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Potential of establishing a 'global standardised growth curve' (gSGC) for optical dating of quartz from sediments

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Potential of establishing a ‘global standardised growth curve’ (gSGC) for optical dating of quartz from sediments

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Abstract

We report investigations of the optically stimulated luminescence (OSL) signals of sedimentary quartz from different regions of Asia, Africa, Europe and North America using a single-aliquot regenerative-dose (SAR) procedure. We show that variations in the shape of dose response curves (DRCs), or growth curves, of the test dose sensitivity-corrected OSL signals among single aliquots composed of multiple grains can be greatly reduced by normalising the DRCs using one of the regenerative dose OSL signals. We refer to this regenerative-dose normalisation procedure as ‘re-normalisation’. We find a common re-normalised DRC extends to doses of ~250 Gy for samples that differ significantly in terms of geological provenance, sedimentary context and depositional age. This feature permits the development of a ‘global standardised growth curve’ (gSGC) for OSL signals from single aliquots of quartz. The equivalent dose (D_e) of an aliquot can be estimated from the natural signal, one regenerative dose signal and their corresponding test dose signals. For the variety of samples investigated, we find that D_e estimates obtained from the gSGC are consistent with those obtained using full SAR procedures for doses of up to ~250 Gy. Use of the gSGC for single aliquots would greatly reduce the time required to estimate the D_e values of older samples and for a large number of aliquots.

Keywords: quartz grains, optically stimulated luminescence, standardised growth curve, dose response curve.

1. Introduction

The single-aliquot regenerative-dose (SAR) procedure ([Galbraith et al., 1999](#); [Murray and Wintle, 2000](#)) has been widely adopted for optically stimulated luminescence (OSL) dating of quartz. In this procedure, the OSL signals induced by stimulating the natural dose (L_n) and each of the regenerative doses (L_x) are followed by measurements of the OSL signal induced by a test dose (T_x). The latter are used to monitor and correct for sensitivity changes associated with the various laboratory treatments, including irradiation, preheating and optical stimulation. The equivalent dose (D_e) is determined by projecting the sensitivity-corrected natural signal (L_n/T_n) on the dose response curve (DRC), or growth curve, constructed from the sensitivity-corrected regenerative dose signals (L_x/T_x).

A decade ago, [Roberts and Duller \(2004\)](#) suggested that T_x not only corrected for within-aliquot sensitivity changes but also acted as a between-aliquot normalisation step, allowing different aliquots from the same or different samples to be compared directly. This offered a potential means of creating a standardised growth curve (SGC), from which the D_e could be estimated based solely on measurements of L_n and T_n . They tested this idea on single aliquots of sand-sized quartz (six dune and beach samples from Tasmania, Australia) and silt-sized polymineral mixtures (five samples from the Chinese Loess Plateau). Their SGC approach was extended subsequently to single aliquots of quartz from different regions, including agricultural and archaeological deposits in Scotland ([Burbidge et al., 2006](#)), loess from Asia, Africa, Europe, and North America ([Lai, 2006](#); [Lai et al., 2007](#)), sand dunes in South Africa and Florida ([Telfer et al., 2008](#)), lacustrine samples from the Qinghai–Tibetan Plateau in China ([Long et al., 2010](#)) and fluvial deposits from the Lower Mississippi Valley in the USA ([Shen and Mauz, 2011](#)). The SGC method offers a useful means of saving on instrument time when D_e estimates are required for a large number of aliquots or for high-resolution dating, with applications to

sand-sized quartz restricted mostly to aliquots with D_e values in the low dose, near-linear region of the growth curve.

In this study, we examine the DRCs of single aliquots of quartz from sedimentary samples from different regions of Asia, Africa, Europe and North America. We show a large between-aliquot variation in the DRCs of the sensitivity-corrected OSL signals from separate aliquots of the same and different samples, and describe a normalisation method that can largely eliminate the inter-aliquot variation among the sensitivity-corrected DRCs. By comparing the normalised and sensitivity-corrected DRCs for a variety of quartz samples, we propose the possibility of constructing a ‘global standardised growth curve’ (gSGC) for single aliquots, providing a rapid means of reliable D_e estimation.

2. Sample descriptions

In this study, 19 sediment samples deposited in a variety of environmental settings in different parts of Asia, Africa, Europe and North America were examined to assess the variability in the DRCs of quartz OSL. Among the samples, 7 are new to this study and 12 are from published studies in which the raw DRC data are provided (see Table 1 for the data sources); for one of the latter samples (MST-19), both the fine-grain and coarse-grain fractions were measured (Timar-Gabor et al., 2012). The sample locations, measurement conditions and D_e ranges (or approximate D_e values) are summarised in Table 1. Sample DC-1 is a fluvial sediment from Shanxi province in China. Sample DGF-1 is a lacustrine sediment taken from the southern edge of the Mu Us Desert (MUD) in central China, and has been studied previously by [Li and Li \(2006\)](#). HLD-3 is a sample of dune sand taken from He Er Hong De in the Hulun Buir Desert (HBD) in northeast China ([Li et al., 2002](#)), and Sm5 is a dune sediment from the Shimao section on the southeastern margin of the Mu Us Desert ([Sun et al., 1999](#); [Li et al., 2011](#)). MS-5450 is a Chinese loess sample collected at Mangshan in Henan, and two additional loess samples, TB-8 and TB-10, originate from Tibet ([Sun et al., 2007](#)). Despite their different geological provenances, the OSL signals of these quartz samples are dominated by the fast

component (Fig. 1), which is the most suitable for the SAR procedure (Galbraith et al., 1999; Wintle and Murray, 2006). Additional data for samples examined in other studies were also investigated (Table 1), including loess samples from China, Germany, Romania and the USA, and fluvial deposits from Morocco and the USA (Lai et al., 2007; Shen and Mauz, 2011; Timar-Gabor et al., 2012).

