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Abstract

Loess and fluvial sand are important materials for dating river terraces and alluvial fans. This study focuses on the methodological aspects of dating loess and fluvial deposits from the northern flank of the Tian Shan range, China, using sand-sized quartz and potassium (K) feldspar. Luminescence characteristics of quartz and K-feldspar were studied for searching suitable dating procedures. Our results indicate that 1) most quartz aliquots were contaminated by feldspar, and were dated using a post-infrared optically stimulated luminescence (post-IR OSL) procedure. A Fast ratio acceptance threshold of 15 can be applied to select these aliquots with post-IR OSL signals dominated by quartz OSL; 2) the multi-elevated-temperature post-IR IR stimulated luminescence (MET-pIRIR) procedures are applicable for K-feldspar. A test dose of ~30% of the natural dose is appropriate for dating of older (>10 ka) samples. An Age (T, t) plateau test can be used to evaluate the dating results; 3) for the loess samples, both quartz and K-feldspar were well bleached and are suitable for dating. Dating using K-feldspar is preferred for its higher efficiency; 4) for the fluvial sand samples, only the quartz grains were fully bleached. Single-aliquot dating of quartz gives reliable ages.

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**Optical dating of aeolian and fluvial sediments in north Tian Shan range, China:
luminescence characteristics and methodological aspects**

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Abstract: Loess and fluvial sand are important materials for dating river terraces and alluvial fans. This study focuses on the methodological aspects of dating loess and fluvial deposits from the northern flank of the Tian Shan range, China, using sand-sized quartz and potassium (K) feldspar. Luminescence characteristics of quartz and K-feldspar were studied for searching suitable dating procedures. Our results indicate that 1) most quartz aliquots were contaminated by feldspar, and were dated using a post-infrared optically stimulated luminescence (post-IR OSL) procedure. A Fast ratio acceptance threshold of 15 can be applied to select these aliquots with post-IR OSL signals dominated by quartz OSL; 2) the multi-elevated-temperature post-IR IR stimulated luminescence (MET-pIRIR) procedures are applicable for K-feldspar. A test dose of ~30% of the natural dose is appropriate for dating of older (>10 ka) samples. An Age (T, t) plateau test can be used to evaluate the dating results; 3) for the loess samples, both quartz and K-feldspar were well bleached and are suitable for dating. Dating using K-feldspar is preferred for its higher efficiency; 4) for the fluvial sand samples, only the quartz grains were fully bleached. Single-aliquot dating of quartz gives reliable ages.

Keywords: *Optical dating; Loess & fluvial sands; Tian Shan; Quartz OSL; K-feldspar post-IR IRSL*

1. Introduction

Tian Shan Range in northwest China is one of the most active orogenic belts in central Asia (Fig. S1a). It was initially formed in late Paleozoic, and was reactivated during the Cenozoic India–Eurasia collision (Molnar and Tapponnier, 1975). To interpret its mountain-building process and kinematics within the Indo-Asian collision zone, understanding the deformation pattern and uplifting rate of Tian Shan is significant. On the north piedmont of Tian Shan, extensive river terraces and alluvial fans (Fig. S1b) provide important records for the regional climatic and neotectonic evolution history (Deng et al., 2000). It is, therefore, important to establish a reliable chronological frame for these geomorphological markers, which can be achieved using optically stimulated luminescence (OSL) technique.

OSL dating has been widely applied to aeolian (e.g. Li et al., 2002, 2007; Lai, 2010) and fluvial sediments (e.g. Zhang et al., 2009) in north China. Recently, some attempts have been made to date aeolian and waterlain sediments in the northern flank of the Tian Shan, based on luminescence dating of quartz and K-feldspar (e.g., Lu et al., 2010; Gong et al., 2014). However, till date, there is no systematic investigation about the luminescence behaviors of the sediments from this region, which are important for developing suitable dating procedures. For quartz OSL dating, the single-aliquot regeneration (SAR) method (Murray and Wintle, 2000) is most suitable for samples with an OSL signal dominated by the fast component (Wintle and Murray, 2006). Alternatively, K-feldspar can be dated using infrared-stimulated luminescence (IRSL) technique. The recently developed the post-IR IRSL (pIRIR) procedures (e.g., Thomsen et al., 2008; Li and Li, 2011) can effectively overcome the problem of age underestimation associated with anomalous fading (Spooner, 1994). However, the performance of pIRIR procedures is sample dependent and may be affected by measurement conditions such as preheat/IR measurement temperatures and the size of the test dose (Li et al., 2014). Assessment of the extent of bleaching is also important for samples from different depositional settings.

In this paper, loess and fluvial sand samples from river terraces in the north flank of Tian Shan are studied. We examined the luminescence characteristics of sand-sized quartz and K-feldspar, and assessed the extent of bleaching and the reliability of the luminescence dating results for these samples. Potentials and problems about dating sediments from northern Tian Shan are discussed.

2. Samples and instruments

Eight samples aged between 1 and 55 ka were collected from six sections on the fluvial terraces along Anjihai River adjacent to the west end of the Anjihai anticline in the north flank of Tian Shan (Fig. S1b). Samples AJH-01, 02, 06, 09, 12 and 14 are loess samples overlying the fluvial sediments, which show typical characteristics of aeolian sediments. Samples AJH-03 and 05 are fluvial sand samples collected from well-sorted sand lenses within the gravel layers. The stratigraphic sketches of the sampling sections are shown in Fig. S1c, and the details of the samples are summarized in Table S1.

Coarse-grain (63-90, 90-125, 150-180 and 180-250 μm) quartz and K-feldspar were extracted using standard separation technique (Aitken, 1998, see details in supplementary). Small aliquots (~100 grains) were used in all single aliquot measurements. The measurements were performed using automated Risø TL/OSL-DA-12 and TL/OSL-DA-20 readers equipped with $^{90}\text{Sr}/^{90}\text{Y}$ β sources. Luminescence was detected by EMI 9235QA photomultiplier tubes (PMT). Blue (470nm) and IR (870 $\pm$ 40 nm) diodes were used for blue-light and IR stimulations, respectively. For quartz OSL measurements, three 2.5 mm Hoya U-340 filters were fitted in front of the PMT. For K-feldspar IRSL measurements, a filter pack of Schott BG-39 and Corning 7-59 filters was used. An ORIEL solar simulator (model: 68820) was used for bleaching. Details about annual doses are summarized in Table S1.

3. Luminescence characteristics and performance of the protocols

We first investigated the luminescence properties of quartz and K-feldspar from loess samples, which are expected and demonstrated to be well bleached (section 4).

