1-1-2020

Testing the upper limit of luminescence dating based on standardised growth curves for MET-pIRIR signals of K-feldspar grains from northern China

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Publication Details Citation

Rui, X., Li, B., & Guo, Y. (2020). Testing the upper limit of luminescence dating based on standardised growth curves for MET-pIRIR signals of K-feldspar grains from northern China. Faculty of Science, Medicine and Health - Papers: Part B. Retrieved from https://ro.uow.edu.au/smhpapers1/1211

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© 2020 Elsevier B.V. The standardised growth curve (SGC) method has been applied to potassium-rich feldspar (K-feldspar) from samples at the Nihewan Basin, northern China. It was observed that the shapes of SGCs obtained from multiple-aliquot regenerative-dose (MAR) and single-aliquot regenerative-dose (SAR) procedures are different at high doses (>900 Gy) for the multiple-elevated-temperature post-IR IRSL (MET-pIRIR) signals observed at high temperatures (e.g., 290 °C) This difference can be attributed to the presence of residual signals in the MAR SGCs due to solar bleaching involved in the MAR procedure. Similar shapes of the SAR and MAR SGCs were obtained after correcting for the residual signals. Comparing the De estimates for both methods suggests that the SAR SGCs yield more reliable results. We tested the SAR SGCs for sediments from the Dadaopo section in the Nihewan Basin, including materials from the Brunhes/Matuyama (B/M) boundary (~780 ka). By interpolating the central re-normalised Ln/Tn ratios onto the corresponding SAR SGCs, the 290 °C MET-pIRIR signals yielded ages in stratigraphic order, and the sample from the B/M boundary yielded an age estimate broadly consistent with the expected age. Our results suggest that the MET-pIRIR SAR SGC method has the potential to date samples up to ~700–800 ka in this region.

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Rui, X., Li, B. & Guo, Y. (2020). Testing the upper limit of luminescence dating based on standardised growth curves for MET-pIRIR signals of K-feldspar grains from northern China. Quaternary Geochronology, 57

Testing the upper limit of luminescence dating based on standardised growth curves for MET-pIRIR signals of K-feldspar grains from northern China

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Abstract

The standardised growth curve (SGC) method has been applied to potassium-rich feldspar (K-feldspar) from samples at the Nihewan Basin, northern China. It was observed that the shapes of SGCs obtained from multiple-aliquot regenerative-dose (MAR) and single-aliquot regenerative-dose (SAR) procedures are different at high doses (>900 Gy) for the multiple-elevated-temperature post-IR IRSL (MET-pIRIR) signals observed at high temperatures (e.g., 290°C). This difference can be attributed to the presence of residual signals in the MAR SGCs due to solar bleaching involved in the MAR procedure. Similar shapes of the SAR and MAR SGCs were obtained after correcting for the residual signals. Comparing the D_e estimates for both methods suggests that the SAR SGCs yield more reliable results. We tested the SAR SGCs for sediments from the Dadaopo section in the Nihewan Basin, including materials from the Brunhes/Matuyama (B/M) boundary (~780 ka). By interpolating the central re-normalised L_n/T_n ratios onto the corresponding SAR SGCs, the 290°C MET-pIRIR signals yielded ages in stratigraphic order, and the sample from the B/M boundary yielded an age estimate broadly consistent with the expected age. Our results suggest that the MET-pIRIR SAR SGC method has the potential to date samples up to ~700–800 ka in this region.

Keywords: K-feldspar, L_n/T_n ratios, standardised growth curves, residual signal

1 Introduction

Optically stimulated luminescence (OSL) emissions from quartz grains have been widely used for dating sediments in the Quaternary period since the development of the single-aliquot regenerative-dose (SAR) protocol (e.g., Galbraith et al., 1999; Murray and Wintle, 2000; Wintle and Murray, 2006). However, dating of old deposits is limited by the relatively low saturation dose of quartz signals (~100–200 Gy), which restricts its application to dating sediments younger than ~100–200 ka (e.g., Lai, 2010;

Jacobs and Roberts, 2007; Roberts et al., 2015; Chamberlain et al., 2017). Compared to quartz, infrared stimulated luminescence (IRSL) signals from potassium-rich feldspar (K-feldspar) saturate with higher dose, making it a promising candidate to date older sediments (Aitken, 1998). However, the application of IRSL signals has long been limited due to anomalous fading, an effect of leaking trapped electrons at a much faster rate than would be expected from kinetic considerations (Wintle, 1973; Spooner, 1992; Spooner, 1994).

To overcome the age underestimation by anomalous fading, several correction methods have been proposed based on laboratory fading rate (g-value) measurements (e.g., Huntley and Lamothe, 2001; Kars et al., 2008), which are model-dependent and may not be applicable to old samples (Li and Li, 2008). Recent progress shows that following a low-temperature (50°C) IRSL measurement, post-IR IRSL (pIRIR) signals measured at elevated temperatures (i.e., >100°C) have a less-fading component (Thomsen et al., 2008). This has led to the development of the two-step pIRIR protocol (Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2011) and the multiple-elevated-temperature (MET) pIRIR protocol (Li and Li, 2011; Li and Li, 2012). Both protocols have been successfully tested using different types of sediment and equivalent dose (D_e) values consistent with expectations have been reported (e.g. Reimann et al., 2011; Madsen et al., 2011; Li and Li, 2012; Li et al., 2014). Li and Li (2012) have shown that D_e values of up to ~1000 Gy can be measured reliably using the MET-pIRIR method, corresponding to maximum ages of ~300 ka with environmental dose rates of ~3 Gy/ka. Based on the observation of a pre-dose dependency of the sensitivity for the MET-pIRIR signals, Li et al. (2013a, 2014) developed a pre-dose MET-pIRIR (pMET-pIRIR) procedure which can reliably measure doses of up to ~1600 Gy. By applying multiple-aliquot regenerative dose (MAR) pMET-pIRIR procedure on Chinese loess, the characteristic saturation dose (D_0) value of ~800 Gy for the 300 °C IRSL signal was reported by Chen et al. (2015), which allows for reliable measurements of D_e values up to ~1800 Gy ($2.3D_0$).