3. Experimental procedures and analytical facilities

The 7 samples examined as part of this study were prepared for OSL analysis using routine procedures (Aitken, 1998). They were first treated with HCl acid and H₂O₂ solution to remove carbonates and organic matter, respectively, and then dried and sieved to obtain grains of 90–150 and 150–212 µm in diameter (Table 1). Quartz grains were separated from feldspars and heavy minerals using solutions of sodium polytungstate, and then etched with 40% hydrofluoric acid for 45 min before being given a final wash in HCl acid to remove any precipitated fluorides. The mineral grains were mounted on the 10-mm-diameter aluminum discs, using silicone oil as an adhesive, for optical stimulation and measurement. Any residual feldspar contamination of the quartz was checked using the OSL IR depletion ratio (Duller, 2003); none of the measured aliquots showed a significant depletion in the OSL signal following stimulation with IR photons at room temperature.

All measurements were performed on an automated Risø TL/OSL-DA-15 reader equipped with excitation units containing blue light-emitting diodes (470 ± 20 nm at ~ 50 mW/cm²) and IR diodes (830 ± 10 nm at ~ 200 mW/cm²) (Bøtter-Jensen et al., 2000). The OSL signals were detected by an Electron Tubes Ltd 9235B photomultiplier tube fitted with U-340 filters, and laboratory irradiations were carried out using calibrated ⁹⁰Sr/⁹⁰Y beta sources. All OSL stimulations were made at 125°C for 40 s, following a preheat of 260°C for 10s and a cut-heat to 220°C for the regenerative and test doses, respectively; these thermal treatments were chosen on the basis of dose recovery tests, which yielded measured/given dose ratios of 0.94 ± 0.06 for 4 aliquots of sample DC-1, 1.02 ± 0.08 for 6 aliquots of sample DGF-1, 1.05 ± 0.04 for 6 aliquots of sample HLD-3 and 0.98 ± 0.05 for 4 aliquots of sample MS-5450. The resulting OSL signals were calculated from the sum of counts over

the initial 0.4 s of stimulation, with ‘late light’ subtraction (Aitken, 1998) of the background count rate over the final 5 s of stimulation. Table 1 summarises the preheat conditions used for the samples in the other studies, the size of the measured grains and the size of the test doses, the latter ranging from 0.7 to 25.9 Gy.

4. An improved inter-aliquot normalisation procedure

The basic idea of establishing a SGC is that between-aliquot variation can be normalised using T_x . This assumption, however, may not be satisfied for every sample, for reasons that can be demonstrated mathematically. Assuming a single electron trap gives rise to luminescence signal L , (Chen et al., 2001; Wintle and Murray, 2006), the dose response for a specific aliquot or grain (j) can be described using a single saturating exponential function:

$$L_j = S_j(i, D)[N_0 * \left(1 - e^{-\frac{D}{D_0}}\right)] \quad (1)$$

where D is the applied radiation dose, $S_j(i, D)$ is a function of i (the number of the measurement cycle in the SAR sequence) and D , N_0 is the saturation charge population reached at infinite dose ($D = \infty$) and D_0 is the characteristic saturation dose. $S_j(i, D)$ describes the change in luminescence sensitivity (OSL per unit recombining charge) that occurs during measurement of the OSL at various regenerative doses, and is expected to vary from aliquot to aliquot and from grain to grain. Similarly, the test dose signal (T_j) for this aliquot or grain can be described by:

$$T_j = k_j S_j(i, D_t)[N_0 * \left(1 - e^{-\frac{D_t}{D_0}}\right)] \quad (2)$$

where D_t is the test dose and the constant k describes any change in luminescence sensitivity from the natural or regenerative dose step to the test dose measurement. It then follows that the sensitivity-corrected luminescence signal (L_j/T_j) is given by

$$L_j/T_j = \left(1 - e^{-\frac{D}{D_0}}\right) / [k_j \left(1 - e^{-\frac{D_t}{D_0}}\right)] \quad (3)$$

As shown from eqn. 3, the sensitivity-corrected luminescence signal for a specific aliquot or grain (L_j/T_j) is dependent on D_t , D_0 and k_j . Because the size of the test dose will affect the size of the test dose response (T_j), Roberts and Duller (2004) suggested that the sensitivity-corrected luminescence signal (L_j/T_j) can be adjusted for the size of the test dose (D_t) to produce a ‘standardised luminescence signal’ ($(L_j/T_j)*D_t$). This test-dose normalisation method assumes that the test dose is kept within the linear region of the DRC (i.e., the test dose is much smaller than the D_0 value). As a result, eqn. 3 can be simplified to:

$$L_j/T_j = \frac{\left(1 - e^{-\frac{D}{D_0}}\right)}{k_j \frac{D_t}{D_0}} = \frac{D_0}{k_j D_t} \left(1 - e^{-\frac{D}{D_0}}\right) \quad (4)$$

It follows that different aliquots and grains will have similar test dose-corrected OSL signals $[(L/T)*D_t]$ if they share the same D_0 and k values. For young samples with D_e values in the linear region of the DRC (i.e., D is much smaller than D_0), the function of their test dose-corrected signal can be further simplified to:

$$\frac{L}{T} D_t = \frac{D}{k} \quad (5)$$

In eqn. 5, the form of test dose-corrected signal $[(L/T)*D_t]$ is dependent only on the k value, so similar DRCs should be observed for aliquots and grains that have similar k values (e.g., Roberts and Duller, 2004). This requirement may not be satisfied, however, because different grains may have experienced different irradiation, bleaching and thermal histories, and these factors may influence the extent of sensitivity change (i.e., the k value) induced by laboratory irradiation, preheating and bleaching procedures (e.g., Murray and Wintle, 1999a, 1999b; Chen et al., 2001). As a result, the similarity in shape of DRCs for different aliquots and grains depends critically on the between-aliquot and between-grain variations in the k values at low doses (eqn. 5), and on variations in both the k and D_0 values at higher doses (eqn. 4). Single grains of quartz commonly exhibit large variations in D_0 , resulting in DRCs that differ markedly in shape, even at quite low doses (e.g., Roberts et al., 1999;

Jacobs et al., 2003, 2013). For single aliquots, where tens, hundreds or thousands of grains are measured simultaneously, grain-averaging effects will reduce the variability in D_0 values and DRC shapes.

