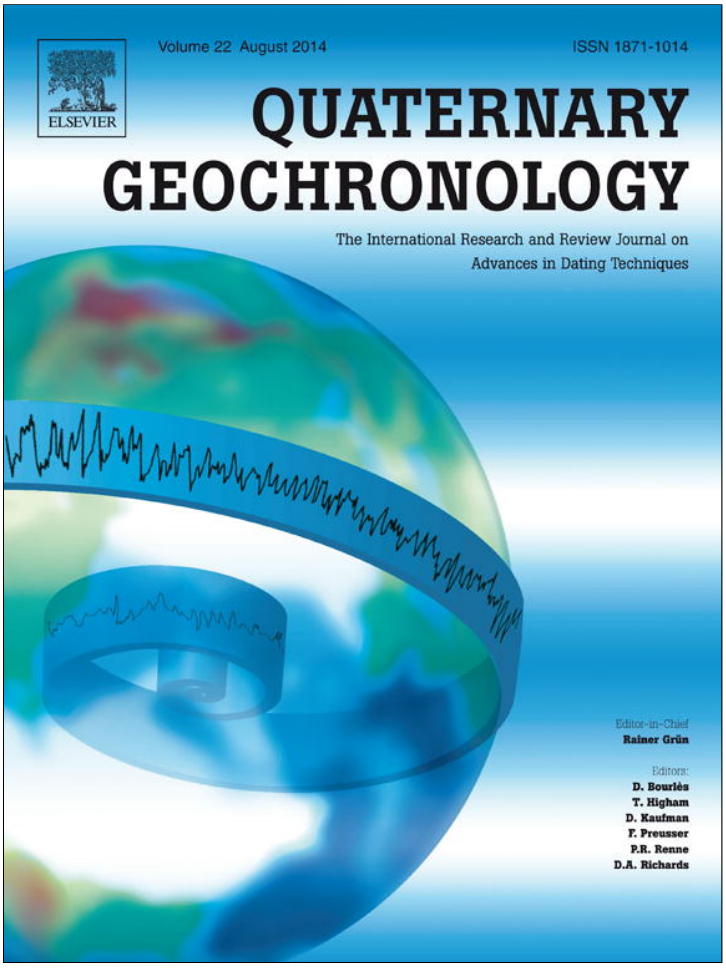
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Research paper

Investigating the optically stimulated luminescence dose saturation behavior for quartz grains from dune sands in China

Zhijun Gong [a,*], Jimin Sun [a], Tongyan Lü [b], Zhonghua Tian [a]

[a] Key laboratory of Cenozoic Geology and Environment, Institute of Geology and Geophysics, Chinese Academy of Science, P.O. Box 9825, Beijing 100029, China
[b] Institute of Geomechanics, Chinese Academy of Geological Sciences, Beijing 100081, China

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ABSTRACT

The optically stimulated luminescence (OSL) signals from quartz have been widely used to estimate the equivalent dose (D_e) of environment radiation after the deposition of mineral grains. However, the usage of quartz is often limited due to the lower saturation behavior compared with feldspar. Saturation limits among quartz (defining the upper dating range) vary significantly. It is important to better understand the reason for various dose saturation behaviors of the quartz OSL signals. In this study, coarse quartz grains were extracted from the Taklimakan Desert and the Hunshandake sandy land in north China and the dose saturation behavior of quartz OSL signals were studied. Our results suggest that the quartz grains produce very different aliquot-specific dose response curves, showing the significant variability in dose saturation characteristics for OSL signals. Laboratory dosing, optical bleaching and heating experiments were designed to simulate their effects on the dose saturation behavior for the quartz OSL. The results demonstrate that cycles of dosing and optical bleaching have insignificant impact on the OSL dose growth curves, while the heating to high temperature (above 400 °C) can significantly change the dose saturation characteristics for the quartz OSL. Such results suggest that the different heating history of quartz might be an important factor for the variability in dose saturation characteristics for OSL signals. Additionally, the quartz grains from the Hunshandake sandy land exhibit lower dose saturation level for OSL signals, compared with that from the Taklimakan Desert. This can be explained that the quartz grains from Hunshandake sandy land are mainly of igneous origin, while the quartz grains from Taklimakan Desert are mainly of metamorphic origin.

