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Quartz OSL and K-feldspar post-IR IRSL dating of loess in the Huangshui river valley, northeastern Tibetan plateau

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A B S T R A C T

The northeastern Tibetan Plateau (NETP) is located in the climatically sensitive semiarid zone between the regions controlled by the East Asian monsoon and the Westerlies and loess deposits there may preserve a record of regional paleoenvironmental change. Establishing a robust loess chronology is critical for interpreting and correlating environmental records. In this study, quartz optical stimulated luminescence (OSL) and K-feldspar post-IR infrared (IR) stimulated luminescence (pIR-IRSL) dating methods have been used to date the Ledu loess section in the Huangshui river valley, on the NETP. In terms of quartz OSL dating, the results from both the 63–90 μm and 38–63 μm quartz fractions are consistent within errors. The reliability of the 63–90 μm K-feldspar pIRIR dating was confirmed by internal check using preheat plateau, dose recovery, anomalous fading, and residual dose tests. The results suggest that the K-feldspar pIRIR signals at stimulation temperatures of 170 and 225 °C were well bleached before deposition of Ledu loess. Comparison between quartz OSL and K-feldspar pIRIR dating indicates that quartz ages older than 50 ka (~150 Gy) may be underestimated. In establishing the chronological framework for the study section, we selected quartz OSL results for ages < 50 ka and the K-feldspar $pIRIR_{170}$ and $pIRIR_{225}$ results for ages > 50 ka. The results demonstrate that aeolian dust accumulated continuously between 67 and 25 ka, and there were two gaps in deposition, between 25 and 2 ka and from 80 to 67 ka.

1. Introduction

The northeastern part of the Tibetan Plateau (TP) is the transition zone between the TP and the Chinese Loess Plateau (CLP); it lies in the monsoon marginal zone and is influenced by the westerlies, East Asian summer monsoon, and plateau monsoon (Liu et al., 2015; Lu et al., 2004). The area is regarded as an ideal region for research on uplift process of the TP, environmental changes, and evolution of the Asian monsoon (An et al., 2012; Lu et al., 2004). Research based on geomorphologic, stratigraphic, geochemical, and zircon U-Pb chronological analysis suggests the northern and northeastern TP forms an important source region for silts deposited in the CLP (Bird et al., 2015; Bowler et al., 1987; Kapp et al., 2011; Li et al., 2013; Licht et al., 2016; Nie et al., 2015; Pullen et al., 2011; Stevens et al., 2013; Liu et al., 2017). During recent decades, the northeastern TP has become a key area for the reconstruction of the palaeoclimatic evolution of central Asia (Stauch et al., 2016). Loess deposits, which are widespread on the NETP, are highly sensitive to shifts in the Asian summer and winter monsoon and/or northern hemisphere westerly circulation (Liu & Ding, 1998; Lu et al. 2004). Loess deposits also preserve important information on Quaternary climate change and atmospheric dust flux, however, a robust chronological framework is critical for retrieving this environmental information (Roberts, 2008; Timar-Gabor et al., 2011).

OSL dating of quartz using the single-aliquot regenerative-dose (SAR) dating protocol is now being widely applied to late Quaternary sediments (Murray and Olley, 2002; Wintle and Murray, 2006). Over the past decade, a considerable amount of research has been undertaken on luminescence dating of Chinese loess-paleosol sequences,

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mainly based on quartz OSL (Buylaert et al., 2007, 2008; Lai, 2010; Stevens et al., 2006, 2008), and recently this has been applied to the NETP (Liu and Lai, 2013; Liu et al., 2015, 2016; Yu and Lai, 2014). Buylaert et al. (2008) conducted low stratigraphic resolution quartz OSL dating on loess from the NETP, and reported that the quartz ages saturated at ~50 ka. In an analysis of 30 high resolution quartz OSL ages, Wang et al. (2015a) revealed episodic accumulation of loess from 30 to 0.2 ka. However, quartz OSL dating is generally limited to a saturation dose of 120–150 Gy, for loess deposits with a typical dose rate 3–4 Gy/ka, which restricts its use to the last 40–50 ka (Buylaert et al., 2007; Chapot et al. 2012; Li et al., 2016; Roberts, 2008; Timar-Gabor and Wintle, 2013; Yi et al., 2015, 2016). Feldspar luminescence provides an alternative sediment dating method that has a higher saturation dose compared to quartz (Huntley and Lamothe, 2001). However, most feldspar suffers anomalous fading, which results in underestimation of the true age (Spooner, 1994; Wintle, 1973). Recently, two new protocols have been developed, post-IR IRSL (pIRIR) and multi-elevated-temperature post-IR IRSL (MET-pIRIR), to obtain lower fading rates and improve the reliability of feldspar luminescence dating (Buylaert et al., 2009, 2012; Li and Li, 2011, 2012; Thiel et al., 2011; Thomsen et al., 2008).

In this study, we used quartz OSL and K-feldspar pIRIR methods to date Ledu loess in the Huangshui river valley, NETP. We first explored the characteristics of quartz SAR OSL, then investigated the luminescence characteristics of the elevated temperature IRSL signals in a SAR post-IR IRSL protocol. The reliability of K-feldspar pIRIR age estimates was confirmed by internal checks of luminescence characteristics, a fading test, and by comparison of the quartz and K-feldspar ages. Finally, we used the luminescence ages to explore the continuity of loess deposition in the Huangshui river valley, to test recent suggestions of episodic loess deposition on the NETP (Liu et al., 2017; Wang et al., 2015a).

