Journal of Asian Earth Sciences 91 (2014) 154–162

Contents lists available at ScienceDirect

Journal of Asian Earth Sciences

journal homepage: www.elsevier.com/locate/jseaes

Optically stimulated luminescence dating of sand-dune formed within the Little Ice Age

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ARTICLE INFO

Article history:
Received 6 March 2014
Received in revised form 15 May 2014
Accepted 16 May 2014
Available online 24 May 2014

Keywords:
Sand dunes
OSL dating
SAR
Dim and young quartz
Little Ice Age

ABSTRACT

Optically Stimulated Luminescence (OSL) dating has been intensively used to date the late Quaternary deposits. The Single-Aliquot Regenerative-dose (SAR) protocol was applied to measure coarse quartz grains extracted from Dali sand dunes, central China. It was found that this popular method could not accurately date sand dunes activities that recently happened. This is due to the low OSL sensitivity of quartz grains, leading to a failure in OSL dating that has also been met in other sediments before. To overcome this limitation, quartz grains were heated to 500 °C to increase the OSL output for one magnitude. Sensitivity changes could also be corrected by the following test dose OSL responses and has no influence on OSL ages. Thus it is suggested to carry out the SAR protocol for dim and young quartz OSL samples with additional annealing step (e.g. 500 °C) after the measurements of natural cycle. The resultant OSL ages proved that the last sand-dune activities happened during the Little Ice Age (~400 years ago), which was further supported by independent age control (~1600 AD) from historical documents.

1. Introduction

Optically Stimulated Luminescence (OSL) dating has been used intensively to construct the chronology of late Quaternary sediments for geology, climate change and archeological studies, following Murray and Wintle's (2000, 2003) demonstration of the robustness of a Single-Aliquot Regenerative-dose (SAR) protocol. Murray and Olley (2002) validated the SAR protocol by obtaining about 50 OSL ages from sediments of aeolian and fluvial origins and comparing them with independent ages. Therefore, OSL dating using the SAR protocol on quartz has been widely accepted by the Quaternary community (Wintle, 2008), e.g. Li et al. (2002, 2007) and Zhao et al. (2012) have successfully reconstructed the Holocene sand dunes activities in Chinese deserts.

In the past 15 years, the SAR protocol has been applied to young sediments, i.e. with OSL ages of less than 1000 years (e.g. Olley et al., 1998; Ballarini et al., 2003; Rodnight et al., 2005; Madsen et al., 2005, 2007; Ballarini et al., 2007a,b; Argyilan et al., 2010), more recently Kunz et al. (2014) used different age models to estimate the reliability of OSL dating in young fluvial sediments. This has provided new insights into the past millennial climate changes where other numeric dating methods are limited in their application. This period is of great importance, because human activities interact with climate changes. Madsen and Murray (2009) reviewed OSL dating of young sediments and pointed out that many dating studies had failed, and had not been published, because the OSL signals were too weak. Weak signals, with a small number of photon counts giving a low signal/noise ratio, give rise to two problems for equivalent dose determination. Firstly, the natural OSL signal and its corresponding OSL response to a test dose used to monitor the OSL sensitivity have large statistical uncertainties. Secondly, there is a similar effect on the OSL measurements used in the SAR protocol to construct the dose response curve.

Recently, we applied OSL dating to young sand dunes in central China where the quartz OSL signal is dim. Here we report our attempts to overcome the problems that result from the low signal/noise ratio when using the SAR protocol, and then date the last sand dunes activities in Dali field, central China.

2. Study area

The Dali dune field lies in the region between the Luo River and the Wei River, first-order tributaries of the Yellow River, and has the length and width of about 40 and 12 km, respectively (Fig. 1). The shapes of most of sand dunes are barchans and barchan ridge, similar to those of the sand field (e.g. Mu Us Desert) in North China. It is suspected that the Dali dune field is a product of the valley topography and its source areas are likely to be the floodplains of the Luo and Wei Rivers. Dali sandy land is located

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http://dx.doi.org/10.1016/j.jseaes.2014.05.012
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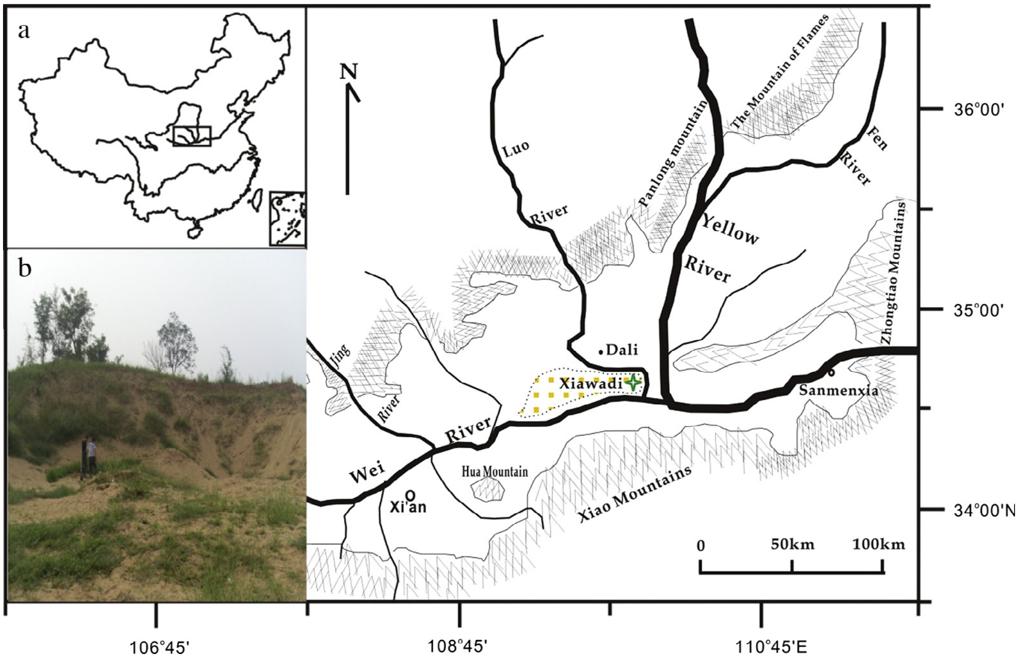