1. Introduction

Natural quartz serves as an important dosimeter of ionising radiation in luminescence dating of sediments (Aitken, 1998; Wintle and Murray, 2006; Preusser et al., 2009; Rhodes, 2011). The optically stimulated luminescence (OSL) signals from quartz grains have been widely applied to estimate deposition ages of late Quaternary sediments by using the single-aliquot regenerative-dose (SAR) procedure (Murray and Wintle, 2000). In such approach, the measured sensitivity-corrected natural OSL signal is compared with equivalent sensitivity-corrected signals regenerated by laboratory irradiation of the same aliquot of quartz. Comparison of the natural OSL intensity with the laboratory generated dose response curves (DRCs) allows the determination of the equivalent dose (D_e) of the quartz grains. If a single type of trap is assumed to be responsible for the OSL signal, the DRCs of sedimentary quartz can be described by a single saturating exponential function in the form of $I = I_{max}(1 - \exp(-D/D_0))$, where I is the OSL intensity due to dose D (Gy), I_{max} is the saturation luminescence intensity and D_0 (Gy) is the dose characteristic level of the dose response curve (Aitken, 1998). In routine luminescence dating of quartz samples, it is often found that, compared with a single saturating exponential function, the single exponential plus linear function can give much better fitting to the OSL dose response curves, when the regeneration dose reaches beyond 100–200 Gy (e.g. Roberts and Duller, 2004; Lai, 2006; Lai et al., 2008; Pawley et al., 2008; Lowick et al., 2010). This is because there are multiple sources responsible for the OSL production of quartz instead of the single trap source. In order to obtain reliable estimation of natural dose for the quartz grains, it is prudent to ensure that the D_e was less than $2D_0$ (Wintle and Murray, 2006).

The OSL signals from natural quartz grains comprise several physically distinct components, e.g. fast, medium and slow components (Bailey et al., 1997; Jain et al., 2003; Li and Li, 2006). For the OSL signals dominated by the fast component, a variety of D_0 values

* Corresponding author.
E-mail address: gongzj@mail.iggcas.ac.cn (Z. Gong).

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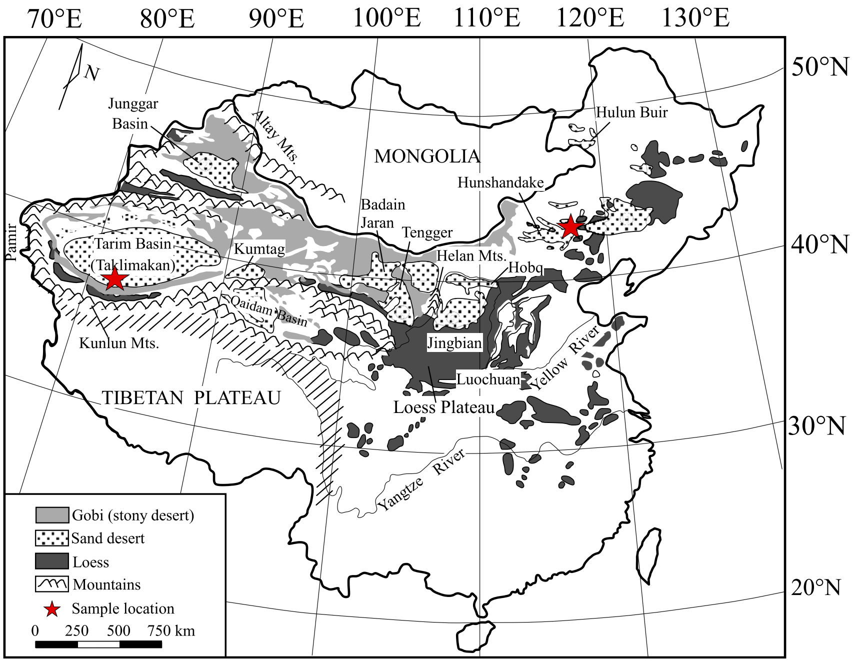

Fig. 1. Map showing mountains, gobi (stony desert), sand desert, and loess distributions as well as the sampling sites in China (modified from Sun and Zhu, 2010).

were reported for the quartz samples in different regions and sedimentary environments, e.g. 55 Gy (Roberts and Duller, 2004), 69 Gy (Singarayer and Bailey, 2004), 75 Gy (Banerjee et al., 2003), 90 Gy (Huntley et al., 1996), 97 Gy (Jain et al., 2005) and 190 Gy (Singarayer and Bailey, 2003). Even for quartz grains of a same sample from a given site, significant variations in dose saturation characteristics were also identified and the values (D_0) were reported from 40 Gy to 600 Gy (Duller et al., 2000). An implication from these studies is that the construction of individual DRCs for quartz OSL signals are necessary for individual aliquot or grain in the estimation of D_e, due to the potential significant variability in the saturation behavior among dose growth curve characteristics. In contrast to above results, some studies on quartz OSL demonstrated that similar DRCs or values (D_0) can be obtained in the laboratory for samples from the same site or geographical area. This idea has been applied in the use of standardized or common growth curves for determining D_e for quartz samples within the same study area (Roberts and Duller, 2004; Lai, 2006; Lai et al., 2007). The advantage of such method is the quick measurement of D_e. However, the assumption of the very similar DRCs or values (D_0) for quartz OSL signals among individual quartz grains must be evaluated.

