen et al., 2013). For example, the sensitivity could vary significantly from the measurement of L_n to the following T_n after using these high temperature stimulations. The multiple-aliquot method, however, may avoid such potential problem because only the first cycle of measurements is used for D_e determination. In addition, multiple-aliquot measurement may also avoid other cumulative effects, such as thermal transfer, which may occur during repeated measurement cycles in a SAR procedure (e.g. Qin and Zhou, 2012). Moreover, compared to the SAR method, the MAR method can effectively save laboratory/machine time, particularly for dating old samples when large radiation doses of a few thousand grays may be applied.

In this study we aim to test the performance of a pMET-pIRIR procedure based on a multiple-aliquot regenerative-dose (MAR) measurement (Li et al., 2013), using sand-sized K-feldspar extracted from Chinese loess. The aim is to 1) compare the shape of the DRC for sensitivity-corrected and -uncorrected MET-pIRIR signals; 2) validate the performance of MAR pMET-pIRIR procedure and 3) investigate the maximum dating limit for dating Chinese loess using the pMET-pIRIR procedures.

2. Samples and analytical facilities

2.1. Samples from Luochuan and Jingbian section

The loess samples used in this study were collected from two sites, the Luochuan section in central Chinese Loess Plateau and the Jingbian section at the transition zone between the Mu Us Desert and Chinese Loess Plateau (Fig. 1). The Luochuan section has been extensively studied previously (e.g. Liu, 1985). The chronology of the 252-m-thick Jingbian section has been reported using a correlation of climate proxies to the marine oxygen isotopic record (Deng et al., 2006).

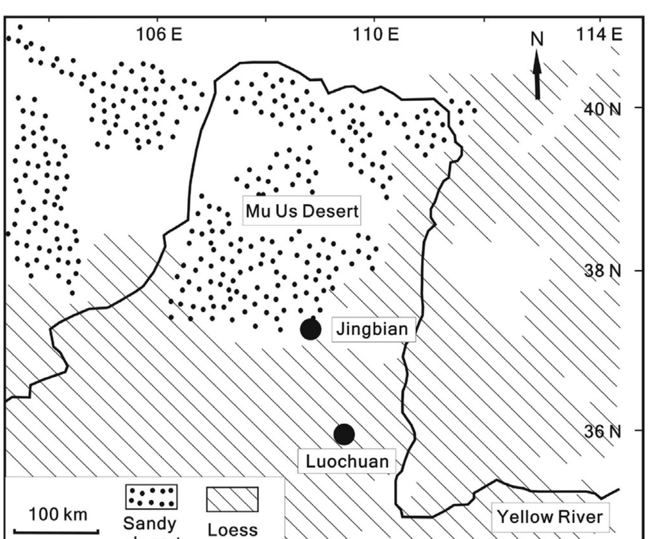

Fig. 1. Schematic map of the Luochuan section in the central Chinese Loess Plateau, and the Jingbian section from northern part of the Chinese Loess Plateau.

One sample LC-S5-1 was collected from the top of the palaeosol layer S5 at the Luochuan section. The top of S5 has been allocated to MIS 13 and, hence, this sample has an age estimate of ~480 ka (~1550 Gy) based on stratigraphic correlation (e.g. Hao et al., 2012). For the Jingbian section, one sample GJL-S8-1 was collected from the bottom of the palaeosol layer S8, corresponding to the region of the B/M boundary and has an age of 780 ± 20 ka (e.g. Coe et al., 2004; Deng et al., 2006; Wang et al., 2006). This sample is expected to have received a natural dose of about 2500 Gy based on its expected age and environmental dose rate (Table 1). Hence, it can be regarded as a 'field-saturated' sample and its IRSL traps should be in equilibrium between electron filling and escaping (e.g. Lamothe et al., 2003; Huntley and Lian, 2006).

All samples were collected by hammering stainless steel tubes (5 cm in diameter) into the cleaned section face. The tubes were then removed and wrapped in light-proof plastic for transport to the Luminescence Dating Laboratory at the University of Hong Kong.