More recently, Li et al. (2015a, 2016) proposed using regenerative-dose normalisation (re-normalisation) and least-squares normalisation (LS-normalisation) methods to reduce the between-aliquot differences in dose response curve (DRC) shapes for single aliquots of quartz and K-feldspars. As a result, the standardised growth curves (SGCs) of SAR and MAR could be established for K-feldspar (Li et al., 2015b, 2017a). With the establishment of SGC, Li et al. (2017b, 2018) suggested a method for D_e determination based on the full (‘untruncated’) distribution of L_n/T_n ratios for all aliquots, so-called L_n/T_n method (Jacobs et al., 2019; Hu et al., 2019), and showed that it can overcome the problem of D_e underestimation for samples with natural doses close to saturation. These methods have the potential to date older sediments and extend the dating range to the early middle Pleistocene or possibly the late early Pleistocene.

In this study, the MAR and SAR SGCs were established using K-feldspar extracted from different locations in the Nihewan Basin, northern China. We show that different shapes in the MAR and SAR SGCs at high temperatures can be attributed to the different amounts of residual signal in the MAR and SAR SGCs. Correction of the residual signal resulted in consistent results between the two methods. We tested the methods using sediments from the Dadaopo section in the Nihewan Basin, including material from the Brunhes/Matuyama (B/M) boundary, investigated the validity of D_e estimation using the SGCs and L_n/T_n ratios for samples over the last 780 ka, and explored their corresponding dating limit.

2 Sample collection, analytical facilities, and dose rate determination

2.1 Samples

The Nihewan Basin is located at the north-eastern margin of the Chinese Loess Plateau, about 150 km west of Beijing (Fig. 1a). Samples from ten different locations in the basin (Fig. 1b), including Dadaopo (DDP), Donggutuo (DGT), Hutouliang (two localities: HTL-Loc1 and HTL-Loc2), Heshangping (HSP), Hongya (HY), Taiergou (TEG), Majuanpo (MJP), Xiashazui (XSZ), Zhaojiaping (ZJP), were used to establish SGCs. The deposits of these sections are composed of loess, fluvial, and fluvial-lacustrine sediments. The deposit type, grain size, and D_e ranges for each sample are summarised in Table 1.

Among these sections, the chronology of the 129-m-thick Dadaopo section (also known as Haojiatai) has been reported using the magnetostratigraphical method (Zhu et al., 2004). Seven samples from the Dadaopo section were investigated in detail to test the upper dating limit of the MET-pIRIR and the SGC procedures. The deepest sample from this section (DDP-1) was collected from the B/M boundary (Fig. 2) and has an age estimate of ~780 ka. The other samples from this section (DDP-2, -3, -4, -5, -6, -7) were collected above the sample DDP-1. A summary of sampling depths is shown in Table 2.

All samples in this study were collected either in stainless steel tubes or as blocks. After the tubes or blocks were removed, they were immediately wrapped in light-proof plastic and transported to the Luminescence Dating Laboratory at the University of Wollongong for analysis.

2.2 Experimental procedures and analytical facilities

Sample preparation was conducted under subdued red-light condition. About 2 cm from both ends of the sampling tubes and the outer surfaces (~1 cm) of the blocks were removed as they may have been exposed to sunlight during sampling. The central part of the samples was treated with hydrochloric acid (HCl) and hydrogen peroxide (H_2O_2) to remove carbonates and organic material, respectively, and then wet sieved to obtain grains of 63–90, 90–125, 90–150 or 125–180 µm diameter. K-feldspar grains were separated from quartz and heavy minerals using a heavy liquid solution (2.58 g/cm^3). These grains were then etched in 10% HF acid for ~40 min to remove the alpha-irradiated outer layer and adhesive clays. The etched feldspar grains were then washed in HCl acid to remove any precipitated fluorides.

The collected K-feldspar grains mounted as a monolayer on the central ~5 mm diameter portion of each stainless-steel disc (9.8 mm diameter) were used for the IRSL measurements. The IRSL measurements were made on automated Risø OSL/TL readers (models DA-15 and DA-20) with infrared diodes (870 nm). Irradiation was carried out within the readers using a calibrated $^{90}Sr/^{90}Y$ beta source. The signals were measured with an Electron Tubes 9235B photo-multiplier (PM) tube through a filter pack of Schott BG-39 and Corning 7-59 filters. A solar simulator (Dr Hönle UVACUBE 400) was used for bleaching in relevant experiments.

For the MET-pIRIR measurements, infrared stimulations were performed successively at six temperatures (50, 100, 150, 200, 250 and 290°C) for 100 s. The net pIRIR signals were calculated as the sum of counts in the first 10 s of pIRIR decay minus a 'late light' background estimated from the mean count rate over the final 10 s. For each IRSL measurement, an 'IR-off' period of up to 50 s prior to stimulation was applied to monitor and minimise the isothermal decay signal (Fu et al., 2012). A solar simulator bleaching for 4 hours was applied at the end of each SAR cycle to reset the pre-dose 'memory' from the preceding cycle (Li et al., 2014).

2.3 Dose rate determinations

For environmental dose rate determination, the U, Th and K contents of samples from DDP were measured using a combination of ICP-MS (for U and Th) and ICP-OES (for K). Cosmic-ray dose rates were estimated from the burial depth of each sample and the latitude, longitude and altitude of the site (Prescott and Hutton, 1994). For the calculation of the internal dose rate, the internal K content was assumed to be 12 ± 1% based on scanning electron microscopy analysis of single-grain K-feldspar samples from the same region (Rui et al., 2019). The minor contribution from Rb was calculated by assuming a concentration of 400 ± 100 ppm (Huntley and Hancock, 2001).