Burbidge et al. (2006) recommended that one additional regenerative dose response should be measured to check the validity of SGC, and Telfer et al. (2008) reiterated this recommendation. Rather than using the method proposed by Burbidge et al. (2006), we propose here a method to reduce the effect of between-aliquot variation in k values for normalising the DRCs of single aliquots. We achieve this by standardising the DRCs using one of the regenerative dose signals – that is, by dividing the sensitivity-corrected natural (L_n/T_n) and regenerative-dose (L_x/T_x) signals in each SAR cycle by the signal (L_{r1}/T_{r1}) of the regenerative dose (D_{r1}) chosen for normalisation. We refer to this procedure as ‘regenerative-dose normalisation’ or ‘re-normalisation’. Based on eqn. 3, it follows the re-normalised sensitivity-corrected OSL signal can be expressed as:

$$\frac{L_x/T_x}{L_{r1}/T_{r1}} = \left(1 - e^{-\frac{D}{D_0}}\right) / \left(1 - e^{-\frac{D_{r1}}{D_0}}\right) \quad (6)$$

From eqn. 6, it can be seen that re-normalisation produces a DRC independent of k and, hence, independent of the irradiation, bleaching and thermal histories of the grains comprising the different aliquots. We expect, therefore, that the re-normalisation procedure should reduce the between-aliquot variation in DRC shape to a greater extent than is achieved using the SGC method of Roberts and Duller (2004).

We recognise that the re-normalisation method cannot eliminate all inter-aliquot variation in DRC shape, because eqn. 6 is based on the assumption that k is independent of the preceding dose, and this may not be true. Using the 110°C TL peak as a monitor of OSL sensitivity change (Murray and Roberts, 1998), Murray and Wintle (2000) found that sensitivity changes between the natural or regenerative dose step and the test dose measurement (i.e., the k value) were dose-dependent for their quartz samples. They also suggested, however, that this dependency did not influence the accuracy of

D_e estimation using the sensitivity-corrected OSL signal (Wintle and Murray, 2006), so any dose dependency in k should also not affect the accuracy of D_e estimation based on re-normalised DRCs.

To test whether the re-normalisation method can improve the standardisation of DRC shapes for different aliquots of the same sample, we compared the DRCs of six single aliquots of quartz from sample Sm5 (Fig. 2a). The sensitivity-corrected OSL signals (L_x/T_x) show a large inter-aliquot scatter (filled diamonds, left-hand scale of Fig. 2a), which is reduced significantly (open circles, right-hand scale) when these data are re-normalised using the regenerative dose signal at 172 Gy (indicated by the arrow). The relative standard deviation (RSD) in L_x/T_x ranges from 14% to 21% at regenerative doses of 86, 172, 258 and 344 Gy, and is reduced to 5–8% after re-normalisation. The same trend is observed for the sensitivity-corrected natural signals (L_n/T_n), which are plotted on the y-axis: the RSD in L_n/T_n is 29%, decreasing to 18% after re-normalisation using the 172 Gy signal.

Fig. 2b–f show similar comparative plots of the DRCs for five other samples (HLD-3, TB-8, DC-1, DGF-1 and MS-5450). Four of these (HLD-3, TB-8, DC-1 and DGF-1) exhibit a similar pattern to Sm5, namely a reduced inter-aliquot scatter among the natural and regenerative dose signals after applying the re-normalisation procedure. By contrast, sample MS-5450 shows no obvious improvement after re-normalisation (Fig. 2f). The latter sample has a much smaller inter-aliquot variation in L_x/T_x ratios than the other samples examined, which suggests that these aliquots vary much less in their k values. Accordingly, T_x is sufficient to reduce the inter-aliquot variations for this particular sample, and re-normalisation offers limited additional benefit.

We note that the magnitude of reduction in scatter of the L_n/T_n values depends on which regenerative dose signal is used for re-normalisation. Using the data of Sm5 (Fig. 2a) as an example, Fig. 3 illustrates how the choice of re-normalisation signal affects the extent of inter-aliquot variation. The RSD in the L_n/T_n ratios (plotted on the y-axis) is highest before re-normalisation and is lowest using the regenerative dose signal at 86 Gy or 172 Gy for re-normalisation. These two doses are closest to the sample D_e (Fig. 2a), and similar results were observed for the other samples. Optimum

results were generally obtained using the regenerative dose signal that matched most closely the intensity of the natural signal (indicated by arrows in Fig. 2), which we attribute to the reduction in scatter associated with any dose dependency of k .

As eqn. 6 is independent of k , another implication is that re-normalisation should be independent of the experimental treatments applied to the regenerative and test doses. We tested this proposition on sample DGF-1 by changing the preheat and cut-heat temperatures for the regenerative and test doses, respectively, and by varying the size of test dose (Fig. 4). The test dose-corrected signals $[(L_x/T_x)*D_t]$ are plotted against regenerative dose in Fig. 4a, with the natural signals shown on the y-axis. The between-aliquot scatter is large, suggesting that these different measurement conditions may give rise to variations in k . The scatter is greatly reduced after re-normalisation using the regenerative dose signal at 86 Gy and, moreover, no differences can be detected between aliquots that received different treatments. This suggests that the re-normalisation procedure can potentially generate a standardised shape for the quartz DRCs of separate aliquots of the same sample under a variety of measurement conditions.