2.2. Experimental procedures and analytical facilities

Under subdued red light, each end of the sample tubes was scraped away for water content and dose rate measurements. The samples were firstly treated by 10% HCl and 20% H_2O_2 to remove carbonates and organic materials. Grains within the range of 63–90 um for Luochuan section and 90–125 μm for Jingbian section were obtained by dry sieving. K-feldspar grains were then separated by sodium polytungstate heavy liquid. These grains were etched using 10% hydrofluoric acid for 10 min to etch the surface to remove the outer parts with alpha-irradiation contributions. Then K-feldspar grains were mounted on 9.8-mm-diameter aluminium discs with silicone oil as an adhesive. The area covered by sample grains is a rough circle with a diameter of about 5 mm, i.e. about several hundred grains for each disc.

An automated Risø TL-DA-20 reader was used for measurements, with IR diodes for stimulation (870 ± 40 nm) and a $^{90}Sr/^{90}Y$ beta source. IRSL signals were detected through the combination of one Schott BG-39 and one Corning 7–59 filters to restrict transmission to 320–480 nm. A stimulation of 100 s was adopted for each IRSL measurement. It is noted that before giving the IR stimulation at various temperatures, samples were kept at 50, 100, 150, 200, 250 and 300 °C for 10, 10, 10, 20, 50 and 50 s, respectively, to minimize the isothermal decay signals (Fu et al., 2012). For integration, the initial 10 s of signals were used, with the 'late light' subtraction over the final 10 s of signals (Aitken, 1998).

For environmental dose rate determination, the U and Th contents and their contributions to the external gamma and beta dose rates were estimated from thick-source alpha counting (TSAC) measurements (Aitken, 1985). The K contents were measured using X-ray fluorescence. The contribution from cosmic-rays dose rate was estimated from the burial depth of each of the samples and the latitude, longitude and altitude of the two sections (Prescott and Hutton, 1994). These external components of the total dose rate were adjusted for long-term water content, using estimated water contents of 10 ± 5 and 25 ± 5% for the samples from Jingbian and Luochuan, respectively, based on measured field water content. The internal beta dose rate was estimated by assuming K and Rb concentrations of 13 ± 1% and 400 ± 100 ppm, respectively (Huntley

Table 1
Summary of samples, dosimetry, expected age, and D_e and ages obtained using MAR pMET-pIRIR procedure.

Sample	Depth (m)	Unit	Expected age (ka)[a]	Grain size (um)	Alpha counting rate (cts/s)	K Content (%)	Cosmic ray (Gy/ka)	Water content (%)	Ext. Dose rate (Gy/ka)	Int. Dose rate (Gy/ka)	Total dose rate (Gy/ka)	Expected D_e (Gy)	Measured D_e (Gy)[b]
LC-S5-1	36.5	S5	480 ± 15	63–90	12.31 ± 0.181	1.85	0.021	25 ± 5	2.87 ± 0.10	0.36 ± 0.03	3.23 ± 0.11	1550 ± 72	1360 + 226/-167
GJL-S8-1	90.0	S8	780 ± 20	90–125	11.06 ± 0.165	1.40	0.002	10 ± 5	2.72 ± 0.11	0.49 ± 0.04	3.21 ± 0.12	2504 ± 114	–[b]

[a] The expected ages were obtained from stratigraphic correlations to marine oxygen isotopic records (Liu, 1985; Ding et al., 2002; Deng et al., 2006; Hao et al., 2012).
[b] The D_e values shown are obtained using the L_x/T_2 signal measured at 300 °C. The sample GJL-S8-1 has natural signals reaching the saturation level, so no D_e estimate was obtained.

and Baril, 1997; Huntley and Hancock, 2001; Zhao and Li, 2005; Li et al., 2008). The dosimetry data for both samples are summarised in Table 1.