In this study, coarse quartz grains of aeolian sand samples were extracted and their dose saturation behaviors for the quartz OSL signals were studied. This paper aims to address the following topics: (1) examining the dose saturation behaviors of quartz OSL from the aeolian sands, to see if the quartz grains exhibit different OSL dose saturation characteristics. (2) simulating the effects of irradiation, optical bleaching and heating on the dose saturation behaviors for the quartz OSL signals, because the sedimentary quartz grains might experience different irradiation, bleaching and heating history, which might make their OSL dose saturation characteristics different.

2. Materials and methods

2.1. Geological setting

Modern dune sand samples (TK-1 and HS-1) were collected from the Taklimakan Desert and the Hunshandake sandy land in China, respectively (Fig. 1). The Taklimakan Desert is the largest sand sea in China, occupying an area of 337,000 km^2 (Sun and Liu, 2006). It situates in the Tarim Basin surrounded by the Tianshan mountains to the north, the Kunlun mountains to the south and the Pamir Plateau to the west. A great mount of metamorphic rocks distribute in the orogenic belts surrounding the Taklimakan Desert (Fig. 2a). Previous studies have suggested that quartz grains from the Taklimakan Desert produce relatively dim OSL signals (e.g., Zheng et al., 2009; Lü and Sun, 2011), as the most of quartz grains are metamorphic origin (Ma, 2002; Yang et al., 2008). The Hunshandake sandy land occupies an area of 21,400 km^2 in northeastern China (Fig. 1) (Zhu et al., 1980). Mesozoic (mainly Jurassic and Cretaceous) and Paleozoic volcanic rocks are widespread around the sandy land (Ma, 2002) (Fig. 2b). By investigating oxygen isotopic compositions of quartz, Yang et al. (2008) proved that the sands in the Hunshandake sandy land are mainly of igneous origin. Thus, the quartz grains in the Hunshandake sandy land can produce very bright luminescence signals (Zheng et al., 2009; Lü and Sun, 2011), as they had been subjected to the heating to high temperature during the formation of the host rock or later volcanic activities (e.g. felsic magmas can erupt at temperatures as high as 650–750 °C or even above).

2.2. Sample preparation

The pre-treatments and luminescence measurements for the quartz samples were completed in the Luminescence Dating Laboratory in the Institute of Geology and Geophysics, Chinese Academy of Science. The bulk samples were treated with 10% hydrochloric acid (HCl) and 10% hydrogen peroxide (H_2O_2) to remove carbonates and organic materials, respectively. Grains between 90 and 125 μm were selected by dry sieving. Quartz grains were separated using sodium polytungstate heavy liquid with density between 2.62 and 2.75 g/cm^3. The quartz grains were etched with 40% hydrofluoric acid (HF) for 80 min to remove feldspar. HCl (10%) was used again to dissolve any residual fluorides after etching before final rinsing and drying. The etched grains were mounted as a monolayer on aluminum discs of 10 mm diameter using silicone oil as an adhesive. Grains covered the