5. Comparing single-aliquot DRCs for different quartz samples

For the Chinese samples examined thus far, the reduction in inter-aliquot variation achieved using the re-normalisation procedure (Fig. 2–4) indicates that between-aliquot variations in k can be reduced substantially by selecting one of the regenerative dose signals for re-normalisation. We now investigate the extent to which the re-normalised DRCs for these samples are similar in overall shape.

In Fig. 5a, the test dose-corrected signals $[(L_x/T_x)*D_t]$ are plotted against regenerative dose for each aliquot, revealing a large scatter in values among individual aliquots of the same and different samples; this confirms our earlier contention that T_x cannot account sufficiently for the observed variations in these samples. Fig. 5b shows the re-normalised data sets, in which the L_x/T_x ratios for individual aliquots have been multiplied by a scaling factor, such that the regenerative dose DRC for each sample passes through a re-normalised L_x/T_x value of unity at 172 Gy; this is equivalent to

normalising each data set using the regenerative dose signal at 172 Gy. The value of 172 Gy was chosen because it lies in the middle of the applied dose range (0–344 Gy), thereby providing a check on the efficacy of re-normalisation at both lower and higher doses. There is a significant reduction in inter-aliquot and inter-sample variation after this re-normalisation, with the DRCs of the different samples showing a very similar pattern of growth with dose up to ~350 Gy. This suggests that between-sample variations in k have been adequately corrected for and that these samples share a similar characteristic saturation dose (D_0).

We next explored whether these features are common to quartz samples in other regions also, using published data on sediments from Africa, Europe and North America (Table 1, Fig. 6). These studies reported only the average L_x/T_x signal for a group of aliquots, rather than the data for individual aliquots, so we are restricted to making comparisons of the sample-average results. In addition, the exact L_x/T_x values were not reported for the two grain-size fractions of the Romanian sample (MST-19 (FG) and MST-19 (CG); [Timar-Gabor et al., 2012](#)) or for the ‘synthetic’ sample TNE from Australia (created from 27 aliquots of six samples; [Roberts and Duller, 2004](#)). The DRCs for these samples are shown by dotted and dashed lines in Fig. 6, based on the curve-fitting parameters provided by the authors. For sample TNE, however, we restricted DRC construction to a maximum dose of up to 125 Gy, as this was the dose limit used by [Roberts and Duller \(2004\)](#) to estimate the curve-fitting parameters. For the samples examined by [Lai \(2006\)](#), [Lai et al. \(2007\)](#) and [Shen and Mauz \(2011\)](#), and for the 7 samples measured for this study, the data sets are based on the average of six aliquots, with the associated standard deviations shown as error bars in Fig. 6.

For these samples, we observed a large variation in the test dose-corrected signals $[(L_x/T_x)*D_t]$ at different regenerative doses (Fig. 6a), suggesting that T_x cannot account sufficiently for the observed variations in these samples. One possible cause of this variation is the wide range in size of the test doses (0.7–25.9 Gy; Table 1), which may give rise to a test dose dependency in $[(L_x/T_x)*D_t]$ ([Shen and Mauz, 2011](#)). However, we cannot find any obvious correlation between the size of the test dose and

the trend of the DRCs in Fig. 6a, so we suggest instead that the DRC variations in Fig. 6a are due mainly to between-sample variability in the k and D_0 values. The single-aliquot DRCs of the test dose-corrected signals from each sample were fitted using a single saturating exponential function $Y = A[1 - \exp(-X/D_0)] + Y_0$, where Y is the test dose-corrected signal, X is the regenerative dose, D_0 is the characteristic saturation dose, and A and Y_0 are constants. The corresponding D_0 values are summarised in Table 1. We obtained a wide range of D_0 values (from about 55 to 250 Gy), but most of the D_0 values are between 100 and 150 Gy.

If the data sets in Fig. 6a are re-normalised to unity at a regenerative dose of 172 Gy (i.e., as in Fig. 5b), then the between-sample variation is reduced significantly and, except for the fine-grain quartz sample MST-19 (FG) from Romania, the re-normalised DRCs are similar in shape up to a dose of ~250 Gy (Fig. 6b,c). Above 250 Gy, the coarse-grain and fine-grain data sets for the Romanian sample (MST-19) appear to diverge from the general line of best fit to the other samples (Fig. 6b), but we note that the coarse-grain fraction follows the trend line much more closely than does the fine-grain fraction at doses below 250 Gy (Fig. 6c). The similarity in DRC shapes observed in Fig. 6b and 6c suggests that inter-sample variation in k can be corrected for using the re-normalisation procedure, at least for doses of up to ~250 Gy. At higher doses, inter-sample variations in D_0 values or sample-specific saturation limits are likely to play an increasingly important role.

From the results shown in Fig. 6b and 6c, there appear to be good prospects of establishing a ‘global standardised growth curve’ (gSGC) to ~250 Gy for quartz samples from different regions by using the re-normalisation procedure. The data in the 0–250 Gy dose range are well described by a single saturating exponential function of the form $Y = A[1 - \exp(-X/D_0)]$, for which the best-fit D_0 value is 110 Gy, and by a single saturating exponential function plus linear component (shown as the solid black line in Fig. 6b,c). The presence of such a linear component in the high-dose response of quartz OSL for some samples is not yet fully understood. As it may be a laboratory artefact, rather than reflecting the true dose response of quartz in the natural environment, it has been suggested that

the D_e values obtained from this region should be treated with caution in the absence of independent age control (e.g., Lai, 2010; Lowick and Preusser, 2011). Table 2 lists the best-fit parameters for the latter function when applied to the re-normalised data sets of all samples (excluding the Romanian sample) in Fig. 6b (0–450 Gy) and Fig. 6c (0–250 Gy).