3. MAR pMET-pIRIR procedure

A MAR pMET-pIRIR procedure, similar to that proposed by Li et al. (2013), was adopted for D_e determination. In this study, the multiple IR stimulations were achieved at 50, 100, 150, 200, 250 and 300 °C, respectively. We show that a higher MET-pIRIR stimulation temperature above 250 °C was necessary to achieve a non-fading signal for the field-saturated sample (see next sections for details). The detailed experimental procedure is shown in Supplementary Materials. In this procedure, the K-feldspar grains from each sample were divided into different groups: one group (12 aliquots) is used for natural signal measurement and the other groups (4–6 aliquots for each group) are used for measuring regenerative-dose signals. For the regenerative-dose groups, the aliquots were firstly bleached by solar simulator for ~2 h (model: Oriel 68820) to clean the light-sensitive natural signals, which is also to simulate the bleaching conditions in natural environment. After that different regenerative doses were given to different groups of bleached aliquots. Six regenerative doses 0, 125, 375, 750, 1250, and 2000 Gy were applied to the samples LC-S5-1, and six regenerative doses 0, 260, 780, 1300, 2080 and 3120 Gy were applied to the field saturated sample GJL-S8-1. The aliquots were then preheated at 330 °C for 60 s, to facilitate the measurement of 300 °C MET-pIRIR signal. The natural (L_n) or regenerative signals (L_x) from each of the groups were subsequently measured using multiple IR stimulations at 50, 100, 150, 200, 250 and 300 °C, respectively. After that, a test dose of 75 Gy was given and the corresponding test dose signals (T_1) were measured in the same way as the natural or regenerative dose signals. Finally, all the aliquots were heated to 500 °C and then given another test dose (75 Gy) and the corresponding signals (T_2) were measured in the same way as T_1.

Li et al. (2013) reported that T_1 signals are dependent on the pre-dose given to induce regenerative signals, and such a 'memory' of pre-dose could be reset by applying a high-temperature annealing. As a result, T_2 signals contain no information of pre-dose dependent sensitivity and, hence, can be used for among-aliquot normalisation, which is key for multiple-aliquot measurement. Based on the MAR pMET-pIRIR procedure, D_e can be estimated by each of the signals L_x/T_1, L_x/T_2 and T_1/T_2, respectively. However, due to the limited dose dependency of the T_1 signals, which may prevent a precise D_e determination for our samples (Li et al., 2014a), we investigated the L_x/T_1 and L_x/T_2 signals only in this study.

4. Results and discussions

4.1. The shape of DRC and saturation level

The DRCs of L_x/T_1 and L_x/T_2 signals from the sample LC-S5-1 for different MET-pIRIR signals are shown in Fig. 2a and b, respectively. To facilitate comparisons, all signals were normalised to unity at the regenerative dose of 375 Gy. The DRC data shown in Fig. 2 were fitted using a single saturating exponential function of the form $I = I_0*(1 - e^{-D/D_0}) + y$, where I is the normalised IRSL intensity, D is the regenerative dose, the value of $I_0 + y$ is the saturation value of the curve, and D_0 is the characteristic saturation dose. For the L_x/T_1 signals, it is shown that the 50 °C IRSL signal has the highest saturation level with a highest D_0 value of 843 Gy. Compare to the 50 °C IRSL signal, other MET-pIRIR signals at elevated temperatures grow indistinguishably to about ~600 Gy, and then become depressed at different levels. The D_0 value decreases from 843 to about 463 Gy with the stimulation temperature increases from 50 to 300 °C. Similar behaviours have also been reported for pIRIR (50, 290) signal and MET-pIRIR signals for aeolian sediment from