The external dose rates were corrected for long-term water attenuation (Aitken, 1985). The measured (field) water contents for our samples range from a few percent for the loess sample (DDP-7) to up to 20% for the lacustrine samples (Table 2). We, however, consider that the measured field water contents underestimate the long-term water contents. This is because the Nihewan Basin used to be covered by a paleolake from the late Pliocene to the late Pleistocene until it was drained by the Sanggan River (Yuan et al., 2011). This means that the lacustrine sediments were initially saturated with water but then dried out after the disappearance of the Nihewan paleolake. Hence, the field water contents of the dried lacustrine sediments cannot represent the average water contents since deposition. In order to estimate the effective water contents for the lacustrine samples, saturated water contents were measured using several sub-samples from DDP-3, which yielded values of ~30–40%. Given that the paleolake has been dry for the last ~100–300 ka (Guo et al., 2016), we have adopted a value of 25 ± 10% as an estimate for the long-term average water content for the lacustrine samples by taking both the periods of being saturated with water and being dried out into account. We assigned a large relative error (~40%) to this value, so that it will capture any likely fluctuation of water content at the 2σ range (from 5 to 45%). For the loess sample, we have adopted a value of 10 ± 5%, based on the previous studies on loess in this region and in the Chinese Loess Plateau (Buylaert et al., 2008; Zhao et al., 2010; Guo et al., 2016). The dosimetry data for all samples from DDP are summarised in Table 2.

3 Results

3.1 Establishment of SGCs

3.1.1 SAR and MAR SGCs

To construct the SAR SGCs, the SAR MET-pIRIR procedure (Table 3) of Li et al. (2014) was applied to 7 samples (DDP-3, DGT-1, HSP-1, HTL-Loc2-1, HY-1, TEG-1, ZJP-4). Fig. 3 shows representative natural IRSL (50°C) and MET-pIRIR (100–290°C) decay curves for an aliquot of sample TEG-1. The intensities of the MET-pIRIR signals are on the order of several thousands of counts per 0.1 s. The obtained SAR SGCs (after LS-normalisation) were normalised using the signal corresponding to a regenerative dose of 373 Gy (Fig. 4, black dashed lines). Details about normalisation and curve fitting are presented separately (Rui et al., 2020).

To investigate if there is any issue associated with the sensitivity change during measurement of the natural signal and any possible cumulative sensitivity change induced by the successive dose and measurement cycles in the SAR procedure (e.g., Chen et al., 2013; Wallinga et al., 2000; Zhang, 2018), the MAR SGCs were also tested and compared with the SAR SGCs. A total of 70 aliquots from 12 samples (DDP-2, DDP-3, DGT-1, HSP-1, HTL-Loc2-1, HY-1, HY-3, TEG-1, MJP-1, XSZ-1, ZJP-2, ZJP-4) were bleached in the solar simulator for ~8 h. These aliquots were then given different regenerative doses, ranging from 0 to ~2400 Gy, and were measured using the MAR MET-pIRIR procedure in Table 4. After measuring the L_n and T_n signals, each aliquot was bleached for 4 h in the solar simulator and then given a regenerative dose of 373 Gy and a subsequent test dose of 60 Gy to measure L_r/T_r. The obtained re-normalised ratios ($\frac{Ln/Tn}{Lr/Tr}$) were fitted by the general order kinetic (GOK) function (Guralnik et al., 2015) to establish the MAR SGCs (Fig. 4, red solid lines).

For both the SAR and MAR SGCs, the signal values for a 373 Gy dose value were re-scaled to unity, so their shapes are directly comparable. At 50°C, the SAR SGC exhibits a steeper growth at low doses and reaches a slightly lower saturation level (Fig. 4a). Less than 4% difference in SGC shapes and only a slight difference in saturation levels are observed at stimulation temperatures of 100 and 150°C (Fig. 4b and 4c). For the IRSL signals at 200, 250 and 290°C, the MAR and SAR SGCs are indistinguishable (less than 5% difference) at doses up to ~1550, ~1100 and ~900 Gy, respectively, but start to diverge at higher doses, with the MAR SGCs reaching a slightly higher saturation signal level than the SAR SGCs (Fig. 4d–4f). The results suggest that considerably different D_e results will be obtained from the SAR and MAR SGCs at high doses for the 200, 250 and 290°C MET-pIRIR signals. It is, therefore, important to investigate which of the two sets of SGCs is more reliable.

3.1.2 Effect of residual signal

The MAR procedure includes solar bleaching, giving a laboratory beta dose, and measuring the signal, which is exactly the same as the first cycle for a dose recovery test. Hence, a mismatch between the MAR and SAR SGCs would indicate a failure in the dose recovery test. Previous studies have demonstrated that a failure of the dose recovery test could result from the charge carry-over from the measurement of the regenerative-dose signal to the test dose signal (e.g., Nian et a., 2016; Yi et al., 2016; Colarossi et al., 2019) or from an initial sensitivity change during the measurement of the 'natural' signal (e.g., Wallinga et al., 2000; Chen et al., 2013; Li et al., 2013a; Kars et al., 2014; Zhang, 2018; Qin et al., 2018). Based on a dose recovery study using the same samples from the Nihewan Basin (Rui et al., 2020), both of these effects were ruled out as the main reason for the failure of dose recovery. Instead, they demonstrated that residual signal plays an important role in the failure of dose recovery at high doses. Previous studies suggested that the pIRIR signals may contain both the dose-dependent bleachable and non-bleachable (residual) signals (Li et al., 2013b; Rui et al., 2020). The bleachable and residual signals of the MAR and SAR SGCs can be compared to study the causes of the differences in the SGCs. To separate the residual signal and the bleachable signal, the signal-subtraction method can be applied, in which the residual signal is measured for the regenerative dose to estimate its corresponding bleachable signal (Li et al., 2013b).

The residual signal measurements for the SAR SGCs were conducted on a relatively young sample (HTL-Loc1-2, D_e = 38.3 ± 5.3 Gy). The obtained residual and bleachable SAR SGCs were provided in Fig. 5a–5f (red solid lines and red dashed lines, respectively). The residual signals, normalised using the same regenerative-dose (373 Gy) signals, show a clear dose dependency for higher-temperature stimulations (e.g., 250 and 290°C).