6. D_e estimation using the global standardised growth curve

A gSGC for quartz OSL should facilitate the rapid and reliable estimation of D_e from a large number of single aliquots, and it differs in only one significant respect from the SGC method used by previous studies (e.g., Roberts and Duller, 2004; Lai, 2006; Lai et al., 2007): the re-normalisation method requires a regenerative dose OSL signal (and its corresponding test dose signal) to be measured in addition to the sensitivity-corrected natural signal (L_n/T_n), which is projected on to the L_x/T_x SGC to estimate the D_e . As a result, the D_e for a single aliquot of quartz can be determined from its natural signal (L_n), one regenerative dose signal (L_{r1}), their corresponding test dose signals (T_n , T_{r1}), and the gSGC.

As demonstrated in Fig. 3, the chosen regenerative dose should be as close as possible in size to that of the natural dose to minimise the scatter in the re-normalised L_n/T_n signals. It may be necessary, therefore, to choose alternative regenerative doses for different samples, resulting in a variety of re-normalisation factors. The latter can be related to the gSGC as follows:

$$\eta \frac{L}{T} = f(D) \quad (7)$$

where $f(D)$ is the gSGC function (as parameterised in Table 2) and η is the re-normalisation factor (which differs from aliquot to aliquot). The L_n/T_n and L_{r1}/T_{r1} ratios for each aliquot can then be expressed as:

$$\eta \frac{L_n}{T_n} = f(D_e) \quad (8)$$

and

$$\eta \frac{L_{r1}}{T_{r1}} = f(D_{r1}) \quad (9)$$

where D_{r1} is the regenerative dose chosen for re-normalisation.

The re-normalisation factor η can, thus, be determined for each aliquot as:

$$\eta = \frac{f(D_{r1})}{L_{r1}/T_{r1}} = \frac{f(D_e)}{L_n/T_n} \quad (10)$$

Since all of the parameters in this equation (i.e., $f(D)$, D_{r1} , L_n/T_n and L_{r1}/T_{r1}) are known, the D_e can be calculated by solving eqn. 10 using an iterative method.

Here we provide a worked example using the data for a single aliquot of sample HLD-3. Based on the best-fitting parameters for the 0–250 Gy data sets in Fig. 6c (see Table 2), the function of the gSGC is $f(D) = 0.787[1 - \exp(-D/73.9)] + 0.001539D + 0.01791$. Eqn. 10 is thus:

$$\frac{0.787[1 - \exp(-D_{r1}/73.9)] + 0.001539D_{r1} + 0.01791}{L_{r1}/T_{r1}} = \frac{0.787[1 - \exp(-D_e/73.9)] + 0.001539D_e + 0.01791}{L_n/T_n} \quad (11)$$

For the specific aliquot chosen as an example, the values of L_n/T_n and L_{r1}/T_{r1} are 2.361 ± 0.087 and 2.744 ± 0.091 , respectively, using a regenerative dose (D_{r1}) of 34.4 Gy for re-normalisation.

Substituting the values of D_{r1} , L_n/T_n and L_{r1}/T_{r1} into eqn. 11, and then solving it iteratively, gives a gSGC D_e value of 28.4 ± 1.3 Gy, which is consistent with the D_e of 27.3 ± 1.3 Gy obtained using the full SAR procedure for this aliquot.

To test the reliability of D_e estimation using the gSGC shown by the solid black line in Fig. 6c, 56 individual aliquots from the 7 Chinese samples measured in this study (Table 1) were analysed as described above, and the D_e values compared with those obtained using the full SAR procedure for each aliquot. All of the aliquots measured for these samples passed the standard tests of acceptable SAR performance – that is, negligible recuperation (<5%) and a mean recycling ratio consistent with unity (1.01 ± 0.01). The parameters of the best-fitting saturating exponential plus linear gSGC (0–250 Gy data in Table 2) were combined with the aliquot-specific variables in eqn. 11 to estimate the gSGC

D_e value for each aliquot. These values are plotted against the individual SAR D_e values in Fig. 7a with the data up to 250 Gy shown as filled symbols and the data >250 Gy shown as open symbols. The results shows that the D_e estimates obtained from both methods are consistent at 2σ , with just one exception. The sensitivity-corrected natural signal of the latter aliquot (from sample Sm5) lies above the sensitivity-corrected intensity of the largest applied regenerative dose (344 Gy), so the D_e was estimated by extrapolation, which may account for this discrepancy. Furthermore, the extrapolated D_e value of ~394 Gy for this aliquot is significantly greater than 250 Gy, which is the maximum dose used to derive the fitting parameters of the gSGC.

The concordance between the D_e values of the two methods for 55 of the 56 aliquots is further demonstrated in Fig. 7b, which plots the ratios of the gSGC and SAR D_e values against the corresponding SAR D_e values; the same data are displayed in a radial plot in Fig. 7c. The statistical consistency of these ratios (i.e., 98% within 2σ of unity) suggests that the gSGC method can produce reliable estimates of D_e up to ~250 Gy, and possibly to higher doses. We note that the gSGC D_e estimates have smaller analytical uncertainties than their SAR D_e counterparts, especially at higher doses. This slight increase in precision reflects the large number of data points used to constrain the fitting parameters of the gSGC, compared to the few data points used to fit the DRC for any individual aliquot. However, for samples with growth curves that deviate significantly from the gSGC, D_e values estimated from the latter may not be accurate.

7. Discussion

Equation 3 shows that the sensitivity-corrected regenerative dose OSL signals (L_x/T_x) measured in the SAR procedure are dependent on two factors: the k and D_0 values. The former is determined by sensitivity changes occurring between the natural or regenerative dose step and the measurement of the test dose signal in the same SAR cycle. The existence and extent of such sensitivity changes are expected to be sample-specific and contingent on the particular measurement conditions, for the reasons given below.