3.1. Quartz

Although repeated HF etching was conducted, nearly all aliquots of the quartz fractions exhibited weak IRSL signals (from 10 to < 500 cts/0.1 s), and yielded IR depletion ratios (Duller, 2003) between ~ 0.3 and 1.0 (>70% below 0.7, Fig.S3a), indicating that feldspar had not been completely removed. This may be because of the feldspar inclusions in quartz grains. To minimize the impact of feldspar contamination, a post-IR OSL procedure (e.g. Zhang and Zhou, 2007) was adopted (Table 1a). In this procedure, an IR bleaching of 100 s at 125°C was conducted before the post-IR OSL measurement (with blue light stimulation at 125°C for 40 s) to remove the feldspar signal. For samples < 10 ka, a 200°C preheat for 10 s was conducted; for older samples, a 240°C preheat for 10 s was applied. The corresponding cutheat temperatures were 160 and 200°C, respectively. These measurement conditions were validated by the dose recovery experiments (Fig. S4). In equivalent dose (D_e) calculation, the initial 0.2 s integral of the post-IR OSL signal minus backgrounds (BG) estimated from the last 5 s integral were used. For the accepted aliquots (see below), the recuperation was generally below 5%, and the recycling ratios were generally within 1.0 ± 0.1 . Only a few aliquots did not fulfill these criteria and were rejected (<10%).

It has been reported that the feldspar contamination may not be totally eliminated using a post-IR OSL procedure, although it can significantly reduce it (Roberts, 2007). Therefore, the main difficulty for dating our samples remained the elimination of feldspar contamination, as we found this contamination could significantly affect the dating results (see below). Our initial measurements revealed a large inter-aliquot

variability in decay rate and brightness for the post-IR OSL decay curves. Some aliquots showed stronger luminescence intensity and decay faster, whereas much more aliquots showed weaker luminescence signal and slower decay rate (Fig. S2). As the decay of the OSL signal from feldspar is usually much slower than the fast component of quartz OSL (e.g. Li and Li, 2006b), it is expected that aliquots showing higher decay rate are less influenced by feldspar contamination. To characterize the post-IR OSL decay rates for different aliquots, we calculated the Fast ratio (Madsen et al., 2009; Durcan and Duller, 2011) for our samples, which is defined as:

$$\text{Fast ratio} = (L_{0-0.2s} - \text{BG}) / (L_{2-2.2s} - \text{BG})$$

where $L_{0-0.2s}$ and $L_{2-2.2s}$ are the counts from 0 to 0.2 s and 2 to 2.2 s of the post-IR OSL stimulation, respectively. The former parameter is a proxy for the fast component, and the latter is a proxy for the medium and slow components (Fig. S2). BG is the background evaluated from the last 5 s of the post-IR OSL signal. A higher value of the Fast ratio indicates a higher decay rate and a greater dominance of the fast component, i.e., a greater dominance of quartz OSL signal. In Fig.1, the Fast ratio distribution of 42 aliquots of a representative loess sample AJH-06 is shown. The values of the Fast ratio vary significantly between 2 and 42, indicating a great variability of the fast component proportion among aliquots.

To test if the Fast ratio can effectively be used to identify those aliquots with post-IR OSL signal dominated by quartz OSL, a range of Fast ratio acceptance threshold was tested for D_e calculation, i.e. the aliquots with Fast ratio lower than this threshold were discarded for D_e calculation. Fig. 2a shows that for sample AJH-06, when the threshold value varies from 1 to 15, the D_e value obtained gradually increases from 23 to 31 Gy; when a Fast ratio threshold of 15 or higher is used, a plateau in D_e (~ 31 Gy) is reached. A comparison of this D_e plateau with the expected D_e based on the K-feldspar MET-pIRIR age shows that they agree very well (Fig. 2a). This supports that the D_e estimation based on the Fast ratio- D_e plateau is reliable. Compared with

the D_e plateau, D_e values for aliquots with a Fast ratio < 15 showed underestimation. This underestimation is expected to be mainly because of the interference of feldspar signal, and may also partially be due to the effect of unstable medium component from quartz (e.g. Li and Li, 2006a). Apart from identifying aliquots with more reliable D_e , applying a higher Fast ratio threshold selection also reduces the value of overdispersion (Galbraith et al., 1999), which is similar to the findings of Duller (2012). Fig. 2b shows that with the Fast ratio threshold increasing from 1 to 15, the overdispersion value decreases from $\sim 33\%$ to less than 15%. This implies that the overdispersion in D_e of our samples largely arises from the variable luminescence characteristics among different aliquots. Fig. 2c shows that as the Fast ratio threshold increases, the percentage of aliquots accepted reduces obviously. For sample AJH-06, only 30% of the measured aliquots passed a Fast ratio threshold of 15.

The IR depletion ratio is another criterion that demonstrates the significance of feldspar contamination (Duller, 2003). A positive correlation was observed between the Fast ratio and the IR depletion ratio for our samples (e.g. Fig.S3b). Therefore, it is expected that using IR depletion ratio as a rejection criterion may also help identifying post-IR OSL signals dominated by quartz OSL signal. The effect of IR depletion ratio acceptance threshold on D_e is illustrated in Fig.S3c, using sample AJH-06. It is observed that when an IR depletion ratio acceptance threshold value of 0.7 or above is used, a plateau in D_e can be reached. This IR depletion ratio- D_e plateau is consistent with the Fast ratio- D_e plateau (Fig. 2a), suggesting that the two criteria take effect in a similar manner. Compared with the IR depletion ratio, the advantage of the Fast ratio is that it can be directly obtained from the post-IR OSL signal, i.e., it requires no further measurements; besides that, it is more straightforward to interpret the value of a Fast ratio threshold. Based on these, the Fast ratio is selected as the leading rejection criteria in this study. Based on Fig. 2, which is representative for our samples, a Fast ratio acceptance threshold of 15 was selected for our samples. This value means that for the accepted aliquots, $> 85\%$ of the signal in the first channel originates from the fast component. After the Fast ratio threshold selection, the

accepted aliquots generally showed brighter luminescence signals compared to the rejected aliquots (as in Fig. S2a). For our loess samples, ~11–33% of the aliquots measured were accepted after applying a Fast ratio threshold of 15. An even lower percentage (< 10%) was accepted for the fluvial sand samples. Such a low percentage of acceptable aliquots implies that dating our samples using quartz would be a time-consuming choice.

3.2. K-feldspar

K-feldspar was measured using two modified multi-elevated-temperature pIRIR procedures (MET-pIRIR, Li and Li, 2011). For samples of Holocene ages, a three-step pIRIR procedure with lower IR stimulation temperatures at 110, 140 and 170°C was applied (low-temperature pIRIR in Table 1b, Fu and Li, 2013); for older samples, a three-step pIRIR procedure with higher IR stimulation temperatures at 150, 200 and 250°C was conducted (high-temperature pIRIR in Table 1c, Fu, 2014). Examples of IRSL and pIRIR decay curves for the two procedures are given in Fig.S5. In D_e calculations, the initial 2 s integral of each signal minus the BG estimated from the last 5 s were used.

