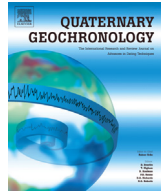




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## Optical dating of aeolian and fluvial sediments in north Tian Shan range, China: Luminescence characteristics and methodological aspects

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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.

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### 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

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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  $\mu\text{m}$ ) 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  $^{90}\text{Sr}/^{90}\text{Y}$   $\beta$  sources. Luminescence was detected by EMI 9235QA photomultiplier tubes (PMT). Blue (470 nm) and IR (870 $\Delta$ 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 in relevant experiments. Details about annual doses are summarized in Table S1.

## 3. Luminescence characteristics and performance of the procedures

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  $1.0 \pm 0.1$ . 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

**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.

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

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

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

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