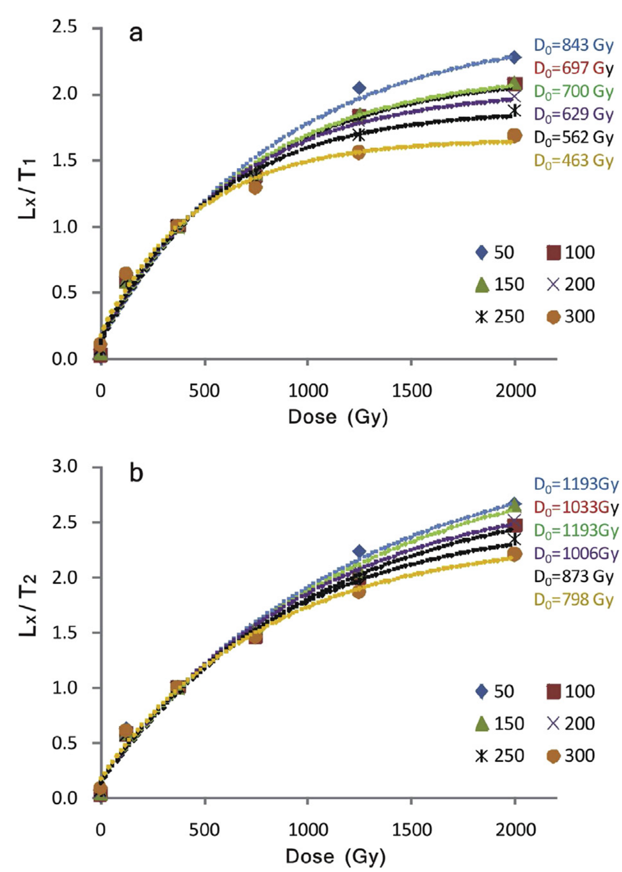

Fig. 2. The shape of DRCs and D_0 values for sample LC-S5-1. a) The DRCs of L_x/T_1 signals for different MET-pIRIR signals stimulated at 50–300 °C; and b) The DRCs of L_x/T_2 signals. Note D_0 values for L_x/T_2 signals are systematically higher than that of the L_x/T_1 signals. To facilitate comparisons, all signals were first normalised to unity at the regenerative dose of 375 Gy. The DRC data were fitted using a single saturating exponential function all through this study.

central China (e.g. Li et al., 2014b). This suggests that, although increasing the stimulation temperatures for pIRIR signals could be beneficial to obtain a less-fading component (Li and Li, 2011a), it may also result in an earlier saturation in DRC, which limits the upper dating range.

In Fig. 2b, similar behaviour is also observed for the L_x/T_2 signals and the D_0 value decreases from 1193 to about 798 Gy with the stimulation temperature increases from 50 to 300 °C. Compared with L_x/T_1 signal in Fig. 2a, systematically higher D_0 values are obtained, suggesting that L_x/T_2 signals saturate at higher doses and, hence, can be used for dating older samples. If a $2.3D_0$ value (Galbraith and Roberts, 2012) is considered as the upper limit of reliable D_e estimation (corresponding to 90% of the saturation value), it is possible to estimate D_e values as high as ~1800 Gy using the sensitivity-uncorrected signal (L_x/T_2) measured at 300 °C.

4.2. Results from the field saturated sample

For a field-saturated sample, IRSL traps are expected to be in equilibrium between electron filling and escaping, and it is expected that the natural signal intensity should be at or close to the saturation level, if a stable signal is obtained. Otherwise a natural signal significantly below the laboratory saturation level would indicate that the signals probably suffer from anomalous fading. To check whether a non-fading signal has been obtained using the pMET-pIRIR procedure, the field-saturated sample GJL-S8-1 was investigated.