In the SAR procedure, several laboratory treatments are administered to an aliquot between the measurements of L_x and T_x , including optical stimulation, irradiation and preheating. These experimental conditions may influence the k value and, hence, the shape of the DRC. For example, OSL stimulation may de-sensitise the sample due to the recombination of luminescence centres with electrons excited from the OSL traps, reducing the number of hole centres subsequently available for radiative recombination (e.g., Bailey et al., 1997; Wintle and Murray, 1997; Bailey, 2001). In addition, irradiation may change the relative concentrations of luminescence centres and ‘killer’ (non-radiative) centres, causing dose quenching of sensitivity (Zimmerman, 1971). Furthermore, preheating may play an important role in changing the sensitivity of quartz OSL (Roberts et al., 1994; Stokes, 1994; Murray and Wintle, 1999b; Wintle and Murray, 1999). The k value may, thus, be influenced by the experimental treatments applied ahead of the measurement of T_x .

A further potential complication is that even if identical SAR procedures and measurement conditions are applied systematically to different samples, they may respond differently in terms of changes in sensitivity (k). Such differences are to be expected, because not all grains on an aliquot will share the same irradiation, bleaching or thermal histories, and each of these may affect the luminescence properties (and, hence, the k value). Fortunately, as demonstrated in principle by eqn. 6 and in practice by the data in Fig. 2 and 3, between-aliquot variations in k can be reduced substantially by applying a normalisation using one of the regenerative dose signals. Re-normalisation cannot eliminate the uncertainty associated with any dose dependency in $\underline{k}$, but the latter is thought to have no influence on D_e estimation (Murray and Wintle, 2000; Wintle and Murray, 2006).

In addition to reducing the extent of variation in the DRCs for individual aliquots of the same sample, we have demonstrated that re-normalisation can also reduce the variation in DRCs between samples (Fig. 5 and 6). This allows the direct comparison of DRCs for different samples, even though they were measured in different laboratories using a range of experimental conditions (Table 1). The re-normalised DRCs of these samples have similar shapes up to a dose of ~250 Gy, beyond which the

effects of saturation become more important, resulting in divergence of the re-normalised DRCs for one of the samples (MST-19). It has been demonstrated previously that the characteristic saturation dose (D_0) of quartz OSL is strongly influenced by thermal treatment (Chen et al., 2001), and its value may be reduced significantly by heating to a high temperature (Lai et al., 2008; Gong et al., 2014). But the behaviour of the quartz OSL signal in the high-dose range remains poorly understood, including whether the linear component is a laboratory artefact or a meaningful physical property (e.g., Lowick and Preusser, 2011; Timar-Gabor et al., 2012). Based on these considerations, we do not recommend using the SGC method to estimate D_e values greater than ~ 250 Gy.

The establishment of a gSGC may greatly benefit OSL dating of quartz, in view of its compensating effect for inter-aliquot and inter-sample differences in k and D_0 values, and will be especially useful for studies that require a large number of independent, single-aliquot estimates of D_e . Because the re-normalisation method underpinning the gSGC is less dependent on the laboratory conditions used for measuring the OSL signal (Fig. 4) and reduces inter-aliquot variations to a much greater extent than the SGC method proposed by Roberts and Duller (2004), it should yield more precise and accurate estimates of D_e . This is true for the samples examined here, as demonstrated in Fig. 8a: this shows the same data as displayed in Fig. 7a, but with SAR D_e values plotted against with the corresponding SGC D_e estimates (obtained by projecting the L_n/T_n signal of each aliquot on to the respective sample-average L_x/T_x DRC). The corresponding D_e ratios are presented in Fig. 8b,c. These plots reveal that, unlike the gSGC data in Fig. 7, large deviations can occur from a D_e ratio of unity, even for aliquots of the same sample using the sample-average L_x/T_x DRC. These results are much poorer than those reported in previous studies (e.g., Roberts and Duller, 2004; Lai et al., 2007), which we attribute to the less homogeneous inter-aliquot behaviour of the samples investigated here.

By reducing inter-aliquot and inter-sample differences in DRC shape using the re-normalisation procedure, it is feasible to tentatively construct a globally applicable SGC (Fig. 6). Clearly, further work is required to validate the gSGC developed here. The measurement time

required to estimate D_e values using the gSGC is significantly less than that needed to make full SAR analyses. When making these measurements, we recommend that a regenerative dose close to the natural dose is chosen, to reduce the extent of between-aliquot variation in the re-normalised results (Fig. 3).

Furthermore, following the original suggestion by Roberts and Duller (2004) that a limited number of aliquots could be analysed using a full SAR procedure to confirm whether application of a SGC is appropriate, we recommend that the complete SAR procedure should be applied to a few aliquots (4–6, say) from each sample or site, and the resultant DRCs compared against the gSGC to verify its suitability to estimate D_e values for the remaining aliquots. If a sample yields a re-normalised DRC that deviates significantly from the gSGC described here – for example, the fine-grain fraction of sample MST-19 (Fig. 6c) – then it may be necessary to estimate the D_e values for the remaining aliquots from measurements of L_n , T_n , L_{r1} and T_{r1} and the re-normalised DRC specific to that sample, using eqn. 10.

By examining some aliquots using the full SAR procedure – including routine tests for IR depletion of the OSL signal, recycling ratio and recuperation – each sample can be checked for its luminescence behaviour and SAR performance. Satisfactory outcomes of these tests can be used as acceptance criteria to then apply the gSGC method to the remaining aliquots with confidence. Incorporating such tests directly in the gSGC procedure would substantially increase the measurement time and, thus, diminish one of the chief benefits of the gSGC approach.