Recently, a plateau test termed the D_e (T, t) plot (T and t represent the IR measurement temperature and time, respectively) has been proposed to justify the pIRIR procedures and for evaluating the dating results (Fu, 2014). This plot is a combination of the D_e -T plot (Li and Li, 2011) that indicates the effect of T on D_e , and the D_e (t) plot (Bailey, 2000), which demonstrates the effect of t on D_e . The presence of a plateau in the D_e (T, t) plot indicates that a non-fading signal has been achieved, and the sample was well bleached (Fu, 2014). To justify the procedures in Table 1b and c, this test has been applied to our dating results. In Fig. 3a and b, examples of Age (T, t) plots (similar to the D_e (T, t) plot, but with age plotted) for two well-bleached loess samples AJH-02 and AJH-12, measured using the low-temperature and high-temperature pIRIR methods (Table 1b and c) respectively

are shown. For comparison, the Age (T, t) plots obtained using the five-step MET-pIRIR methods (Li and Li, 2011; Fu and Li, 2013) and the quartz post-IR OSL ages are also given. For all pIRIR methods, age plateaus were achieved in the later part of the Age (T, t) plots, which were consistent with the quartz ages, indicating that non-fading signals have been obtained. The age plateaus obtained using the three-step pIRIR methods were quite consistent with that of the five-step MET-pIRIR methods (Fig.3). This suggests that the three-step pIRIR procedures (Table 1b and c) can isolate the non-fading signals as effectively as the five-step MET-pIRIR methods, when using the Age (T, t) plot as a plateau test. Based on the Age (T, t) plot tests as in Fig. 3, the 170 and 250°C pIRIR signals in the low-temperature and high-temperature three-step pIRIR procedures (Table 1b and c) are non-fading. Therefore, they were used in final age calculations for our samples, with no fading correction being applied. The pIRIR procedures in Table 1b and c also yielded acceptable recuperations (<10%) and good recycling ratios (within 1.0 ± 0.1) for all the samples.

The residual doses of the low-temperature pIRIR signals after 4 hours' solar bleaching were measured to be ~ 1 Gy or lower, which are much smaller than that of the high-temperature pIRIR signals (~ 3 Gy) (Fig. S6). Therefore, applying the low-temperature pIRIR procedure for younger samples is advantageous for minimizing the effects of residual dose, as suggested by Fu and Li (2013).

Qin and Zhou (2012) have reported a dependence of D_e on the size of test dose in pIRIR dating. To see such effect, we measured two loess samples AJH-01 (~ 1 ka) and 14 (~ 28 ka) using different test doses, using the low-temperature and high-temperature pIRIR procedures (Table 1b and c), respectively (Fig. 4a and b). The low-temperature pIRIR procedure shows no dependence of the D_e on the test dose, when the test doses varies from 3 to 20 Gy (Fig. 4a). For the high-temperature pIRIR procedure (Fig. 4b), there is no systematic dependence of D_e on test dose between 25 to 50 Gy, but when the test dose is smaller than 25 Gy or larger than 50 Gy, the D_e value decreases with the test dose increasing. This suggests that the size of test dose

can affect the dating results for the high-temperature pIRIR procedure. To determine appropriate test doses for our pIRIR procedures, dose recovery tests were conducted (Fig. 4c and d). In the experiments, aliquots of sample AJH-01 were divided into two groups. In one group, the aliquots were given a β irradiation of 13.6 Gy, and then measured using the low-temperature pIRIR procedure (Table 1b), with test dose varying between 13 and 200% of the given dose; in another group, a β irradiation of 126.9 Gy was administered to the aliquots, and these aliquots were then measured using the high-temperature pIRIR procedure (Table 1c), with the test dose varying between 5 and 100% of the given dose. The natural dose was subtracted when calculating the dose recovery ratio. Fig. 4c shows that for the low-temperature pIRIR procedure, there is no dependence of the dose recovery ratios on the test dose. All the signals give dose recovery ratios between 0.95 and 1.07 with different test doses. For the high-temperature pIRIR procedure, however, the pIRIR signals could recover the given dose only when the test dose is $< 40\%$ of the given dose (Fig. 4d, dose recovery ratios are ~ 0.93 and 0.96 for the pIRIR 200 and 250°C signals). When the test dose increases from 40 to 100% of the given dose, the dose recovery ratios for the pIRIR signals decrease from > 0.9 to ~ 0.8 . The results in Fig. 4 imply that for our low-temperature pIRIR procedure, test doses up to $\sim 200\%$ of the natural doses are all applicable; but for the high-temperature pIRIR procedure, using a large test dose ($> 40\%$ of the natural dose) should be avoided. On the basis of Fig. 4, we administrated test doses of ~ 50 to 100% of the natural doses when applying the low-temperature pIRIR procedure, and $\sim 30\%$ of the natural doses when applying the high-temperature pIRIR procedure in final age measurements.

4. The extent of signal resetting

To assess the extent of signal resetting for our samples, the D_e distribution characteristics of our samples were studied, and the ages of quartz and K-feldspar were compared. In K-feldspar age calculations, we subtracted the residual doses for younger samples (< 10 ka) dated using the low-temperature pIRIR procedure,

although they are quite small. For older samples (>10 ka) dated using the high-temperature procedure, it is not necessary to make the correction as the residual doses are negligible compared to the natural ones ($< 3\%$).

Fig. 5 shows the quartz and K-feldspar D_e distributions of three typical samples and their K-feldspar Age (T, t) plots. For a younger loess sample AJH-01 (~ 1 ka, Fig. 5a, b and c) and an older loess sample AJH-12 (~ 50 ka, Fig. 5d, e and f), all the quartz and K-feldspar D_e values distribute homogeneously, with small overdispersion lower than 0.20. Most of the D_e values are within 2σ of the central values obtained using the central age model (Galbraith et al., 1999, CAM) for all signals. In the K-feldspar Age (T, t) plots, age plateaus consistent with the quartz post-IR OSL ages have been obtained (Fig. 5c and f). The D_e distribution characteristics and Age (T, t) plots for samples AJH-01 and 12 are representative for our loess samples. These characteristics indicate that both quartz and K-feldspar fractions in our loess samples were fully bleached, and therefore are suitable for dating.