The natural signals and laboratory DRCs for the different MET-

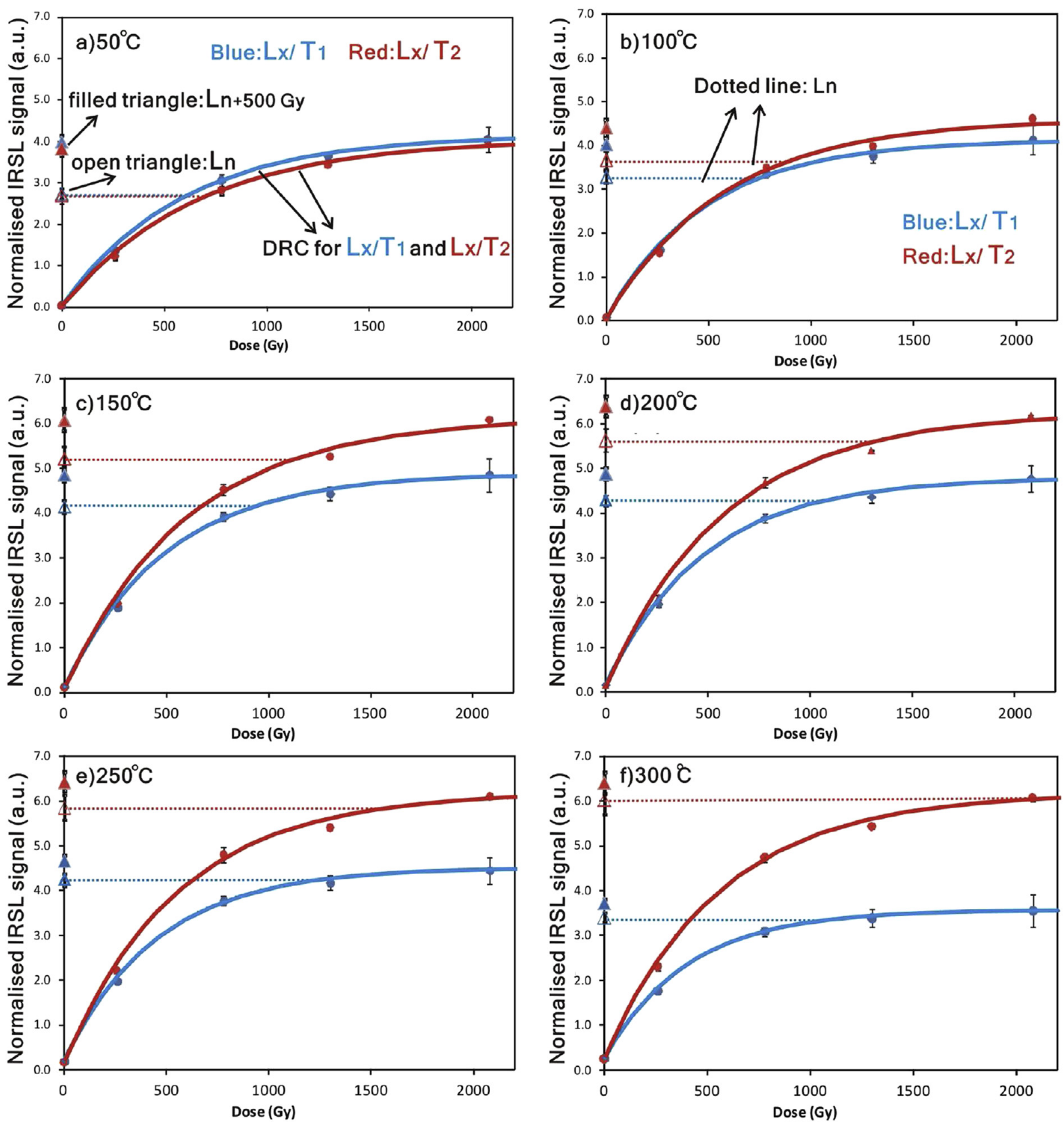

Fig. 3. DRC for a field saturated sample GJL-S8-1 (with an expected D_e of ~2500 Gy) from Jingbian section. Note the natural signal intensities (L_n) are represented by open triangles, while the N+β (500 Gy) signals ($L_{n+\beta}$) are shown by filled triangles. Blue and red dotted line shows the level of L_n/T_1 and L_n/T_2, respectively. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

pIRIR signals measured at different stimulation temperatures are shown in Fig. 3a–f. In Fig. 3a, it is observed that the natural signal of 50 °C IRSL for both sensitivity-corrected (L_x/T_1) and uncorrected (L_x/T_2) signals (open triangles and dotted lines) are considerably below the saturation level, implying that the 50 °C IRSL signal contains a large portion of fading component. As the stimulation temperature increases, however, the natural signal intensity approaches saturation level (Fig. 3a–f), i.e., the ratios between the natural intensity and the saturation level are 66, 80, 85, 89, 94 and 96% for the L_x/T_1 signals and 66, 80, 85, 90, 95 and 97% for the L_x/T_2 signals measured at 50, 100, 150, 200, 250 and 300 °C, respectively. The consistency between the natural signal and the laboratory saturation level for the 300 °C MET-pIRIR signal (Fig. 3f) confirms that a non-fading signal is obtained.