To estimate the residual signals for the MAR SGCs, eight samples (DDP-3, DDP-7, HSP-1, HTL-Loc2-1, HY-3, TEG-1, MJP-1, ZJP-4) were bleached by solar simulator for 8 h to remove their natural signals. Following the solar bleaching, a total of 50 aliquots were given different regenerative doses, ranging from 0 to 2500 Gy. They were then bleached for 8 h to remove all the bleachable signals before measuring the residual signals and the corresponding test dose signals. After that, a regenerative dose (D_r = 373 Gy) was applied and measured to normalise between-aliquot variation (Table 4). In contrast to the residual signals for the SAR SGCs, the residual signals for the MAR SGCs appear to have a smaller or no dependency on dose (Fig. 5g–5l, dots). These residual signals were subtracted from the total signals (Fig. 5g–5l, black dashed lines) to calculate the bleachable MAR signals (Fig. 5g–5l, red dashed lines).

The results from Fig. 5m show that the SAR SGC still differs from the MAR SGC at 50°C after correcting residual signals, which has been explained as the initial sensitivity change (see Rui et al., 2020). The bleachable IRSL signals at 100 and 150°C share similar SGCs (Fig. 5n and 5o). After removing residual signal, the differences in SGC shapes for the high-temperature pIRIR signals were significantly reduced. Both the curves for the 200 and 250 °C signals become indistinguishable up to ~1000 Gy before they start to diverge slightly beyond 1000 Gy and differ by ~6% at ~2000 Gy (Fig. 5p and 5q). For the 290°C signal (Fig. 5r), however, both the SAR and MAR SGCs remain indistinguishable up to ~1500 Gy and differ by less than 4% up to ~2300 Gy. The results indicate that the differences in the shapes of the SAR and MAR SGCs for the high-temperature pIRIR signals (Fig. 4f) can be attributed to the effect of the residual signal.

3.1.3 D_e comparison using SAR and MAR SGCs

To test the reliability of the SAR and MAR SGCs, D_e estimates obtained using both the SAR and MAR SGCs were compared for sample HY-3. We first estimated the bleachable signals of the natural aliquots. Four discs were bleached for 8 h by solar simulator to remove all bleachable signals before being measured using the same procedure described above. The bleachable component of the normalised natural signal intensity (1.23 ± 0.02) has been estimated by subtracting the normalised residual signal (0.15 ± 0.01) from the corresponding total natural signal (1.38 ± 0.02) (2σ error). The D_e obtained by interpolating the natural (total) signal at 290°C onto the corresponding total SAR SGC (1064 ± 70 Gy, 2σ error) is consistent with the D_e obtained by interpolating the bleachable signal onto the bleachable SAR and MAR SGCs (954 ± 67 and 976 ± 62 Gy, respectively, 2σ error) (Fig. 6), which suggests the SAR SGCs are reliable for D_e estimation. However, the D_e obtained from the total signal based on the total MAR SGC (844 ± 46 Gy, 2σ error) is significantly underestimated, suggesting that the total MAR SGCs contain more significant residual signals than do the natural signals.

3.2 Dating results

3.2.1 Dating results for DDP section

For seven samples from the DDP section, 10–20 aliquots were used to measure the natural signals. Fig. 7 shows the distributions of the re-normalised ratios ($\frac{Ln/Tn}{Lr/Tr}$) for the MET-pIRIR signals measured at a stimulation temperature of 290°C. The re-normalised ratios are all distributed around a central value and most of them are consistent with each other at 2σ.

The proportions of K-feldspar aliquots with saturated natural signals at 290°C (i.e., the re-normalised ratios consistent with or higher than the maximum signal level of the corresponding SGC) range from zero for the youngest sample (DDP-7) to ~25% for the oldest sample (DDP-1). Several studies have shown that rejecting a large number of the saturated aliquots may result in significant underestimation of the final D_e values, due to truncation of the full D_e distribution (Li et al., 2016; Guo et al., 2017). To avoid this problem, instead of projecting the re-normalised natural signal for each aliquot onto the SGC, the central age model (CAM) was applied to the re-normalised L_n/T_n ratios and the resulting CAM ratio of each IR temperature was projected onto the corresponding total SAR SGC to estimate their final D_e values. Fig. 8 shows the obtained SAR D_e values and the standard errors plotted against stimulation temperatures. Except for sample DDP-5, which shows a 'plateau' in D_e values between 250 and 290°C, all other samples exhibit a systematic increase in D_e with stimulation temperature. The increase of D_e with stimulation temperature can be explained by either 1) the MET-pIRIR signal measured at 250°C is still subject to fading and the signal measured at 290°C fades negligibly; or 2) the signals measured at 250 and 290°C are both subject to fading, but the fading rate is smaller for the 290°C signal.

One way to test whether the MET-pIRIR$_{290}$ signal is fading or not is to compare the derived ages against independent age controls. The MET-pIRIR$_{290}$ ages of all the samples are in stratigraphic order (Table 5 apparent ages), increasing down-profile from the top (72 ± 3 ka, DDP-7) to the B/M boundary (679 ± 127 ka, DDP-1); the latter is statistically consistent with the expected age of ~780 ka. Given the large uncertainty in the age (~ 20%), however, we cannot safely rule out that there is no fading in the MET-pIRIR$_{290}$ signal.