Fig. 5g, h and i show an example of a fluvial sand sample AJH-05 (~ 4 ka). The quartz D_e distribution shows well-bleached characteristics, with an overdispersion of 0.14 (Fig. 5g). Larger scatter in D_e , however, are observed for different signals from K-feldspar (Fig. 5h). The overdispersions of the 110, 140 and 170°C signals are 0.23, 0.23 and 0.33, respectively. The greater scatter of K-feldspar D_e might possibly be attributed to two reasons: incomplete signal resetting or inhomogeneity in micro-dosimetry. Given a homogeneous D_e distribution for quartz, incomplete bleaching is more likely to be the cause. The K-feldspar Age (T, t) plot for sample AJH-05 shows an increasing pattern and yields no plateau (Fig. 5i). Fu (2014) showed that IRSL signal related to the later part of the Age (T, t) plot (with T and t increasing) is harder to bleach. Based on this, it is suggested that an increasing pattern in the Age (T, t) plot such as in Fig. 5i implies insufficient bleaching. An overestimation of K-feldspar pIRIR ages compared with the quartz age (Fig. 5i) also supports that the K-feldspar was not fully bleached. Another fluvial sand sample AJH-03 behaves

similar to that of sample AJH-05. The above characteristics suggest that the quartz grains in the fluvial sand samples should be better bleached than K-feldspar grains during deposition, and should give more reliable dating results for these samples. To further check whether the quartz grains in our fluvial sand samples were well bleached, additional single grain measurements have been conducted for quartz grains from the two fluvial sand samples (see details in Appendix G). The quartz single grain overdispersions of the two fluvial sand samples were both 0.17. The single grain ages (5.09 ± 0.47 and 4.40 ± 0.29 ka) agree well with the single aliquot ages (5.32 ± 0.29 and 4.14 ± 0.29 ka). These support that quartz grains in the fluvial sand samples were well-bleached, and can be dated using single aliquot dating.

The quartz post-IR OSL and K-feldspar pIRIR ages for all samples are compared in Fig. 6 (and summarized in Table S2). For all loess samples, the two ages (obtained using CAM) are consistent with each other within uncertainties. This further indicates that both quartz and K-feldspar in the loess samples were well bleached, and can both be used for dating. The CAM ages of K-feldspar overestimate the quartz ages for the two fluvial sand samples (Fig.6). This implies that the K-feldspar was insufficiently reset, as suggested by the Age (T, t) plot (Fig. 5i). To test whether a well-bleached component can be separated using an age model, the minimum age model (MAM, Galbraith et al., 1999) has also been applied to the K-feldspar dating results of the fluvial sand samples. In running the MAM, the σ_b value was set to be 0.10, on the basis of dose recovery results, and also allowance made for micro-dosimetry heterogeneity. For one sample, AJH-05, the K-feldspar MAM age agrees with the quartz age; however, for the other sample AJH-03, the K-feldspar MAM age still shows overestimation (Fig. 6). This implies that applying the MAM to single aliquot dating results for K-feldspar can select aliquots with smaller D_e , but it may not necessarily eliminate the effect of partial bleaching, because of the averaging effect when measuring multi-grains (e.g. Trauerstein et al., 2014). Therefore, single aliquot dating using quartz is more appropriate for the fluvial sand samples.

5. Discussion and Conclusions

Quartz and K-feldspar from loess and fluvial sand samples in the north flank of Tian Shan showed complicated luminescence characteristics. The post-IR OSL signals from quartz exhibited significant inter-aliquot difference in signal brightness and decay rate. The majority of aliquots have dim post-IR OSL signals dominated by non-fast components, whereas only a small proportion of them have bright and fast-component-dominated post-IR OSL signals. This indicates great differences in sensitivity and fast component content between quartz grains. Quartz was reported to be sensitized, especially in their fast component, with longer transportation distance (e.g. Preusser et al., 2009). Therefore, it is deduced that the abundant dim quartz grains in our samples may originate from nearby bedrocks (e.g. from the Tian Shan range), whereas the fewer bright and fast-component dominated quartz grains might be from a more distant origin (bright grains in fluvial sand samples might be of aeolian origin, and afterwards deposited into the river). We suggest applying a Fast ratio acceptance threshold of 15 to the post-IR OSL signals to extract the fast-component dominated aliquots, which are less affected by feldspar contamination and more favorable for dating. This approach is shown to be fast and effective for our samples. However, given the low proportion of the fast-component dominated grains, dating these samples using quartz is rather time consuming. An efficient method is to measure the natural signal only, and then select only those grains or aliquots dominated by the fast component for further measurement of regenerative doses.

K-feldspar dating provides another option. The Age (T, t) plateau tests confirmed that the multi-step pIRIR dating procedures (Table 1) can isolate non-fading signals for our samples (Fig. 3). It is also advised to combine such plateau test (Li and Li, 2011; Fu, 2014) with D_e distribution characteristics to reveal the extent of signal resetting for the K-feldspar grains (as in Fig. 5). Large micro-dosimetry variation can result in significant scatter in D_e distribution, similar to the effect of partial bleaching. However, as whether a plateau can be achieved in the Age (T, t) plot is only related to

anomalous fading and residual dose (partial bleaching) (Fu, 2014) rather than D_e distribution, the micro-dosimetry variation does not have a significant effect on the Age (T, t) plot. A dependence of D_e values on test dose was observed when dating older (> 10 ka) K-feldspar samples using the high temperature pIRIR procedure (Fig. 4). A test dose of $\sim 30\%$ of the natural dose is suggested to be used in dating these older samples (Fig. 4). It is interesting to note that our observation is contrary to the results of Qin and Zhou (2012), who suggested using larger test doses in pIRIR measurements, as it can reduce the effect of thermal transfer. A low recuperation ($< 10\%$) has been observed for all signals of our samples. Therefore, we deduce that factors other than thermal transfer, such as the dose-induced sensitivity change (Li et al., 2013), may account for this dependence.

D_e distribution and age comparison show that both quartz and K-feldspar in our loess samples were well bleached (Figs. 5 and 6). We recommend using K-feldspar for dating the loess samples, as it is less time consuming and more precise than dating using quartz. However, for fluvial sand samples, K-feldspar grains are found to be insufficiently bleached, whereas quartz grains are found to be well bleached (Figs. 5 and 6). This is expected because the IRSL signal from K-feldspar is bleached slower than the quartz OSL signal (Godfrey-Smith et al., 1988), whereas the pIRIR signal is even harder to bleach (e.g. Li and Li, 2011; Fu et al., 2012b). Based on this, quartz is suitable for dating fluvial sand samples in the studied area. A single-grain pIRIR dating method for K-feldspar (e.g. Reimann et al. 2012, Nian et al. 2012, Trauerstein et al. 2014) might be developed in the future to date these poorly bleached samples.

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Captions

Fig. 1. Histogram showing the Fast ratio distribution of post-IR OSL signals for 42 quartz aliquots of loess sample AJH-06.