To further confirm that the pattern of increase in the natural signal compared to the laboratory saturation level with IR stimulation temperature (Fig. 3a–f) is caused by reduced anomalous fading rather than sensitivity change, we measured the signal of N+β by giving a dose of 500 Gy on top of the natural dose. It is expected that, for a field-saturated sample, the N+β signals ($L_{n+\beta}$) should be consistent with the natural signal (L_n) if a stable (non-fading) signal is obtained. Similarly, the N+β intensity should be higher than the natural intensity if an unstable or fading signal is measured. The result for the $L_{n+\beta}$ signals are shown in Fig. 3 (filled triangles). For 50 °C signal (Fig. 3a), the additional 500 Gy has resulted in ~46 and ~42% increase in the IRSL intensities for both L_x/T_1 and L_x/T_2 signals, respectively, confirming that 50 °C IRSL signal contains a large portion of fading component. At the higher stimulation temperatures, this discrepancy between L_n (open triangles) and $L_{n+\beta}$ (filled triangles) decreased, indicating reduced anomalous fading rates. A negligible increase in $L_{n+\beta}$ was observed for the signals measured at 300 °C (Fig. 3f), confirming a negligible fading component present in the 300 °C MET-pIRIR signal.

4.3. Results from the sample of S5-1

It has been noted that high temperature pIRIR signals are hard to bleach and large variations in residual dose have been reported (e.g. Li et al., 2014b). Several studies on Chinese loess have reported relative small residual doses up to a few Gy for the pIRIR signals (e.g. Li and Li, 2011a; Fu et al., 2012), we, therefore, ignore the effect of residual doses to the D_e estimation in this study.

The same MAR pMET-pIRIR procedure was applied to the sample LC-S5-1 from the top of palaeosol layer S5 of the Luochuan section. It is noted that the L_x/T_1 signals measured at elevated temperatures (>200 °C) are saturated above 1000 Gy (Fig. 2), and, as a result, they cannot produce precise D_e values for our sample. We, therefore, only determine the D_e values using the L_x/T_2 signals, and these are shown as a function of IR stimulation temperature in Fig. 4. For comparison, the expected D_e value, based on expected ages and measured environmental dose rates (Table 1), is also shown as the shaded belt in the plot. It is noted that asymptotic uncertainties on the D_e result are shown as error bars of different length on upper and lower side, because the natural signals for our sample lie close to the saturation region of the DRCs, which result in larger upper uncertainties. The 50 °C IRSL signal yields the lowest D_e values, which are considerably underestimated when compared to the expected D_e values. Increased D_e values were obtained with increasing stimulation temperatures, suggesting a reduction of the fading component as the stimulation temperature increased. The 300 °C MET-pIRIR signal yielded a D_e value of 1360 + 226/-167 Gy for the sample LC-S5-1, which is broadly consistent with the expected D_e values of 1550 ± 72 Gy obtained from the expected age and measured environmental dose rate for the sample. These results further confirm that the 300 °C MET-pIRIR signal suffers from negligible fading and a reliable D_e value could be obtained for Luochuan sample S5-1.

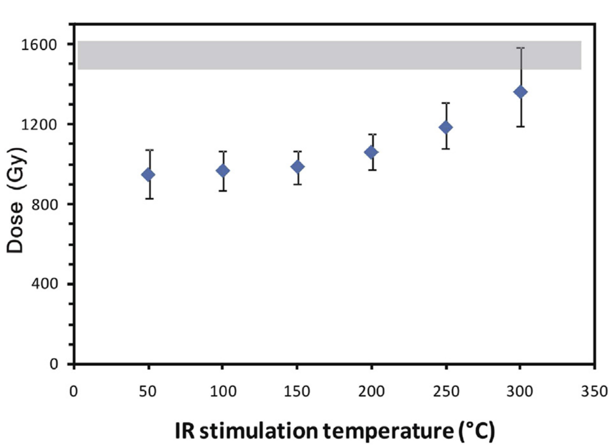

Fig. 4. The D_e values plot against stimulation temperatures (D_e-T plot) for LC-S5-1. The grey belt show the expected D_e values (Table 1).