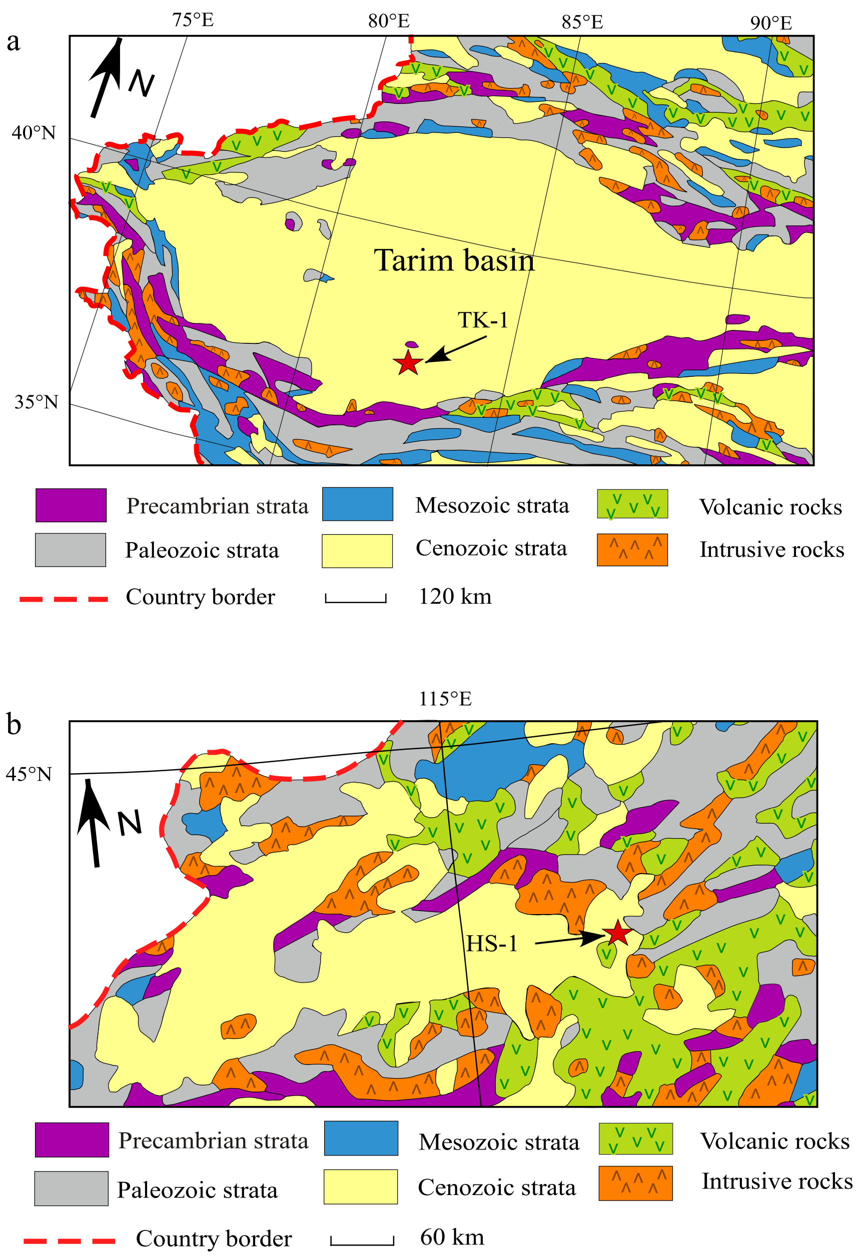

Fig. 2. Geological map showing the strata of the orogenic belts surrounding the Taklimakan Desert (a) and the Hunshandake sandy land (b) (modified after 1:1000000 geological map of China). Red stars indicate sample locations. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

central ~3 mm diameter portion of each disc, corresponding to several hundred grains per aliquot. The purity of quartz grains was tested by measuring the infrared stimulated luminescence (IRSL) and 110 °C thermal luminescence (TL) peak (Li et al., 2002). Any response to IR stimulation led to the rejection of the aliquots.

2.3. Laboratory equipment

The OSL measurements were performed using automated TL/OSL reader (Risø-TL/OSL-15). The reader is equipped with excitation units of blue light emitting diodes (LEDs) ($\Delta = 470 \pm 30$ nm) and a solid state infrared (IR) laser ($\Delta = 830 \pm 10$ nm). The blue LEDs deliver up to 50 mW/cm^2 at the sample. 90% of their full powers were used for stimulation in this study. The quartz OSL signals were detected through a Hoya U-340 filter, which allows a transmission from 290 nm to 370 nm with a peak at ~340 nm (Aitken, 1998). Irradiation was carried out using a $^{90}Sr/^{90}Y$ beta source built into the reader, which delivers the dose rate of 0.0854 Gy/s to the aliquots of quartz.

3. Experimental details and results

3.1. SAR dose response curves

Five aliquots of quartz of TK-1 were at first bleached with blue light at 280 °C for 100 s to remove natural OSL signals. The aliquots were then given a sequence of irradiation doses in the laboratory to construct the SAR DRCs (Table 1) (Murray and Wintle, 2000). The OSL measurements (L_x) for the aliquots were performed with blue light at 125 °C for 40 s with regenerative dose of 0, 17.1, 42.7, 85.4, 128.1, 170.9, 256.3, 341.7 and 512.6 Gy, each followed by a measurement of signal (T_x) induced by a test dose of 21.4 Gy. Preheating at 260 °C for 10 s and cut-heat at 220 °C were applied to the aliquots before OSL stimulation for the regenerative dose and test dose OSL measurement, respectively. Additionally, a repeated dose of 85.4 Gy was added to the end of sequence for checking the reproducibility of the sensitivity correction (i.e. recycling ratio). The resulting OSL signals (L_x and T_x) were calculated by integrating counts in the initial 1 s of the OSL decay curve after subtraction using the averaged signal of the last 2 s. For the five aliquots, their recycling ratios are all with 10% of unity.

The representative SAR DRCs of TK-1 are shown in Fig. 3. It is found that the growth curves for the quartz OSL can be fitted by a single saturating exponential plus a linear component (Lai, 2010; Lowick et al., 2010). The significant variability in the dose saturation characteristics are observed among the aliquots of quartz of TK-1. Values (D_0), the dose characteristic level of the dose response curves, are from ∼122 Gy to ∼254 Gy for the five aliquots. Our results show that the coarse quartz grains from the Taklimakan Desert in western China exhibits very different dose saturation behaviors for OSL signals among aliquots.