Fig. 2. The impacts of varying the Fast ratio acceptance threshold value upon a) the D_e value obtained; b) the overdispersion of the D_e values for the accepted aliquots, and c) the percentage of aliquots accepted. The data are from 42 aliquots of quartz of sample AJH-06. The gray belt in a) represents the expected D_e from K-feldspar age.

Fig. 3. Age (T, t) plots for K-feldspar from representative samples. a) K-feldspar ages of sample AJH-02, obtained using the low-temperature three-step pIRIR procedure in Table 1b (open circles) and the 30°C-increment five-step MET-pIRIR method of Fu and Li (2013) (open diamonds), plotted against the cumulative IR measurement time; b) K-feldspar ages of sample AJH-12, obtained using the high-temperature three-step pIRIR procedure in Table 1c (open circles) and the 50°C-increment five-step MET-pIRIR method of Li and Li (2011) (open diamonds), plotted against the cumulative IR measurement time. The quartz post-IR OSL ages are given as gray belts.

Fig. 4. The dependence of D_e values on test dose for K-feldspar dating procedures. a) Natural doses for the low-temperature pIRIR procedure (Table 1b), using sample AJH-01 (~1 ka); b) Natural doses for the high temperature pIRIR procedure (Table 1c), using sample AJH-14 (~28 ka); c) Dose recovery results for the low-temperature procedure; d) Dose recovery results for the high-temperature procedure. See details of dose recovery tests in the text.

Fig. 5. D_e distributions and K-feldspar Age (T, t) plots for three representative samples: (i) a younger loess sample AJH-01; (ii) an older loess sample AJH-12, and (iii) a

fluvial sand sample AJH-05. a), d) and g): quartz post-IR OSL D_e values shown as radial plots; b), e) and h): K-feldspar IRSL and pIRIR D_e values shown as radial plots. c), f) and i): Age (T, t) plots for K-feldspar, with the quartz post-IR OSL ages given as gray belts for comparison. Note that for samples AJH-01 and 05, the K-feldspar fractions were measured using the low-temperature pIRIR procedure (Table 1b), whereas for sample AJH-12, the K-feldspar fraction was measured using the high-temperature pIRIR procedure (Table 1c). All central values in the radial plots were obtained using the CAM.

Fig. 6. Comparison between quartz post-IR OSL and K-feldspar pIRIR ages. K-feldspar pIRIR ages measured with the low-temperature and high-temperature pIRIR procedures (Table 1b and c) were from the 170 and 250°C pIRIR signals, respectively. For the loess samples (open circle), CAM was applied to both quartz and K-feldspar fractions. For the fluvial sand samples, CAM was applied to quartz, and CAM (open triangle) and MAM (open diamond) were applied to K-feldspar separately. The inset shows enlarged scale of 0-12 ka.

Table 1 Summary of dating procedures: a) the post-IR OSL procedure for quartz; b) the low temperature and c) the high temperature post-IR IRSL procedures for K-feldspar

	(a) post-IR OSL for quartz	(b) Low-temperature pIRIR for K-feldspar ^a	(c) High-temperature pIRIR for K-feldspar ^a
Step	Treatment/Observed	Treatment/Observed	Treatment/Observed
1	Regenerative dose	Regenerative dose	Regenerative dose
2	PH (240 or 200°C, 10s)	PH (200°C, 60s)	PH (300°C, 10s)
3	IRSL (125°C, 100s)	IRSL (110°C, 100s)/ Lx ₁₁₀	IRSL (150°C, 100s)/ Lx ₁₅₀
4	OSL (125°C, 40s)/ Lx	IRSL (140°C, 100s)/ Lx ₁₄₀	IRSL (200°C, 100s)/ Lx ₂₀₀
5	Test dose	IRSL (170°C, 100s)/ Lx ₁₇₀	IRSL (250°C, 100s)/ Lx ₂₅₀
6	CH (200 or 160°C)	Test dose	Test dose
7	IRSL (125°C, 100s)	PH (200°C, 60s)	PH (300°C, 10s)
8	OSL (125°C, 40s)/ Tx	IRSL (110°C, 100s)/ Tx ₁₁₀	IRSL (150°C, 100s)/ Tx ₁₅₀
9	OSL (260 or 220°C, 100s)	IRSL (140°C, 100s)/ Tx ₁₄₀	IRSL (200°C, 100s)/ Tx ₂₀₀
10	Return to step 1	IRSL (170°C, 100s)/ Tx ₁₇₀	IRSL (250°C, 100s)/ Tx ₂₅₀
11		IRSL (280°C, 100s)	IRSL (320°C, 100s)
12		Return to step 1	Return to step 1

^a For each IRSL and pIRIR measurement, an “IR-off” period of 5 s was applied to minimize the isothermal signal (Fu et al., 2012a).

Note: PH refers to preheat, and CH refers to cutheat.

Fig. 1.

Fig. 2.

Fig. 3.

Fig. 4.

Fig. 5.

Fig. 6.

Supplementary

Appendix A: Sampling area and sampling sections

Fig.S1. a) Geologic map showing the general structural setting in central Asia and the location of the Tian Shan Range (after Gong et al., 2014). b) Geologic interpretative map in northern foreland of Tian Shan (after Gong et al., 2014), showing the location of the sampling sections (the red star). Abbr. for anticline names: QSH: Qingshuihe; QG: Qigu; CJ: Chnagji; AJH: Anjihai; DSZ: Dushanzi; HGS; Huoergus; HTB: Hutubi; MNS: Manas; TGL: Tugulu; XH: Xihu; c) Stratigraphy, sampling depths and final ages for the sampling sections. The final ages of the loess samples are calculated from the weighted mean of the quartz post-IR OSL ages and the K-feldspar pIRIR ages. The final ages of the fluvial sand samples are the post-IR OSL ages of quartz.

Appendix B: Sample preparation

Sample preparation was carried out under subdued red light. Quartz and K-feldspar grains were extracted using a standard separation technique (Aitken, 1998). The original samples were first treated with 10% HCl and 30% H₂O₂ to remove carbonates and organic matters, respectively. Then, the samples were dry-sieved to obtain grain sizes of 63-90, 90-125, 150-180 and 180-250 μm . To separate quartz and K-feldspar grains, density separation was carried out using heavy liquids with densities of 2.58, 2.62 and 2.75 g/cm³. Grains with density lower than 2.58 g/cm³ are mainly K-feldspar (e.g. Li et al., 2011). These grains were etched with 10% HF for 40 minutes to remove the α -dosed layer. Grains with density between 2.62 and 2.75 g/cm³ were etched with 40% HF for 90 minutes, with a purpose of extracting pure quartz extracts. However, probably due to presence of feldspar inclusion within quartz grains, we only got quartz extracts contaminated with feldspar, as indicated by the presence of infrared stimulated luminescence (IRSL) signals for our quartz aliquots (e.g. Smith et al., 1990; Stokes, 1992). For single aliquot dating, the grains were mounted on 9.8 mm diameter aluminum discs using the “Silkospay” silicone oil, and small aliquots (~100 grains) were used in all measurements. For single grain dating, 1-cm-diameter aluminum sample discs with ten by ten arrays of holes drilled into the surfaces (Duller and Murray, 2000; Bøtter-Jensen et al., 2000; Duller, 2004) were used to carry the measured grains.