In order to further test whether the MET-pIRIR$_{290}$ signal suffers from fading or not, we conducted a laboratory fading test on sample DDP-1. We adopted a single-aliquot procedure similar to that described by Auclair et al. (2003), but based on the protocol of Table 3. Twelve discs that had been used for D_e measurements were given an infrared bleaching at 340°C to ensure that the infrared-sensitive traps were empty. A dose of ~136 Gy was administered using a laboratory beta source and the irradiated aliquots were then preheated and stored for different periods of time at room temperature (~20°C) before the MET-pIRIR signals were measured. For practical reasons, an infrared bleaching at 340°C for 100 s, rather than a solar simulator bleach for 4 h, was given at the end of each SAR cycle to minimise the size of any residual signals. All the measured MET-pIRIR L_x/T_x values were normalised by the immediately measured values (i.e., promptly) and then plotted against the storage times (Fig. 9a). Fig. 9b shows the corresponding fading rates and that the 50°C IRSL signal has the highest fading rate (3.9 ± 1.0 %/decade). As the g-values have large uncertainties, there are no observable significant changes for the higher temperature MET-pIRIR signals, which range from 0.2 ± 1.0 %/decade (MET-pIRIR$_{100}$ signal) to 0.7 ± 0.5 %/decade (MET-pIRIR$_{290}$). The fading test appears to support that there is a negligible fading rate of the MET-pIRIR$_{290}$ signal in the samples from DDP.

However, it has been reported that the fading rates are dose dependent, with older samples suffering from more fading (Huntley and Lian, 2006; Li and Li., 2008). This suggests that a small fading rate observed at low dose in a laboratory fading test may not necessarily represent the true average fading rate for natural samples, especially for old samples such as DDP-1. In order to estimate the natural fading rate, we calculated the expected non-fading natural signal based on the expected natural dose of sample DDP-1 (~2145 Gy); the latter was based on its expected age of ~780 ka (Zhu et al., 2004) and its measured environmental dose rate of 2.75 Gy/ka (Table 2). By interpolating the expected dose onto the SAR SGC at 290°C, the expected L_n/T_n value of 1.56 can be obtained. Hence, the measured natural MET-pIRIR$_{290}$ signal for sample DDP-1 (1.53 ± 0.04) had reached 98.1 ± 2.6% of the expected level. Given the oldest sample should suffer from the maximum fading due to the dose-dependency of anomalous fading (Li and Li, 2008), sample DDP-1 provides a maximum estimate of the overall fading for all the other samples, i.e., the maximum underestimation due to fading ranges from 0 to ~7% at 95% confidence interval. Since a fading rate of 1% would roughly result in ~10% underestimation in age for young samples (Huntely and Lamothe, 2001), this suggests that the overall anomalous fading rates for our samples should be smaller than 1 %/decade, which is consistent with our fading test results in Fig. 9.

3.2.2 Simulating the effect of natural signal loss

In order to estimate the maximum ages for the younger samples, we have used the measured-to-expected ratio of the natural signal for DDP-1 as a guide and assumed that they have the same anomalous fading rates. We applied the percentage loss (98.1 ± 2.6%) in natural signal to correct all samples from the DDP section, i.e., by dividing the natural signals from the younger samples by 0.981 ± 0.026 and then estimating the corresponding D_e values and ages. The results are listed in Table 5. For the youngest sample (DDP-7), the age increased by ~4% from the apparent (72.0 ± 3.5 ka) to the fading-corrected maximum age (74.8 ± 5.5 ka). The ages increased by ~8–11% for the samples older than 300 ka. Given younger samples should suffer less fading than older samples, the true ages of these samples should be between the apparent ages and the maximum ages (Fig. 2).

In order to further investigate the influence of fading of the natural signal on D_e estimation, we modelled six scenarios, in which different percentages (f) of natural signal (L_N) are lost due to anomalous fading, including 0.5%, 1.5%, 2.5%, 3.5%, 5% and 7.5%. For each scenario, we modelled a range of samples with paleodoses (P) ranging from 0 to 2400 Gy and calculated the corresponding L_N for each P value based on the parameters of the SAR SGC at 290°C. D_e values can be obtained by interpolating the modelled natural signal ($L_N' = (1–f) * L_N$) onto the corresponding SAR SGC.

Fig. 10 shows the modelled D_e/P ratios calculated using different percentage losses in natural signals. It shows that the extent of underestimation in D_e increases with dose, which is expected because a small change in signal will result in a significant change in D_e at the non-linear range of the growth curve. If the signal lost is small (e.g. < 1.5%), the underestimation in D_e values can be up to 10% for P values as high as 2200 Gy. For a signal loss of 2.5%, the underestimation of D_e values is less than 10% when P is less than 1100 Gy, but an underestimation of 15% is obtained for P = 2200 Gy. Such an effect is even more severe for a larger natural signal loss (Fig. 10).

4 Discussion

In this study, the SGCs were investigated using both the MAR and SAR procedures based on the MET-pIRIR signals from feldspar grains. For the IRSL signals at 200, 250 and 290°C, the MAR and

SAR SGCs are indistinguishable (less than 5% difference) at doses up to ~1550, ~1100 and ~900 Gy, but start to diverge at higher doses, with the MAR SGCs reaching slightly higher saturation signal levels than the SAR SGCs (Fig. 4d–4f). The different shapes in the SAR and MAR SGCs would imply failures in dose recovery tests and two different sets of D_e values. Several reasons have been reported as the likely sources of the failed dose recovery tests, including the charge carry-over from the measurements of L_x to T_x (e.g., Nian et al., 2016; Yi et al., 2016; Colarossi et al., 2019), the initial sensitivity changes during the measurement of the first signal (i.e., L_n) (e.g., Wallinga et al., 2000; Kars et al., 2014; Zhang, 2018; Qin et al., 2018), and the inappropriate corrections for the residual signals (Rui et al., 2020). Here we demonstrated that the differences between the MAR and SAR SGCs are significantly reduced after correcting for the residual signals, indicating that the residual signals are the main cause for the differences between the SAR and MAR SGCs for our samples. The similarity in the shapes of the MAR and SAR SGCs after correcting residual signals also implies that sensitivity change is not the main reason for the different SGC shapes.