Li and Li (2011a) suggested that a plot of D_e (or age) against IR stimulation temperature, the so-called D_e-T plot, can serve as an indicator for whether a non-fading signal has been obtained. The choice of the highest stimulation temperature in MET-pIRIR procedure is, therefore, dependent on the age of the sample measured. For older samples, a higher top stimulation temperature is generally preferred for the final step of pIRIR stimulation, as it suffers least from anomalous fading comparing to those low-T pIRIR signals (Li and Li, 2011a). This is crucial for old samples because even a small portion of fading component could significantly cause underestimation in D_e value, due to the dose dependency of the anomalous fading rate (e.g., Li and Li, 2008). Li et al. (2014a) observed that the temperature at which the age plateau is reached varies with age. Younger samples (<50 ka), appear to reach the plateau at ~200 °C, whereas older samples (50–200 ka) can only reach the plateau at ~250 °C and above. Li and Li (2012) suggested that a higher MET-pIRIR stimulation temperature above 250 °C (i.e., at 300 °C) was necessary to achieve an age plateau in the A-T plot for their Chinese loess samples (up to ~300 ka). In this study, we found that the D_e values keep increasing above 250 °C for the ~480 ka sample LC-S5-1 (Fig. 4), suggesting that the 250 °C MET-pIRIR signal may still suffer from a small fading rate, which could not be detected using laboratory fading test (Li and Li, 2012). As a result, such a small fading rate may not be important for relatively younger samples (e.g. <300 ka), but it may become significant for older samples (e.g., >300 ka). This is further supported by the observation for the field-saturated sample GJL-S8-1 that the natural MET-pIRIR 250 °C signal intensity is below the laboratory saturation level (Fig. 3e).

In an ideal situation, one should apply another IR stimulation step at higher temperatures (e.g., 350 °C) to check the existence of age plateau. However, this is impractical because a significantly high preheat temperature (>380 °C) would be required to avoid thermoluminescence in the following pIRIR signals, which may significantly deplete the main IRSL trap (e.g., Li and Li, 2011b). Other problems associated with sensitivity changes, residual doses and thermal transfer may also potentially affect the reliability of the results when both preheat and MET-pIRIR measurements are conducted at high temperatures (e.g., >350 °C). Fortunately, we have demonstrated that the MET-pIRIR 300 °C signal suffers from negligible fading, based on the observation that the natural MET-pIRIR 300 °C signal intensity of the field saturated sample GJL-S8-1 is consistent with the laboratory saturation intensity (Fig. 3f).

We, therefore, conclude that the MET-pIRIR 300 °C could be used to date older samples from Chinese loess plateau, even without the presence of age plateau.

5. Conclusion

The MAR pMET-pIRIR procedure provides a great potential for dating Chinese loess. Negligible anomalous fading can be obtained using the MET-pIRIR signal measured at 300 °C. Compared to the conventional sensitivity-corrected MET-pIRIR signals (L_x/T_1), a higher saturation dose level is observed for sensitivity-uncorrected MET-pIRIR signals (L_x/T_2). A D_0 value of ~800 Gy was obtained for the 300 °C L_x/T_2 signal, which allows reliable measurement of D_e up to ~1800 Gy. The MAR pMET-pIRIR procedure allowed successful dating of the top of palaeosol layer S5 from the Luochuan section (~480 ka, corresponding to MIS 13). Our results suggest that the maximum dating limit for Chinese loess using the sensitivity-uncorrected 300 °C MET-pIRIR signals from K-feldspar is about 500–600 ka when a typical environmental dose rate of ~3 Gy/ka is considered.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.quageo.2015.01.002.

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