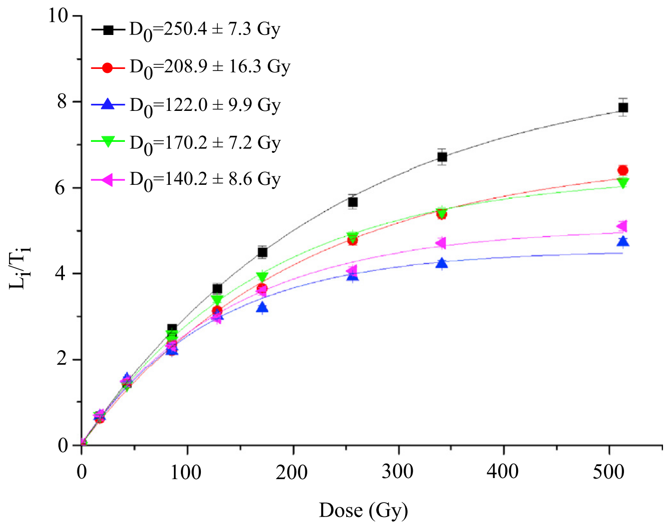

Fig. 3. Coarse-grained quartz SAR dose response curves for the sample TK-1. The five types of symbols represent the results from five different aliquots.

3.2. Simulating the effects of irradiation, optical bleaching and heating on the dose saturation behavior for quartz OSL signals

Previous studies suggested the cycles of irradiation, optical bleaching and heating that occur in the SAR OSL measurements as well as in the natural earth surface processes could affect the sensitivity of quartz samples (Moska and Murray, 2006; Pietsch et al., 2008; Zheng et al., 2009; Lü and Sun, 2011). However, their effects on the dose saturation characteristics for quartz OSL are not well understood. In this study, laboratory irradiation, optical bleaching and heating experiments were designed to simulate their effects on the dose saturation behavior for the quartz OSL.

3.2.1. Dosing/bleaching effect

The effects of repeated dosing/bleaching on the quartz OSL dose growth characteristics were tested by using the experiment procedure in Table 2. An aliquot of quartz of TK-1 was at first given a sequence of irradiation doses in the laboratory to construct an aliquot-specific dose growth curve (Step 1). The aliquot was then operated with twenty cycles of dosing/bleaching (Step 2) before the construction of next dose growth curve (Step 4). The dose level is 21.4 Gy and illumination condition was using blue light at 125 °C for 40 s. Such treatments were repeated to regenerate six dose response curves for the same aliquot (Fig. 4), while twenty additional cycles of laboratory dosing/bleaching were performed among each construction of dose response curve. The results show that the aliquot of quartz produces very similar DRCs for OSL signals, even though it is subjected to many cycles of irradiation and illumination. The values (D_0) of the six DRCs are all at ∼137.9 Gy (Fig. 4). The results suggest that repeated dosing and optical bleaching are unlikely to induce significant change in the dose growth characteristic for the quartz OSL.

To further demonstrate the above results, the same experiment was operated for four aliquots of quartz of TK-1. As the same, six SAR DRCs were generated for each of the four aliquots and twenty cycles of dosing/bleaching were performed among each construction of dose growth curve. Fig. 5 illustrates values (D_0) of DRCs in response to the performed cycles of dosing/bleaching for the four aliquots of quartz, which shows two characteristics: firstly, the four aliquots also produce very different values (D_0) of DRCs, ranging from ∼137.9 to ∼216.8 Gy, further confirming the previous observation that there is significant variability in the OSL dose saturation characteristic for quartz grains from the Taklimakan Desert; secondly, each of the four aliquots produces stable values (D_0) of DRCs, even though the aliquots of quartz were subjected to up to 100 additional cycles of dosing/bleaching. Such results clearly show that cycles of irradiation and optical bleaching have insignificant impacts on the dose saturation characteristics for the quartz OSL.

3.2.2. Heating effect

The effects of heating on the quartz OSL dose growth characteristics were tested by using the experiment procedure in Table 3. An aliquot of quartz of TK-1 was at first given a sequence of irradiation doses in the laboratory to construct an aliquot-specific dose growth curve (step 1). The aliquot was then heated to the

Table 1
Procedures for the construction of SAR DRCs for the quartz OSL (Murray and Wintle, 2000).

Step	Treatment	Observed
1	Blue light bleaching for 100 s at 280 °C	
2	Given dose, D_i[a]	
3	Preheat (260 °C for 10 s)	
4	Stimulate for 40 s at 125 °C	L_i[b]
5	Give test dose, 21.4 Gy	
6	Cut-heat (220 °C)	
7	Stimulate for 40 s at 125 °C	T_i[b]
8	Return to step 2	

[a] The regenerative doses are 0, 17.1, 42.7, 85.4, 128.1, 170.9, 256.3, 341.7 and 512.6 Gy, respectively. Additionally, a repeated dose of 85.4 Gy was added to the end of sequence.
[b] The OSL signals (L_x and T_x) were calculated by integrating counts in the initial 1 s of the OSL decay curve after subtraction using the averaged signal of the last 2 s.

Table 2
Constructions of the quartz SAR DRCs after the aliquots are subjected to many cycles of dosing/bleaching.