The OSL signals of the samples investigated in this study are dominated by the fast component (see Fig. 1 and the references in Table 1), so we expect the gSGC established here to be applicable only to other such samples. The DRC shapes of other components of quartz OSL may be significantly different; for example, the slow components may saturate at much higher doses (Singarayer et al., 2000; Singarayer and Bailey, 2003). The ultrafast and medium components of the OSL signal may also exhibit some undesirable properties, such as thermal instability, and these can

also adversely influence the accuracy of D_e estimates (e.g., [Li and Li, 2006](#); [Jain et al., 2008](#)). As a result, the existence of a common DRC among different samples does not necessarily imply that reliable age estimates will be obtained. Hence, dose recovery tests ([Roberts et al., 1999](#)) and age checks against independently dated samples need to be performed to demonstrate that a series of known doses can be accurately determined from the re-normalised DRC.

Finally, the aliquots measured in this study each consisted of a few hundred to a few thousand quartz grains, depending on the grain size used. Between-grain differences in luminescence behaviour, such as large variations in DRC shape and D_0 values (e.g., [Roberts et al., 1999](#); [Jacobs et al., 2003, 2013](#)), will be homogenised in multi-grain aliquots. While this offers benefits in terms of enabling a gSGC to be constructed for single aliquots, the use of multi-grain aliquots sacrifices the advantages of single-grain dating (e.g., [Jacobs and Roberts, 2007](#); [Duller, 2008](#)). Future research could usefully examine whether the re-normalisation and gSGC procedures can be extended to individual grains of quartz from the same and different samples. The application of these procedures to single-grain OSL dating would maximise the benefits of gSGC construction, greatly reducing the amount of instrument time needed to measure each sample and, thereby, making it practicable to determine D_e values for a much larger number of individual grains. If it is found, however, that single grains are too variable in their OSL behaviours to follow a gSGC, then it would be instructive to determine the smallest aliquot size that can satisfactorily homogenise these grain-to-grain differences.

8. Conclusions

Single aliquots of sedimentary quartz commonly exhibit large variations in the shape of the DRCs constructed from the sensitivity-corrected regenerative dose signals (L_x/T_x). This variability can arise from inter-aliquot variation in the extent of sensitivity change between the natural or regenerative dose step and the subsequent test dose measurement in the same SAR cycle. We have shown in this paper that such variation can be reduced greatly by applying a normalisation procedure using one of the regenerative dose OSL signals ('re-normalisation'). For a range of quartz samples

from different regions of Asia, Africa, Europe and North America, we construct a common re-normalised DRC up to a dose of ~250 Gy, which we tentatively propose as a ‘global standardised growth curve’ (gSGC) for single aliquots of quartz. Equivalent doses can be estimated for other samples using the fitting parameters for the gSGC developed here, together with measurements of the natural signal (L_n), one regenerative dose signal (L_{x1}) and the corresponding test dose signals (T_n , T_{x1}) for each aliquot. When compared to the construction of a full DRC for each aliquot using conventional SAR procedures, the re-normalisation and gSGC procedures described here require much less instrument time, thereby enabling the measurement of D_e values for many more aliquots of each sample.

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Figure captions

Figure 1: Natural OSL decay curves for single aliquots of quartz from six Chinese samples. All curves are normalised to unity at $t = 0$ for comparison. The quartz grains were preheated at 260°C for 10 s and then stimulated at 125°C using blue LEDs. The inset plot shows that the natural signal of DGF-1 is dominated by the fast component of quartz OSL. Three exponential decay functions were fitted to the decay curve to determine the relative contributions of the fast, medium and slow (plus background, BG) components. The other samples are similarly dominated by the fast component.

Figure 2: The sensitivity-corrected natural (L_n/T_n , marked as N) and regenerative dose (L_x/T_x) signals (both plotted on the left-hand axis) and their re-normalised counterparts (right-hand axis). The latter were calculated using the re-normalisation procedure described in the text, in which one of the L_x/T_x signals (indicated by the arrow) is chosen for each of the samples. Note that the re-normalised data (circles) are offset by a few Gy to the right on the x -axis for clarity.

Figure 3: The relative standard deviation (RSD, the standard deviation divided by the mean) for the sensitivity-corrected (no normalisation) and re-normalised L_x/T_x signals of sample Sm5, plotted against the corresponding regenerative doses. The regenerative dose used for normalisation of each data set is shown in the legend. The RSD for the sensitivity-corrected natural (L_n/T_n) signal is shown on the y -axis. The RSD values at zero regenerative dose are not shown; they are high ($\sim 50\%$) due to the low absolute ratios of L_x/T_x at 0 Gy, but the absolute standard deviations are negligible compared to those at higher regenerative doses.

Figure 4: (a) The test dose-corrected signals $[(L_x/T_x)*D_t]$ for sample DGF-1 measured under various experimental conditions, plotted against the corresponding regenerative doses; the test dose-corrected natural signals $[(L_n/T_n)*D_t]$ are shown on the y -axis and marked as N. The legend indicates the preheat/cut-heat temperatures (in $^{\circ}\text{C}$) applied to the regenerative and test doses, respectively, followed by the size of the test dose. (b) The same data as in (a) after re-normalisation.

Figure 5: (a) The test dose-corrected signals for various samples from China (6 aliquots of each), plotted against the corresponding regenerative doses. (b) The re-normalised L_x/T_x signals obtained by multiplying

each of the data sets in (a) so that they pass through a value of unity at 172 Gy. This is equivalent to re-normalising each data set using the regenerative dose signal at 172 Gy. The dashed line shows the best-fit curve using a saturating exponential function plus a linear component.

Figure 6: (a) The test dose-corrected signals for various samples from different regions of the world (Table 1; the average and standard deviation of 6 aliquots is shown for each sample), plotted against the corresponding regenerative doses. (b) The re-normalised L_x/T_x signals obtained by multiplying each of the data sets in (a) so that they pass through a value of unity at 172 Gy. The dotted and dashed lines are the best-fit curves to the data of MST-19 (FG) and MST-19 (CG) (Timar-Gabor et al., 2012) and ‘synthetic’ sample TNE (Roberts and Duller, 2004), as provided in the original papers. (c) The same data sets as shown in (b), but restricted to the 0–250 Gy dose range. The solid black lines in (b) and (c) are the best-fit curves determined using a saturating exponential function plus a linear component (Table 2 lists the corresponding fitting parameters).