As the SGC shape can be affected by the residual signal, our study also suggested that the residual signal in the regenerative dose plays an important role in D_e estimation. This is confirmed by the underestimated D_e result using the MAR SGC (Fig. 6), i.e., the residual of the regenerative signal may not be reset by laboratory solar bleaching before building the MAR SGC. In contrast, our investigation of the residual signals for natural samples of different ages from this region suggest that the residual signals are well-reset before burial (Rui et al., 2020). As a result, the SAR SGC will yield more reliable results than the MAR SGC, because the residual signals are reset by IR stimulations during the SAR cycles.

In this study, a recently proposed method was applied for the samples from the Nihewan Basin, northern China, in which the SGCs are established and the CAM re-normalised L_n/T_n ratios from all measured aliquots are projected onto the corresponding SAR SGCs to determine D_e values. Table 5 indicates the MET-pIRIR$_{290}$ ages from the DDP section increase systematically from 72 to 679 ka, in stratigraphical order. The MET-pIRIR$_{290}$ signal yielded a D_e value of 1866^{+382}_{-285} Gy for the sample DDP-1, which is broadly consistent with the expected D_e value of 2145 Gy obtained from the expected age and the measured environmental dose rate for the sample. These results further demonstrate that D_e estimates up to ~1900 Gy or more can be obtained using the SAR SGCs and the normalised L_n/T_n method. The normalised L_n/T_n for the MET-pIRIR$_{290}$ of our oldest sample DDP-1 is 1.53, corresponding to 96% of the maximum level of the SAR SGC curve (1.59), confirming that this method can produce reliable D_e results far beyond the conventionally viewed limit of $2D_0$ or 86% of the maximum level of the growth curve (Wintle and Murray, 2006).

Our study also suggested that even a small amount of natural signal loss (< 2%), as a result of small fading rate that cannot be detected in a laboratory fading test, can result in a large D_e

underestimation at high doses (e.g., > 1000 Gy) (Fig. 10). The poor precision of *g*-values obtained from the laboratory fading test would prevent a reliable fading correction, given the model-dependency of the existing fading correction methods (e.g., Huntley and Lamothe, 2001; Kars et al., 2008). Here we show that it is possible to investigate the maximum underestimation in age caused by fading, by comparing the natural intensity from an old sample with its expected intensity. Ideally, this can be done by measuring samples with known ages and comparing their apparent natural signals against the expected ones. However, samples with independent age controls are not always practical for every site. An alternative way to do that is to measure an ‘infinitely’ old sample from the same region, in which the natural signal should have reached a field-saturation, so that the expected natural signal intensity should be the same as the maximum level of the growth curve.

5 Conclusion

The SGCs were investigated using both the MAR and SAR procedures based on the MET-pIRIR signals from the Nihewan Basin, northern China. Different shapes in the MAR and SAR SGCs were observed for signals measured at high temperatures (> 200°C), which can be attributed to the residual of the regenerative signals. The dating method by projecting the central re-normalised L_n/T_n from the measured aliquots onto the corresponding SAR SGCs was successfully applied to the sediments from the B/M boundary (~780 ka), which shows that the SAR SGC is potentially capable of providing reliable D_e estimation of up to ~1900 Gy.

Acknowledgements

This study was supported by an Australian Research Council Future Fellowship to Bo Li (FT140100384), grants from the National Natural Science Foundation of China to Yujie Guo (No. 41702192) and postgraduate scholarships from the China Scholarship Council and the University of Wollongong to Xue Rui (201506010345). We thank Jiafu Zhang, Baoyin Yuan, Yanyan Yan and Junkang Wang for helps with the field investigations and collection of luminescence samples, Richard G. Roberts, Zenobia Jacobs, Yasaman Jafari and Terry Lachlan for essential support in the luminescence dating laboratory, Mariana Sontag-González for proof reading and Tony Reimann and another anonymous reviewer for their helpful comments.

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Table 1

Summary of depositional contexts, grain sizes, and equivalent dose (D_e) estimates for the studied samples.

Sample code	Section	Deposit type	Grain size (μm)	*Re-normalized* L_n/T_n[a]	Measured equivalent dose (Gy)[b]
DDP-2	DDP	Fluvial-lacustrine	90–125	1.42 ± 0.01	1201.8 ± 51.1
DDP-3	DDP	Fluvial-lacustrine	90–125	1.38 ± 0.01	1048.5 ± 38.9
DDP-7	DDP	Loess	90–125	0.82 ± 0.01	236.6 ± 6.5
DGT-1	DGT	Fluvial-lacustrine	63–90	1.44 ± 0.02	1287.45 ± 77.5
HTL-Loc1-2	HTL-Loc1	Fluvial	125–180	0.30 ± 0.02	38.3 ± 5.3
HTL-Loc2-1	HTL-Loc2	Fluvial-lacustrine	90–150	1.32 ± 0.02	887.1 ± 45.9
HSP-1	HSP	Fluvial-lacustrine	90–125	1.46 ± 0.04	1477.7 ± 223.6
HY-1	HY	Fluvial-lacustrine	90–125	1.46 ± 0.01	1442.5 ± 79.8
HY-3	HY	Fluvial-lacustrine	90–125	1.38 ± 0.01	1096.9 ± 47.5
TEG-1	TEG	Fluvial-lacustrine	90–125	1.46 ± 0.05	1477.9 ± 281.0
MJP-1	MJP	Fluvial-lacustrine	90–125	1.23 ± 0.03	669.3 ± 53.7
XSZ-1	XSZ	Fluvial-lacustrine	125–180	1.22 ± 0.02	659.7 ± 34.0
ZJP-2	ZJP	Fluvial-lacustrine	125–180	1.47 ± 0.03	1456.4 ± 182.9
ZJP-4	ZJP	Fluvial-lacustrine	90–125	1.31 ± 0.02	860.4 ± 50.8

[a] The natural signal (L_n), a single regenerative-dose signal (L_r, given dose 373 Gy) and the corresponding test dose signals (T_n and T_r) were measured for aliquots from each sample. The central age model (CAM) was applied to the re-normalised ratios ($\frac{Ln/Tn}{Lr/Tr}$) to obtain the central value.