Appendix C: Details of the samples and summary of dosimetry

Details about the samples and their annul dose evaluations are summarized in Table S1. For annual dose evaluation, the contributions from U and Th were measured using a thick-source alpha counting technique (Aitken, 1985). The content of K was measured using X-ray fluorescence. Water contents of the loess and fluvial sand samples were estimated to be $10 \pm 2\%$ and $5 \pm 4\%$ respectively, based on the field-measurement results. Cosmic-ray dose rate was calculated following Prescott and Hutton (1994). The internal K and Rb contents were assumed to be $13 \pm 1\%$ and 400 ± 100 ppm, respectively (Huntley and Baril, 1997; Huntley and Hancock, 2001; Zhao and Li, 2005; Li et al., 2007).

Table S1 A summary of the OSL samples and the dosimetry data

Sample	Lithology	Depth (m)	Grain size (μm)	a-counting (cts/ks)	K content (%)	Cosmic ray (Gy/ka)	Water content (%)	Quartz dose rate (Gy/ka)	K-feldspar dose rate (Gy/ka)
AJH-01	loess	0.2	90-125	13.01 ± 0.23	2.21	0.23	10 ± 2	3.92 ± 0.16	4.41 ± 0.16
AJH-02	loess	0.8	90-125	11.81 ± 0.22	2.18	0.22	10 ± 2	3.73 ± 0.15	4.22 ± 0.17
AJH-03	fluvial sand	1.2	150-180 180-250	9.10 ± 0.18	2.14	0.20	5 ± 4	3.44 ± 0.18 3.39 ± 0.18	4.16 ± 0.20
AJH-05	fluvial sand	0.9	150-180 180-250	9.56 ± 0.25	2.15	0.21	5 ± 4	3.52 ± 0.19 3.48 ± 0.18	4.24 ± 0.20
AJH-06	loess	0.6	90-125	12.30 ± 0.23	2.10	0.22	10 ± 2	3.72 ± 0.15	4.21 ± 0.16
AJH-09	loess	3.0	63-90	12.55 ± 0.27	2.04	0.16	10 ± 2	3.67 ± 0.16	4.03 ± 0.16
AJH-12	loess	2.0	90-125	13.48 ± 0.28	1.99	0.18	10 ± 2	3.74 ± 0.16	4.23 ± 0.16
AJH-14	loess	2.0	90-125	11.69 ± 0.26	1.97	0.18	10 ± 2	3.49 ± 0.15	3.98 ± 0.16

Appendix D: Some luminescence characteristics of quartz

For our quartz samples, two types of aliquots have been observed. One type has stronger luminescence intensity and decays faster; while the other type has weaker luminescence signal and a slower decay rate. Representative post-IR OSL decay curves of the two types of aliquots are shown in Fig. S2a and d. Component analysis based on deconvolution of decay curves showed that the former decay curves are dominated by the fast component (Fig. S2b and e). In order to characterize the proportion of the fast component for different aliquots, we calculated the Fast ratio (Madsen et al., 2009; Durcan and Duller, 2011) for our samples. This ratio is defined as signals over a period of time dominated by the fast component divided by signals of a time period dominated by the medium component (both minus backgrounds). It has been suggested that the later time interval should be at the point when the fast component decays to 1% of its initial signal, and the medium components become most dominant (Durcan and Duller, 2011). Based on component analysis for a series of aliquots (as in Fig. S2c and f), the average time for the fast component to decay to ~1% for our samples is ~2 s. Therefore, in this study the Fast ratio is defined as:

$$\text{Fast ratio} = (L_{0-0.2s} - \text{BG}) / (L_{2-2.2s} - \text{BG})$$

where $L_{0-0.2s}$ and $L_{2-2.2s}$ are the integrals of the 0-0.2 s and 2-2.2 s of the post-IR OSL signal, respectively. The backgrounds (BG) were evaluated from last 5 s of the post-IR OSL decay curves.

In the main text, it is shown that the Fast ratio can be used to identify those aliquots with post-IR OSL signal dominated by quartz OSL (Fig.2). Here, we are trying to test whether this can also be achieved using an IR depletion ratio selection. The distribution of IR depletion ratio for 42 aliquots of sample AJH-06 is shown in Fig.S3a. In Fig.S3b, the IR depletion ratios of these aliquots are plotted against their corresponding Fast ratios. Basically, a positive relationship was observed between the two criteria. In Fig. S3c, the impact of varying the IR depletion ratio acceptance threshold value upon the D_e value is shown. A plateau of D_e has been reached when using a threshold value of 0.7 or above. This plateau (~31 Gy) is consistent with the D_e plateau detected using the Fast ratio threshold (Fig.2a), and also agrees with the expected D_e from K-feldspar age (shown as gray belt). This suggests that using the IR

depletion ratio as a rejection criterion might also give reliable dating results. However, considering that 1) obtaining the IR depletion ratios require extra measurements on each aliquot; 2) it is not straightforward to interpret the meaning of a certain IR depletion ratio threshold; and 3) the suitable IR depletion ratio threshold for different samples might not be identical, we did not choose the IR depletion ratio as the primary selection criterion in this study.

In order to validate the post-IR OSL protocol listed in Table 1a, dose recovery tests were carried out using quartz aliquots of sample AJH-06 with Fast ratio >15. Two sets of aliquots (5 aliquots for each set) were bleached using the blue LED for 200 s at 125 °C to remove the natural signals (Murray and Wintle, 2003). After that, the aliquots were stored for 10,000 s, and bleached again using blue light. The two sets of aliquots were then given different laboratory doses (~ 13 and 127 Gy), and measured using different preheat and cutheat temperatures (preheat at 200°C for 10 s and cutheat to 160°C for one set; preheat at 240°C for 10 s and cutheat to 200°C for the other set) . Fig. S4 presents the dose recovery results. The average ratios between the measured and given doses for the two sets of aliquots were 0.95 ± 0.05 and 0.99 ± 0.05 , respectively.

Fig. S2. Post-IR OSL decay curves for two aliquots of quartz from sample AJH-06: (a) an aliquot showing bright and fast-component dominated luminescence signal and (d) an aliquot showing dim and non-fast-component dominated luminescence signal. The insets show the corresponding growth curves. (b) and (e): The decay curves in (a) and (d) were deconvoluted into three exponentially decaying components (fast, medium and slow) using the first order equation. (c) and (f): Relative contributions of different components for decay curves in (a) and (d) plotted against the stimulation time.