Fig. 1. Map showing Dali dune field (yellow dotted area) and Xiawadi section (green asterisk), located between the Luo River and the Wei River. Inset 1a is a map of China with the region of the main map being shaded; inset 1b is a photo of the sand dunes at Dali sand field, central China. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

at the southernmost edge of the Chinese Loess Plateau, currently under a non-arid climatic regime that is not favor for development of sand dunes field. Therefore it is needed to reconstruct the climate changes in this region and further to uncover the cause of sand dunes.

3. Samples, pretreatment and instruments

Eight samples were selected from the Xiawadi section (34°37.59′N, 109°47.67′E) in the Dali dune field in Fig. 1. Two samples (IEE3534 and IEE3565) obtained from the upper and lower part of the section, respectively, were intensively studied to develop the most appropriate SAR protocol for OSL dating. The samples were processed under subdued red light conditions to obtain the 90–125 μm fraction of quartz grains. The sample (approximately 100 g) was first treated with 30% H_2O_2 and 30% HCl to remove organic materials and carbonates, respectively, and then sieved to obtain the 90–125 μm fraction. HF (40%) was added to dissolve feldspar and etch the outer surface of the quartz grains. Finally, the samples were treated with HCl (10%) to remove any fluoride precipitates created during the HF treatment, and no density separation was applied. The grains were scattered on a 1 cm diameter stainless steel disk on which a thin film of oil had been spread. About 4 mg of grains were delivered to each disk in order to maximize the signal, resulting in about 6500 grains per aliquot covering an area of about 9 mm diameter of the disk (Duller, 2008).

All luminescence measurements were carried out using a Daybreak 2200 automated OSL reader equipped with combined blue (470 ± 15 nm) and infrared (IR) (880 ± 80 nm) LED units, and a $^{90}Sr/^{90}Y$ beta source for irradiation. All IR and blue stimulations were performed at 125 °C for 200 s with a nominal stimulation power of ~45 mW/cm^2 for both IR and blue light, and detected using an EMI9235QA photomultiplier tube coupled in front with two 3 mm U-340 (290–370 nm) glass filters. For D_e estimation, the OSL integral from the first 5 s (minus the last 5 s) of the 200 s decay curve was used. The instrumental uncertainty in each OSL measurement is 3% and this was included in the error analysis following Duller (2007).

Table 1
Experimental procedure and parameters.

Step	Treatment	Observed
1	Bleach by SOL 2 for 30 min	PH_x
2	Annealing to x °C for 10 s	
3	Give regeneration dose, 50 Gy	
4	Preheat at 240 °C for 10 s	
5	IR stimulation at 125 °C for 200 s	
6	Blue stimulation at 125 °C for 200 s	L_i
7	Give test dose, 10 Gy	
8	Preheat at 220 °C for 10 s	
9	IR stimulation at 125 °C for 200 s	
10	Blue stimulation at 125 °C for 200 s	T_i
11	Annealing to 500 °C for 10 s	
12	Give test dose, 10 Gy	
13	Preheat at 220 °C for 10 s	
14	IR stimulation at 125 °C for 200 s	
15	Blue stimulation at 125 °C for 200 s	$500T_i$

The purity of the isolated quartz was checked by infrared (IR) stimulation of the purified quartz grains. It is found that the IRSL signal could not be distinguished from the background signal when measured on an empty aliquot. This indicated that the chemically isolated quartz was pure and suitable for OSL dating.

Based on Duller's (2003), the purity of the quartz was also checked by investigating whether the OSL signal could be reduced by IR exposure on single grains. A portion of the sample IEE3534 was annealed to 500 °C and the OSL signal from 10 aliquots was measured after administering an artificial dose of 1 Gy. Afterwards the same aliquots were irradiated again with a dose of 1 Gy and exposed to IR stimulation at 125 °C for 200 s before measuring the OSL. The average IR depletion ratio was 0.98 with a standard deviation of 0.03 for the 10 aliquots that were checked. It was necessary to make the IR depletion test on annealed aliquots to obtain sufficient OSL sensitivity for reproducibility, although the quartz OSL sensitivity increased by more than one order of magnitude.

In this study, the concentrations of U and Th, and K were measured by ICP-MS and ICP-AES, and their values are shown in Table 2, with an error of 3% for U, Th and 1% for K. The weights of wet