[b] The equivalent dose of each sample was obtained by projecting the CAM value of the re-normalized L_n/T_n ratios of the MET-pIRIR$_{290}$ onto the corresponding SAR SGC without any residual correction.

Table 2

Dose rates for samples from the DDP section.

Sample	**Depth (m)**	**Grain size (μm)**	**Field water content (%)**	**Estimated water content (%)**	**U (ppm)**	**Th (ppm)**	**K (%)**	**Environmental dose rate (Gy/ka)**				
								External			**Internal**	**Total**
								Gamma	**Beta**	**Cosmic**		
DDP-7	7	90–125	1.7	10 ± 5	2.36	9.65	1.82	1.06 ± 0.07	1.68 ± 0.11	0.081 ± 0.008	0.46 ± 0.04	3.28 ± 0.13
DDP-6	10	63–90	3.0	25 ± 10	1.58	5.64	1.8	0.70 ± 0.07	1.28 ± 0.13	0.057 ± 0.006	0.38 ± 0.03	2.42 ± 0.14
DDP-5	14.5	90–125	1.8	25 ± 10	2.56	7.12	1.92	0.87 ± 0.08	1.48 ± 0.15	0.037 ± 0.004	0.46 ± 0.04	2.84 ± 0.17
DDP-4	16	63–90	19.2	25 ± 10	3.97	8.59	2.01	1.06 ± 0.10	1.70 ± 0.17	0.032 ± 0.003	0.38 ± 0.03	3.17 ± 0.20
DDP-3	20	90–125	2.8	25 ± 10	2.46	7.96	1.82	0.87 ± 0.08	1.43 ± 0.14	0.023 ± 0.002	0.46 ± 0.04	2.78 ± 0.17
DDP-2	34	90–125	1.2	25 ± 10	1.79	6.95	1.77	0.76 ± 0.07	1.31 ± 0.13	0.010 ± 0.001	0.46 ± 0.04	2.54 ± 0.15
DDP-1	50	90–125	1.7	25 ± 10	1.97	8.52	1.88	0.86 ± 0.08	1.42 ± 0.14	0.005 ± 0.001	0.46 ± 0.04	2.75 ± 0.17

Table 3

Single-aliquot regenerative-dose (SAR) procedure for multiple elevated temperature post-infrared IRSL (MET-pIRIR) measurements (Li et al., 2014).

Step	Treatment	Signal
1	Give regenerative dose, D_i[a]	
2	Preheat at 320°C for 60 s	
3	IRSL measurement at 50°C for 100 s	$L_{x(50)}$
4	IRSL measurement at 100°C for 100 s	$L_{x(100)}$
5	IRSL measurement at 150°C for 100 s	$L_{x(150)}$
6	IRSL measurement at 200°C for 100 s	$L_{x(200)}$
7	IRSL measurement at 250°C for 100 s	$L_{x(250)}$
8	IRSL measurement at 290°C for 100 s	$L_{x(290)}$
9	Give test dose, 60 Gy	
10	Preheat at 320°C for 60 s	
11	IRSL measurement at 50°C for 100 s	$T_{x(50)}$
12	IRSL measurement at 100°C for 100 s	$T_{x(100)}$
13	IRSL measurement at 150°C for 100 s	$T_{x(150)}$
14	IRSL measurement at 200°C for 100 s	$T_{x(200)}$
15	IRSL measurement at 250°C for 100 s	$T_{x(250)}$
16	IRSL measurement at 290°C for 100 s	$T_{x(290)}$
17	Solar simulator bleach for 4 h	
18	Repeat step 1–17 for different D_i	

[a] For the natural sample, $i = 0$ and $D_i = 0$ Gy, and the observed signals are denoted as L_n and T_n.

Table 4

Multiple-aliquot regenerative-dose (MAR) procedure for MET-pIRIR measurements (Li et al., 2017a).

Step	Treatment	Signal
1	Give regenerative dose, D_i[a]	
2	Preheat at 320°C for 60 s	
3	IRSL measurement at 50°C for 100 s	$L_{x(50)}$
4	IRSL measurement at 100°C for 100 s	$L_{x(100)}$
5	IRSL measurement at 150°C for 100 s	$L_{x(150)}$
6	IRSL measurement at 200°C for 100 s	$L_{x(200)}$
7	IRSL measurement at 250°C for 100 s	$L_{x(250)}$
8	IRSL measurement at 290°C for 100 s	$L_{x(290)}$
9	Give test dose, 60 Gy	
10	Preheat at 320°C for 60 s	
11	IRSL measurement at 50°C for 100 s	$T_{x(50)}$
12	IRSL measurement at 100°C for 100 s	$T_{x(100)}$
13	IRSL measurement at 150°C for 100 s	$T_{x(150)}$
14	IRSL measurement at 200°C for 100 s	$T_{x(200)}$
15	IRSL measurement at 250°C for 100 s	$T_{x(250)}$
16	IRSL measurement at 290°C for 100 s	$T_{x(290)}$
17	Solar simulator bleach for 4 h	
18	Give normalisation dose, D_r	
19	Preheat at 320°C for 60 s	
20	IRSL measurement at 50°C for 100 s	$L_{r(50)}$
21	IRSL measurement at 100°C for 100 s	$L_{r(100)}$
22	IRSL measurement at 150°C for 100 s	$L_{r(150)}$
23	IRSL measurement at 200°C for 100 s	$L_{r(200)}$
24	IRSL measurement at 250°C for 100 s	$L_{r(250)}$
25	IRSL measurement at 290°C for 100 s	$L_{r(290)}$
26	Give test dose, 60 Gy	
27	Preheat at 320°C for 60 s	
28	IRSL measurement at 50°C for 100 s	$T_{r(50)}$
29	IRSL measurement at 100°C for 100 s	$T_{r(100)}$
30	IRSL measurement at 150°C for 100 s	$T_{r(150)}$
31	IRSL measurement at 200°C for 100 s	$T_{r(200)}$
32	IRSL measurement at 250°C for 100 s	$T_{r(250)}$
33	IRSL measurement at 290°C for 100 s	$T_{r(290)}$

[a] For the natural sample, $i = 0$ and $D_i = 0$ Gy, and the observed signals are denoted as L_n and T_n.