Step	Procedure	Observed
1	Construction of the DRCs[a]	D_0
2	20 cycles of dosing/bleaching[b]	
3	Construction of the DRCs[a]	D_0
4	Return to step 2	

[a] The procedures for the construction of DRCs are referred to Table 1.
[b] The size of dose is 21.4 Gy, while the aliquots were bleached with blue light at 125 °C for 40 s.

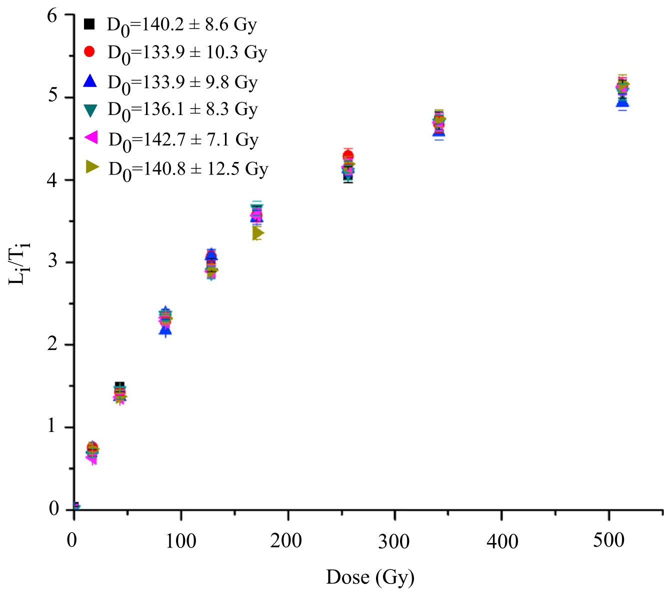

Fig. 4. Constructions of laboratory dose response curves for an aliquot of quartz grains. Among each construction of dose response curve, twenty cycles of dosing/bleaching are performed for the same aliquot.

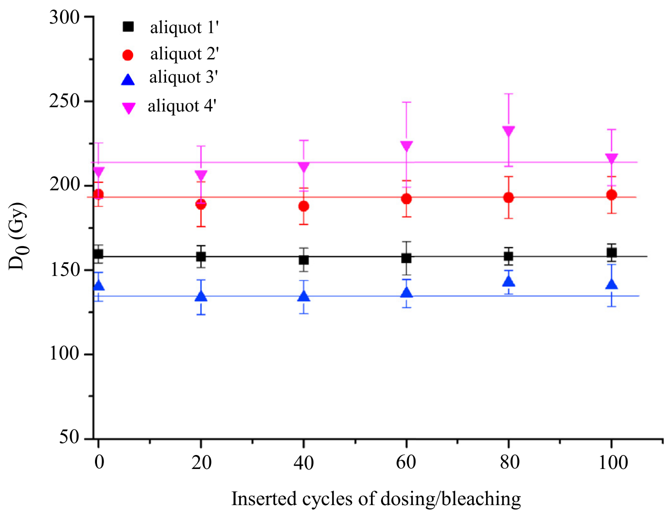

Fig. 5. The D_0 values of laboratory dose response curves for aliquots of quartz grains. Among each construction of dose response curve, twenty cycles of dosing/bleaching are performed for the same aliquot. The added lines are schematic to demonstrate that the quartz produces relative stable D_0 values.

temperature T (325 °C) (step 2) before the construction of next dose response growth curve (step 3). The heating rate was at 5 °C/s. Such treatments were repeated to regenerate eight dose response curves for the same aliquot, while the inserted heating to high temperature (T) among each construction of dose growth curve was progressively raised to 500 °C in the step of 25 °C. The generated DRCs of quartz OSL corresponding to different heating treatment were then compared (Fig. 6). It shows that heating has significant effect on the dose saturation behavior for the quartz OSL, especially for high temperatures (i.e. above 400 °C). From 325 to 375 °C, the DRCs of quartz OSL are very similar and the values (D_0) of DRCs are at ~130.5 Gy. However, the DRCs of quartz OSL progressively changed as the aliquot was heated to high temperature above 400 °C. The value (D_0) of dose growth curve decreased ~47.5% to 68.5 ± 3.5 Gy

Table 3
Constructions of quartz SAR DRCs after the aliquots are heated to different temperatures.

Step	Procedure	Observed
1	Construction of the DRCs[a]	D_0
2	Heat to T (325 °C)[b]	
3	Construction of the DRCs[a]	D_0
4	Return to step 2 and $T = T + 25$ °C	

[a] The procedures for the construction of DRCs are referred to Table 1.
[b] The heating rate operated is at 5 °C/s.

after the aliquot was heated to 500 °C. It is stressed that the recycling ratios in each construction of dose response curves are all within 10%. The results suggest that the heating to high temperature (above 400 °C) lead to the aliquot of quartz of TK-1 produce lower values (D_0) of DRCs and exhibit lower dose saturation level for OSL signals.