Figure 7: (a) D_e estimates for 56 single aliquots of the 7 Chinese samples measured in this study (Table 1). D_e values were estimated in two ways: from the gSGC in Fig. 6b (gSGC D_e) and from construction of full SAR growth curve for each aliquot (SAR D_e). (b) The ratios of the gSGC and SAR D_e values plotted against the latter. (c) A radial plot of the same data as shown in (b). The data up to 250 Gy are shown as filled symbols, and those >250 Gy as open symbols, in each plot.

Figure 8: (a) The same data as shown in Fig. 7a, but with the D_e values estimated from the individual SGC curve specific to each sample (i.e., the average L_x/T_x DRC for a particular sample), plotted against the SAR D_e for each aliquot. (b) The ratios of the individual L_x/T_x SGC and SAR D_e values plotted against the latter. (c) A radial plot of the same data as shown in (b). The data up to 250 Gy are shown as filled symbols, and those >250 Gy as open symbols, in each plot.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Table 1: Summary of study samples, including their geographic locations, depositional environments, and the grain size and measurement conditions used to determine the equivalent dose (D_e) values. The ranges of D_e values estimated using individual SAR DRCs are listed for the 7 Chinese samples measured in this study, and the approximate D_e values (if reported by the original authors) are listed for the other samples. Shen and Mauz (2011) did not provide D_e values for their samples, so approximate D_e values have been estimated from the sensitivity-corrected natural (L_n/T_n) and regenerative dose (L_x/T_x) ratios provided by the authors. Data on OSL sensitivity to a test dose (in counts per Gy) are provided only for the samples measured in this study and were obtained from 6 aliquots of each sample.

Sample	Location	Sediment type	Grain size (μm)	Preheat/cut-heat ($^{\circ}\text{C}$)	Test dose (Gy)	Test dose sensitivity (cts/Gy)	D_e (Gy)	D_0 (Gy) ^a	Data source
DC-1	Shanxi, China	Fluvial	150–212	260/220	24.3	1563–3079	100–250	114 ± 8	This work
DGF-1	Mu Us Desert, China	Lake	90–150	260/220	8.6	87–200	130–180	116 ± 18	This work
HLD-3	Hulun Buir Desert, China	Dune	90–150	260/220	8.6	251–1439	27–40	- ^a	This work
MS-5450	Henan, China	Loess	90–150	260/220	24.3	64–270	96–340	124 ± 9	This work
Sm5	Mu Us Desert, China	Dune	90–150	260/220	25.8	121–205	100–394	138 ± 15	This work
TB-8	Tibet, China	Loess	90–150	260/220	8.5	175–260	62–104	- ^a	This work
TB-10	Tibet, China	Loess	90–150	260/220	17.2	31–100	>300	118 ± 10	This work
LV173	Mississippi Valley, USA	Fluvial	11–15	220/180	23.7	-	~500	174 ± 18	Shen and Mauz, 2011
LV174	Mississippi Valley, USA	Fluvial	11–15	220/180	11.6	-	~130	141 ± 16	Shen and Mauz, 2011
LV290	Mississippi Valley, USA	Fluvial	4–11	220/180	7.4	-	~400	168 ± 16	Shen and Mauz, 2011
LV296	Mississippi Valley, USA	Fluvial	4–11	220/180	3.6	-	~280	189 ± 21	Shen and Mauz, 2011
MST-19 (CG)	Romania	Loess	63–90	220/180	17	-	288 ± 57	142 ± 11	Timar-Gabor et al., 2012
MST-19 (FG)	Romania	Loess	4–11	220/180	17	-	289 ± 9	250 ± 8	Timar-Gabor et al., 2012
LC-1	Shanxi, China	Loess	38–63	260/220	25.9	-	~27	88 ± 5	Lai, 2006
NWS-2-4	Germany	Loess	38–63	260/220	11.3	-	-	98 ± 9	Lai et al., 2007
OWRB50	Wauneta, USA	Loess	38–63	260/220	11.3	-	-	105 ± 7	Lai et al., 2007
Wach2	Wach, USA	Loess	38–63	260/220	11.3	-	-	130 ± 11	Lai et al., 2007
BN top	Bignell Hill, USA	Loess	38–63	260/220	11.3	-	-	139 ± 13	Lai et al., 2007
ESS-10/3	Morocco	Fluvial	38–63	260/220	11.3	-	-	85 ± 5	Lai et al., 2007
TNE ^b	Tasmania, Australia	Dune	180–212	160/160	0.7–4	-	-	55 ± 1	Roberts and Duller, 2004

^a D_0 values were obtained by fitting the average dose response curves for each sample using a single saturating exponential function. The maximum regenerative doses given to samples HLD-3 and TB-8 were too small (<80 Gy) to reliably estimate their D_0 values.

^b TNE is a ‘synthetic’ sample based on 27 aliquots from 6 samples (Roberts and Duller, 2004). Data for the individual samples were not provided by the authors.

Table 2: Summary of the best-fitting parameters for the data sets in Fig. 6b (0–450 Gy) and Fig. 6c (0–250 Gy), using a saturating exponential function plus a linear component ($Y = A[1 - \exp(-X/D_0)] + cX + Y_0$).

Signal	Fitting data of 0–450 Gy	Fitting data of 0–250 Gy
A	0.723 ± 0.014	0.787 ± 0.051
D_0	65.1 ± 0.9	73.9 ± 2.2
c	0.001784 ± 0.000016	0.001539 ± 0.000068
Y_0	0.009159 ± 0.004795	0.01791 ± 0.00490