Table 5

Summary of the uncorrected (measured) and corrected (compensation of 1.9% measured signal to account for signal loss due to anomalous fading) MET-pIRIR$_{290}$ signals and the SAR D_e data for the DDP samples, with the corresponding age results (apparent and maximum ages, respectively).

Sample	Measured signal	Corrected signal	Measured D_e (Gy)	Corrected D_e (Gy)	Apparent age (ka)	Maximum age (ka)
DDP-7	0.82 ± 0.01	0.83 ± 0.02	236.6 ± 6.5	245.8 ± 15.4	72.0 ± 3.5	74.8 ± 5.5
DDP-6	1.29 ± 0.01	1.32 ± 0.04	813.4 ± 33.3	879.1 ± 101.3	336.4 ± 24.4	363.5 ± 47.2
DDP-5	1.40 ± 0.04	1.43 ± 0.06	1124.5 ± 149.9	1241.2 ± 246.7	396.0 ± 57.9	437.2 ± 90.8
DDP-4	1.38 ± 0.02	1.41 ± 0.04	1054.6 ± 60.7	1158.5 ± 165.3	332.3 ± 28.1	365.1 ± 56.8
DDP-3	1.38 ± 0.01	1.40 ± 0.04	1048.5 ± 38.9	1149.2 ± 153.8	377.2± 26.4	413.4 ± 60.6
DDP-2	1.42 ± 0.01	1.45 ± 0.04	1201.8 ± 51.1	1332.3 ± 198.6	472.3 ± 34.5	523.6 ± 84.0
DDP-1	1.53 ± 0.04	1.56	1866.1 ± 330.1	2143.0	679.4 ± 126.9	780

Figures' captions

Fig. 1. (a) Map showing the locations of the Nihewan Basin (red rectangle). (b) DEM map showing the east of the Nihewan Basin, the Sanggan River and the sample locations: Dadaopo (DDP), Donggutuo (DGT), Hutouliang (two localities: HTL-Loc1 and HTL-Loc2), Heshangping (HSP), Hongya (HY), Taiergou (TEG), Majuanpo (MJP), Xiashazui (XSZ), Zhaojiaping (ZJP). Maps were modified from Rui et al. (submitted).

Fig. 2. Stratigraphy and magnetostratigraphy for the DDP section, together with the sample locations and the corresponding MET-pIRIR$_{290}$ ages. As the MET-pIRIR$_{290}$ signal may suffer from fading, the MET-pIRIR$_{290}$ ages are provided as the range between the apparent age (apparent age – 1σ) and the maximum age (maximum age + 1σ). For sample DDP-1 (independent age is 780 ka), the apparent age is 679 ± 127 ka (1σ error). Data of the magnetostratigraphy (blue font) is after Zhu et al. (2004).

Fig. 3. Representative IRSL (50°C) and MET-pIRIR(100–290°C) decay curves for a single aliquot of sample TEG-1, stimulated at different temperatures (shown above each curve).

Fig. 4. Re-normalised MAR L_n/T_n ratios for the samples from the Nihewan Basin, plotted as a function of laboratory dose. Each data point corresponds to one aliquot, with different colours representing different samples. After normalisation to a regenerative dose of 373 Gy, these data points are fitted using a general-order kinetic function, shown by red solid lines. The black dashed lines are the best-fit SGCs obtained from the SAR data; the signal values for a 373 Gy dose value are also normalised to unity.

Fig. 5. Re-normalised SAR (a–f) and MAR residual signals (g–l) at different stimulation temperatures. Each data point corresponds to one aliquot, with different colours representing different samples. These residual signals were fitted using a general-order kinetic function to get the residual SGCs. The black dashed lines in (a–f) and (g–l) are the total SGCs obtained using the SAR and MAR MET-pIRIR procedures, respectively (black and red lines in Fig. 4a–4f, respectively). The residual signals (red solid lines in a–l) were subtracted from the total signals (black dashed lines in a–l) to calculate the bleachable signals (red dashed lines in a–l). The obtained bleachable SAR (black lines) and MAR SGCs (red lines) are compared (m–r) and the 95.4% confidence intervals are shown for the obtained curves (dashed lines).

Fig. 6. Comparison of the 290°C D_e results obtained by the total SAR and MAR SGCs and the bleachable SAR and MAR SGCs for sample HY-3. The natural bleachable signal (open square) is estimated by subtracting the residual signal (filled circle) from the natural signal (total signal, filled square). The SAR and MAR D_e results can be obtained by interpolating the total signal onto the corresponding total SAR and MAR SGCs (black and green curves, respectively). The bleachable SAR

and MAR D_e results can be obtained by interpolating the bleachable natural signal onto the bleachable SAR and MAR SGCs (yellow and blue curves, respectively).

Fig. 7. Re-normalised L_n/T_n ratios for the MET-pIRIR$_{290}$ signals for 7 samples from the DDP section. The CAM ratios (centred on the grey bands) were projected onto the SAR SGC (Fig. 5f) to estimate the D_e values used for age determinations.

Fig. 8. CAM SAR D_e values plotted as a function of infrared stimulation temperatures for the same 7 samples as in Fig. 7. All error bars are at 1σ.

Fig. 9. (a) Anomalous fading results of the IRSL and MET-pIRIR signals from sample DDP-1, where t is the delayed period and t_c is calculated as the time from the middle point of irradiation to the beginning of measurement. The g-values at different stimulation temperatures are summarized in (b).

Fig. 10. Modelled D_e values to paleodose (P) ratios plotted against P. The D_e simulations are based on different percentages (f) of natural signal (L_N) loss due to anomalous fading, including 0.5%, 1.5%, 2.5%, 3.5%, 5% and 7.5%.