Fig. S3. (a) Histogram showing the IR depletion ratio distribution of post-IR OSL signals for 42 quartz aliquots of loess sample AJH-06; (b) IR depletion ratios of aliquots in (a) plotted against their corresponding Fast ratios; (c) the impacts of varying the IR depletion ratio acceptance threshold value upon the D_e value. The gray belt represents the expected D_e from K-feldspar.

Fig. S4. Dose recovery results for the post-IR OSL protocol listed in Table 1a, using quartz aliquots of sample AJH-06 with Fast ratio >15. (a) Five aliquots measured with a preheat of 200°C for 10 s and a cut to 160°C. The given dose was 12.7 Gy; (b) Five aliquots measured with a preheat of 240°C for 10 s and a cut to 200°C. The given dose was 126.9 Gy. Each data point represents one aliquot.

Appendix E: Some luminescence characteristics of K-feldspar

Representative IRSL and pIRIR decay curves obtained using the low-temperature and high-temperature pIRIR procedures (Table 1 b and c) are given in Fig. S5 a and b, respectively.

Residual doses for different samples, measured with the low-temperature and high-temperature procedures (Table 1b and c) are given in Fig. S6. All doses were measured using aliquots after four hours of solar bleaching. This bleaching time was selected because the IRSL and pIRIR signals showed no further decreases when the solar bleaching time is longer than four hours. Fig. S6 shows that using the low-temperature pIRIR protocol can significantly reduce the residual doses.

(a) The low temperature pIRIR protocol: Sample AJH-02

(b) The high temperature pIRIR protocol: Sample AJH-12

Fig. S5. Representative K-feldspar IRSL and post-IR IRSL (pIRIR) decay curves, measured with (a) the low temperature pIRIR protocol in Table 1b and (b) the high temperature pIRIR protocol in Table 1c, using samples AJH-02 and AJH-12, respectively. The insets show the corresponding representative growth curves.

Fig. S6. Residual doses for K-feldspar IRSL and pIRIR signals for different samples, measured with the low-temperature and high-temperature pIRIR protocols (Table 1b and c), plotted against the IR measurement temperatures. All data were measured using aliquots after four hours of solar bleaching.

Appendix F: A summary of the single aliquot dating results

Table S2 A summary of the single aliquot dating results

Sample	Lithology	Mineral	Grain size (μm)	Dose rate (Gy/ka)	Signal	No. of aliquots ^a	Age model	D _e (Gy)	Age (ka)
AJH-01	loess	Quartz	90-125	3.92±0.16	pIR-OSL	12(55)	CAM	4.27±0.25	1.09±0.10
		KF	90-125	4.41±0.16	IRSL (110)	22	CAM	4.47±0.08	0.89±0.05
		KF	90-125	4.41±0.16	pIRIR (140)	22	CAM	5.29±0.12	0.97±0.05
		KF	90-125	4.41±0.16	pIRIR (170)	22	CAM	5.38±0.21	0.97±0.06
AJH-02	loess	Quartz	90-125	3.73±0.15	pIR-OSL	10 (60)	CAM	13.12±1.13	3.52±0.34
		KF	90-125	4.22±0.17	IRSL (110)	21	CAM	12.30±0.22	2.79±0.12
		KF	90-125	4.22±0.17	pIRIR (140)	21	CAM	14.83±0.44	3.28±0.16
		KF	90-125	4.22±0.17	pIRIR (170)	21	CAM	15.76±0.51	3.48±0.20
AJH-03	fluvial sand	Quartz	150-180	3.44±0.18	pIR-OSL	6 (66)	CAM	18.27±0.81	5.32±0.29
		KF	150-180	4.16±0.20	IRSL (110)	40	CAM/MAM	24.22±0.46/20.48±0.97	5.82±0.29/4.92±0.33
		KF	150-180	4.16±0.20	pIRIR (140)	40	CAM/MAM	44.51±1.41/34.47±1.86	10.70±0.61/8.28±0.59
		KF	150-180	4.16±0.20	pIRIR (170)	40	CAM/MAM	50.72±1.80/39.41±2.09	12.19±0.72/9.47±0.67
AJH-05	fluvial sand	Quartz	150-180	3.52±0.19	pIR-OSL	12 (178)	CAM	14.56±0.68	4.14±0.29
		KF	150-180	4.24±0.20	IRSL (110)	50	CAM/MAM	16.74±0.55/12.38±0.59	3.94±0.22/2.84±0.19
		KF	150-180	4.24±0.20	pIRIR (140)	50	CAM/MAM	24.89±0.83/18.62±0.89	5.86±0.34/4.27±0.29
		KF	150-180	4.24±0.20	pIRIR (170)	50	CAM/MAM	27.37±1.28/18.32±1.04	6.45±0.43/4.13±0.31
AJH-06	loess	Quartz	90-125	3.72±0.15	pIR-OSL	12 (42)	CAM	31.04±1.08	8.34±0.45
		KF	90-125	4.21±0.16	IRSL (110)	31	CAM	32.62±0.90	7.62±0.36
		KF	90-125	4.21±0.16	pIRIR (140)	31	CAM	37.22±0.96	8.60±0.40
		KF	90-125	4.21±0.16	pIRIR (170)	31	CAM	38.27±1.07	8.83±0.42
AJH-09	loess	Quartz	63-90	3.67±0.16	pIR-OSL	6 (30)	CAM	187.82±19.38	51.17±5.71
		KF	63-90	4.03±0.16	IRSL (150)	20	CAM	146.776±2.38	36.40±1.55
		KF	63-90	4.03±0.16	pIRIR (200)	20	CAM	209.21±2.53	51.88±2.14
		KF	63-90	4.03±0.16	pIRIR (250)	20	CAM	224.94±3.81	55.78±2.39
AJH-12	loess	Quartz	90-125	3.74±0.16	pIR-OSL	9 (82)	CAM	185.01±14.13	49.43±4.29
		KF	90-125	4.23±0.16	IRSL (150)	16	CAM	145.26±2.45	34.32±1.43
		KF	90-125	4.23±0.16	pIRIR (200)	16	CAM	201.64±4.84	47.64±2.13
		KF	90-125	4.23±0.16	pIRIR (250)	16	CAM	216.49±5.21	51.15±2.30
AJH-14	loess	Quartz	90-125	3.49±0.15	pIR-OSL	14 (42)	CAM	100.77±5.54	28.91±2.03
		KF	90-125	3.98±0.16	IRSL (150)	17	CAM	72.61±1.19	18.27±0.79
		KF	90-125	3.98±0.16	pIRIR (200)	17	CAM	103.22±0.92	25.97±1.07
		KF	90-125	3.98±0.16	pIRIR (250)	17	CAM	112.53±1.64	28.31±1.21

^a For quartz dating, the bracketed numbers are the numbers of aliquots measured in total. The numbers outside brackets are the numbers of aliquots accepted after applying a Fast ratio acceptance threshold of 15.