2. Study area and sampling

The NETP is situated at the junction of areas controlled by the Asian summer monsoon and Westerlies (An et al., 2012). The annual mean precipitation and temperature are ~340 mm and 7 °C, respectively. Loess deposits are widely distributed on the NETP, and are generally coarser and less compacted than those in the central CLP (Lu et al., 2004, 2011; Vriend and Prins, 2005).

The Huangshui river valley contains loess accumulations that are commonly mantled on a series of river terraces within the Xining, Ledu, and Minhe basins (Fig. 1). For this study, we sampled an approximately 23-m thick loess section, which overlies the third terrace of the Huangshui river in the Ledu basin (termed the LD section). The top 10 m of the section comprises typical loess, with a median grain size of around 35 μm, overlying 13 m of light yellow loess alternating with thin reddish-yellow silt layers (Figs. 2 and 3). The loess grain size distribution curves show a typical distribution, with a modal size of ~55 μm (Fig. 4), which is similar to the nearby loess section reported by Wang et al. (2015a). Paleosols are weakly developed and are difficult to identify in the field. Thirty-nine luminescence samples were collected at 50-cm intervals from the freshly excavated profile using light-tight steel cylinders (diameter 5 cm, length 20 cm) (Fig. 2).

3. Materials and methods

All laboratory pretreatments, sample preparation, and luminescence measurements were conducted under subdued red light in the luminescence laboratory at the Qinghai Institute of Salt Lakes, Chinese Academy of Science. Three cm of material at each end of sample tubes was removed and reserved for environment dose rate measurement. The unexposed part in the middle of the tube was used for equivalent dose (De) determination. All samples were treated with 10% HCl and 30% H_2O_2 to remove carbonates and organic matter. Medium (38–63 μm) and coarse (63–90 μm) grain size fractions were obtained by wet sieving. Heavy liquids with densities of 2.62, 2.75, and 2.58 g/cm^3 were used to separate the quartz and K-feldspar fractions of each sample. The 38–63 μm quartz fraction was then treated with silica saturated fluorosilicic acid (H_2SiF_6) for about two weeks, while the 63–90 μm quartz fraction was etched with 40% HF for 60 min, and both were then treated with 10% HCl to remove any fluorides. The purity of the extracted quartz was checked by IR stimulation; where there were obvious IR signals, quartz grains were re-etched with H_2SiF_6 or HF to avoid age underestimation (Duller, 2003; Lai and Brückner, 2008; Roberts, 2007). The K-feldspar 63–90 μm fraction of separates were not HF etched as HF etching on K-feldspar tends to cause deep pitting and to preferentially attack the cleavage planes rather than removing a uniform shell from the grains (Duller, 1994; Long et al., 2014).

Quartz and K-feldspar grains were then mounted as a mono-layer on the central part (~0.7 cm diameter) of stainless steel discs (~0.97 cm diameter) using silicone oil. The OSL signal was measured using an automated Risø TL/OSL-DA-20 reader. Laboratory irradiation was carried out using $^{90}Sr/Y^{90}$ sources mounted within the reader, with a dose rate of 0.089 Gy/s. The OSL signal was obtained after passage through a U-340 filter and the IRSL signal was detected using a photomultiplier tube with the IRSL passing through BG-39 and coring-759 filters.

The environmental dose rate was calculated from measurements of radioactive element concentrations of the surrounding sediment with a small contribution from cosmic rays. For all samples, U and Th concentration and K content was determined using neutron activation analysis at the Chinese Atomic Energy Institute. Calculation of the cosmic dose rate was based on Prescott and Hutton (1994). The measured water content of the loess samples from the section varied between 1 and 6%, however, considering that the section has been exposed by sand excavation for a long time, this is likely to be an underestimate. Based on data from previous loess studies (ChongYi et al., 2012; Lai, 2010; Li et al., 2016), a water content of 10 ± 5% was used in all dose rate calculations. The a-value for 38–63 μm was taken as 0.035 ± 0.003 (Lai et al., 2008). For K-feldspar dose rates, a K concentration of 12.5 ± 0.5% and Rb concentration of 400 ± 100 ppm was assumed (Huntley and Baril, 1997).

4. Luminescence characteristics

4.1. Quartz OSL

A combination of SAR protocol (Murray and Wintle, 2000) and standardized growth curves (SGCs) (Lai, 2006; Lai et al., 2007; Roberts and Duller, 2004), i.e., the SAR-SGC method, was used to determine 39 medium-grained (38–63 μm) and 8 coarse-grained (63–90 μm) quartz D_e. For each sample, 6–8 aliquots were measured by SAR to build a SGC, and then 12–24 additional aliquots were measured by SGC. The final D_e for each sample was derived from the mean of the SAR D_e and the SGC D_e. The net quartz OSL signal was calculated using the initial 0.64 s integral of the OSL decay curve minus the last 8 s integral.

The suitability of the SAR procedure for D_e determination was tested using a dose recovery test (Murray and Wintle, 2003). The dose recovery test was performed on two samples, from the upper (LD-2, 38–63 μm) and a lower (LD-30, 38–63 μm) section. Twenty-four natural aliquots were stimulated twice by blue-light stimulation at 130 °C for 60 s. The bleached aliquots were then given a laboratory dose of 89 Gy (LD-2) and 160 Gy (LD-30), close to their natural D_e. Preheat temperatures ranged from 220 to 300 °C at 20 °C intervals for 10 s, using a heating rate of 5 °C/s, and the cut-heat temperature was fixed at 220 °C for all measurements. At each preheat temperature, four aliquots were measured for calculation of mean values. The ratio of measured to given D_e, and recycling and recuperations ratios for samples LD-2 and LD-30 are plotted in Fig. 5. For sample LD-2, a plateau was observed for temperatures from 220 to 280 °C. The recycling ratios for different