To further demonstrate the above results, the same experiment was operated for four aliquots of quartz of TK-1. As the same, eight DRCs were generated for each of the four aliquots and a heating treatment to temperature (325–500 °C) was inserted among each construction of dose growth curve. The values (D_0) of DRCs were then calculated and compared. Fig. 7a illustrates the values (D_0) of DRCs in response to the different heating temperatures for the four aliquots of quartz. It shows that all the four aliquots produce relative stable values (D_0) of DRCs for heating temperatures from 325 to 375 °C. Then the aliquots of quartz exhibit progressively lower values (D_0) of DRCs, after the aliquots were subjected to high temperature (above 400 °C). The results clearly show that the heating to high temperature (above 400 °C) has significant effects on the dose saturation characteristics for the quartz OSL. Moreover, another type of curve fitting was applied to the data set, using the single exponential function. The corresponding D_0 values were calculated and compared, to see whether there is still a dependence of D_0 values on the heating temperature. Higher D_0 values can be obtained by the single exponential function for the DRCs, compared with the exponential plus linear function (Fig. 7b). It is found that there is still clearly dependence of D_0 values on the heating temperature. The high temperature (above 400 °C) lead to the aliquots of quartz of TK-1 produce lower values (D_0) of DRCs and exhibit

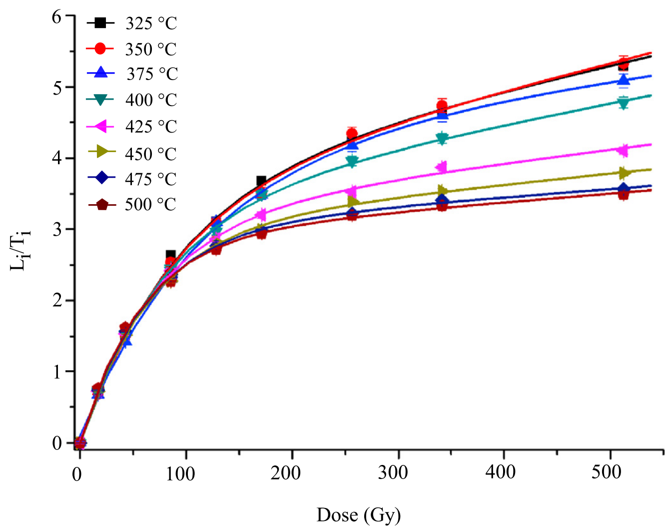

Fig. 6. Constructions of laboratory dose response curves for an aliquot of quartz. Among each construction of dose response curve, a heating treatment to high temperature T (325–500 °C) is performed.

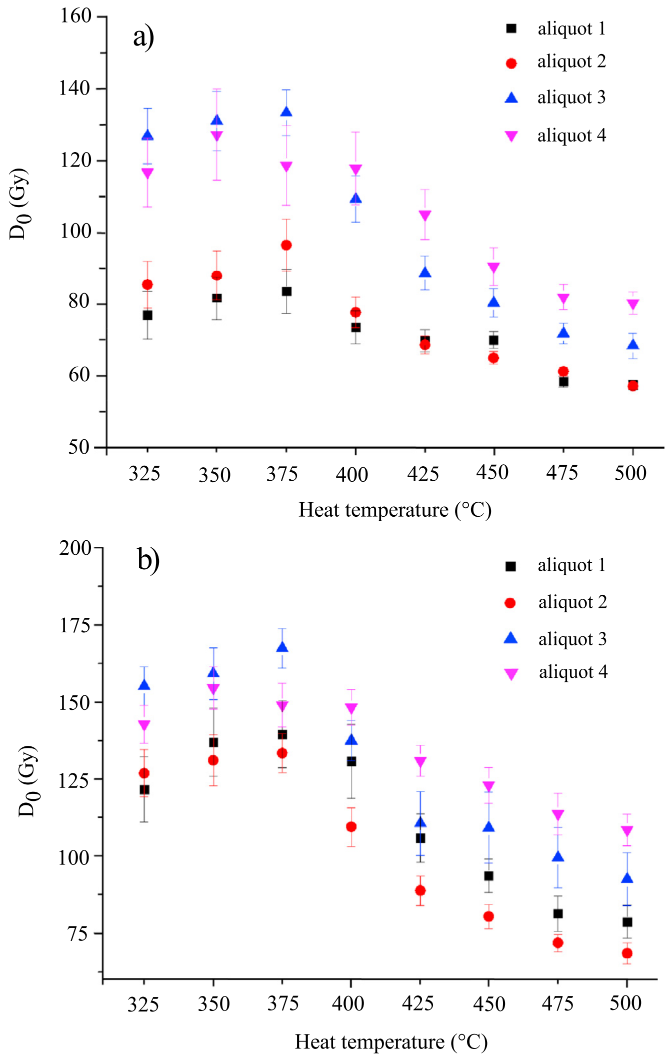

Fig. 7. The values (D_0) of laboratory dose response curves for aliquots of quartz. (a) the DRCs were fitted by the single exponential plus linear function. (b) the DRCs were fitted by the single exponential function. Among each construction of dose response curve, a heating treatment to high temperature T (325–500 °C) is performed.

lower dose saturation level for OSL signals. Lai et al. (2008) also found that the quartz sample extracted from Chinese loess produce the very different dose response curve after the quartz was heated to a temperature of 370 °C at heating rate of 1 °C/s.