Note: For younger K-feldspar samples measured using the low-temperature pIRIR procedure (AJH-01, 02, 03, 05 and 06), the residual doses were corrected using data from Fig. S6.

Appendix G: Single grain dating for quartz from the fluvial sand samples

To further check whether the quartz grains in our fluvial sand samples were well bleached, single grain dating were conducted on quartz grains of the two fluvial sand samples AJH-03 and 05. All single grain measurements were performed using a Risø TL-DA-20 reader equipped with a single-grain attachment (Bøtter-Jensen et al., 2000), with a green laser (532 nm) as light source. A post-IR OSL procedure similar to that in Table 1a, except that the blue LED stimulations (steps 4 and 8) were replaced by green laser stimulations of 1 s at 125°C, has been used in single grain measurements (Table S3).

Examples of single grain post-IR OSL decay curves are shown in Fig. S7. Similar to that of the single aliquot, two types of grains have been observed. One type has stronger luminescence intensity and decays faster (Fig.S7a); while the other type has weaker luminescence signal and a slower decay rate (Fig.S7d). The preliminary rejection criteria follow Jacobs et al. (2006). A Fast ratio acceptance threshold has also been applied to pick up the fast-component dominated grains. Based on component analysis (as in Fig. S7c and f), the average time for the fast component to decay to ~1% for our quartz grains was ~ 0.18 s. Therefore, in this study the Fast ratio for single grain post-IR OSL was defined as:

$$\text{Fast ratio} = (L_{0-0.02s} - \text{BG}) / (L_{0.18-0.20s} - \text{BG})$$

where $L_{0-0.02s}$ and $L_{0.18-0.20s}$ are the integrals of the 0-0.02 s and 0.18-0.20 s of the single grain post-IR OSL signal, respectively. The backgrounds (BG) were evaluated from last 0.1 s of the post-IR OSL decay curves. A Fast ratio acceptance threshold of 15 was applied to the single grain results, similar to that used for single aliquot results.

A total of 9300 and 26400 grains were measured for samples AJH-03 and 05, respectively. However, only 13 and 17 grains passed the rejection criteria for the two samples, respectively. In Fig.S8, single grain D_e of samples AJH-03 and 05 are presented as radial plots. D_e values of these grains distributed randomly around the central value. The overdispersions of them (both 0.17) are similar to the reported overdispersion of well bleached samples (~ 0.20, Olley et al., 2004; Arnold and Roberts, 2009). Table S4 shows that the single grain ages (5.09 ± 0.47 and 4.40 ± 0.29 ka) agreed well with the single aliquot

ages (5.32 ± 0.29 and 4.14 ± 0.29 ka). These results confirm that the quartz grains in our fluvial sand samples were well bleached, and the single aliquot ages of these samples are reliable.

To further confirm the validity of single aliquot dating, the distribution of post-IR OSL signal brightness for individual grains were studied using a cumulative light sum plot (Duller and Murray, 2000), using the 26400 grains of sample AJH-05 (Fig. S9). The luminescence signal brightness varied significantly between grains. The 17 accepted fast-component dominated grains (Fast ratio >15) were all within the brightest 30 grains. These fast-component dominated grains gave rise to $\sim 38\%$ of the total light sum. Given the low proportion of the fast-component dominated grains (0.064%), the probability of the presence of a fast-component dominated grain in a small aliquot of ~ 100 grains is $\sim 6.4\%$. This is similar to the proportion of the fast-component dominated aliquots of $\sim 6.7\%$ in the single aliquot dating results for this sample (12 fast-component dominated aliquots identified from 178 measured aliquots). Taking the average brightness of the fast-component dominated and other grains into account, in a small aliquot containing one fast-component dominated grain (i.e. a fast-component dominated aliquot), the fast-component dominated grain will yields $\sim 91\%$ of the light sum of the whole aliquot on average. Therefore, applying the Fast ratio selection to the single aliquot results is nearly equal to applying the Fast ratio selection to the single grain results, in respect of selecting grains with greater dominance of the fast component.

Table S3 Single-grain dating procedures for quartz

Step	Treatment	Observed
1	Give regenerative dose, D_i	
2	Preheat at 200°C for 10 s	
3	IR bleaching at 125°C for 100 s	
4	Green laser stimulation at 125°C for 1 s	L_x
5	Give test dose, D_t	
6	Cutheat to 160°C	
7	IR bleaching at 125°C for 100 s	
8	Green laser stimulation at 125°C for 1 s	T_x
9	Blue light bleaching at 220°C for 100 s	
10	Return to step 1	

Note: For the first cycle natural signals were recorded. The later five regeneration cycles include a zero dose $D_4=0$ and two repeated regenerative doses $D_1=D_5$.

Table S4 A summary of quartz single grain dating results for the fluvial sand samples

Sample	Grain size (μm)	Dose rate (Gy/ka) ^a	No. of grains ^b	D_e (Gy) ^c	SG Age (ka)	SA age (ka) ^d
AJH-03	180-250	3.39 ± 0.18	13 (9300)	17.29 ± 1.31	5.09 ± 0.47	5.32 ± 0.29
AJH-05	180-250	3.48 ± 0.18	17 (23400)	15.31 ± 0.60	4.40 ± 0.29	4.14 ± 0.29

^a See details in Table S1.

^b The numbers insides brackets represent the number of grains measured in total. The numbers outsides brackets represent the number of grains accepted, after selection using the criteria of Jacobs et al. (2006) and a Fast ratio acceptance threshold of 15.

^c Calculated using the central age model (Galbraith et al., 1999).

^d See details in Table S2.

Fig. S7. Post-IR OSL decay curves for two quartz grains of sample AJH-05: (a) one grain showing a bright OSL signal dominated by the fast component and (d) one grain showing a dim OSL signal dominated by non-fast components. (b) and (e): The decay curves in (a) and (d) deconvoluted into three exponentially decaying components (fast, medium and slow) using the first order equation. (c) and (f): Relative contributions of different components for decay curves in (a) and (d) plotted against stimulation time.

Fig. S8. Single grain D_e distributions for quartz from sample (a) AJH-03 and (b) AJH-05, shown as radial plots. The central D_e value was obtained using the central age model (Galbraith et al., 1999).

Fig. S9. Cumulative light sum plot for grains of sample AJH-05. The percentage of the total light sum is plotted as a function of the percentage of all the grains (26400) measured. Note the log-scale in the x-axis. The fast-component dominated grains accepted (Fast ratio > 15) are marked as red open blocks.

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