4. Discussion

An implication from the above experiments is that bleaching and burial cycles during natural geological processes might have insignificant impact on the dose growth characteristics of quartz OSL signals. The different heating history might play an important role in the significant variability of the dose saturation characteristics for natural quartz grains in different regions and sedimentary environments. To check the above idea, the OSL dose saturation behavior of quartz (HS-1) from the Hunshandake sandy land was studied and compared with that of quartz (TK-1) from the Taklimakan Desert. Our results suggest that quartz grains of HS-1 can produce much brighter luminescence signals than that of TK-1 (Lü and Sun, 2011), because sands in the Hunshandake sandy land are mainly of igneous origin (Ma, 2002; Yang et al., 2008). Most of quartz grains from the Hunshandake sandy land were heated to high temperature during the geological past (Ma, 2002; Yang et al., 2008; Lü and Sun, 2011), in comparison with quartz grains from the Taklimakan Desert.

Twenty aliquots of quartz were given a sequence of irradiation doses to construct the SAR DRCs for HS-1 and TK-1, respectively (Table 1). The values (D_0) of DRCs of HS-1 and TK-1 were then calculated and compared in Fig. 8. It is found that the quartz grains of HS-1 also exhibit significant variability in dose saturation characteristics. Additionally, the quartz grains of HS-1 generally produce lower values (D_0) of DRCs than that of TK-1. The mean value (D_0) of the DRCs of HS-1 is 89.6 Gy, while the mean value (D_0) of the DRCs of TK-1 is ~48% higher at 132.7 Gy. The results show that the heated quartz from Hunshandake sandy land have lower dose saturation level for OSL signals. Thus, the OSL dating can apply to much older sedimentary quartz grains from the Taklimakan Desert than that from the Hunshandake sandy land, if the similar annual dose rates were assumed. Such results further support that the different heating history among natural quartz grains can be an important factor for the significant variability in dose saturation characteristics of OSL signals.

5. Conclusion

Coarse quartz grains from the Taklimakan Desert and the Hunshandake sandy land exhibit very different dose growth characteristics of OSL signals. Through laboratory irradiation, optical bleaching and heating experiments, it shows that the cycles of dosing and bleaching have insignificant impact on the dose saturation behavior. The quartz OSL saturates earlier as the aliquot is heated to high temperature (about 400 °C). The experiments suggest that different heating history plays an important role in the significant variability of dose saturation characteristics of natural quartz grains. Moreover, quartz grains from the Hunshandake sandy land produce much lower values (D_0) of DRCs than that from the Taklimakan Desert, supporting the sands from the Hunshandake sandy land are mainly of igneous origin.

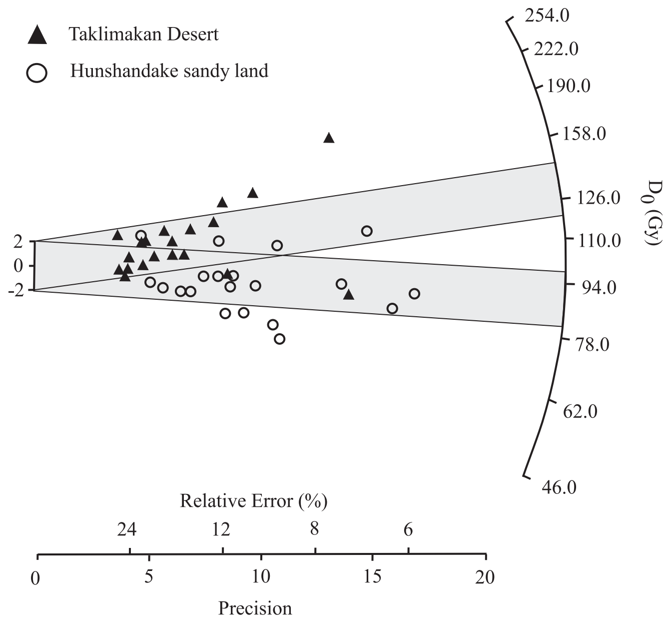

Fig. 8. Radial plot of the values (D_0) of dose response curves for quartz grains from the Taklimakan Desert and the Hunshandake sandy land. The over-dispersion values are calculated at 26.0% and 22.7% for quartz grains from the Taklimakan Desert and the Hunshandake sandy land, respectively.

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