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Abstract

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Keywords

OSL dating, luminescence dating, fast component, curve fitting, deconvolution, CAS

Disciplines

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Optically stimulated luminescence dating of young quartz using the fast component

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Abstract

We have attempted to isolate the fast component of the quartz Optically Stimulated Luminescence (OSL) signal using a curve fitting procedure. By pre-determining the decay constants, the procedure is simple enough to be scripted, allowing a large number of aliquots to be processed. A Monte Carlo error routine is used, in which simulated decay curves are fitted with several exponentials, which vary in their decay rates according to the measured distributions of fast and medium component decay rates. The derived error term is closely related to the intensity of the fast component signal, but is also influenced by the degree of similarity between the equivalent doses of the fast and medium OSL components. There are potential advantages in using this procedure to date both well-bleached and partially bleached quartz, of any depositional age.

1. Introduction

Since the introduction of the single aliquot regenerative dose (SAR) protocol in the late 1990's, the optically stimulated luminescence (OSL) signal of quartz has been widely used to date Late Quaternary sediments. OSL is particularly useful for dating young sediments (those less than 1000 years old), due to the lack of alternative dating methods: plateaux in the radiocarbon calibration curve make radiocarbon difficult to use on

samples less than c.500 years old, even if suitable organic material can be found; ^{210}Pb and ^{137}Cs have upper age limits of 120 and 50 years respectively, and are restricted to fine-grained deposits. Given the near ubiquity of quartz (or feldspar) in sedimentary environments, OSL dating has the potential to bridge the gap between these methods and provide chronological continuity.

There are two oft-cited problems with dating of young quartz. The first is the relatively poor signal-to-noise ratio encountered. Despite this, OSL on young quartz has been shown to agree with independently evaluated ages provided by historical maps (Ballarini et al., 2003) and by radiometric methods (Madsen et al., 2005). A more likely source of error comes through partial bleaching, which is of particular concern for young samples where a weak signal can be swamped by any residual signal present at the time of deposition.

Efforts to overcome partial bleaching have two complimentary routes. Statistical methods have been designed to select the youngest parts of the equivalent dose (D_e) distribution (Galbraith et al., 1999). However, all statistical approaches are dependent on at least some of the dose distribution returning the true burial dose. Much effort has therefore been directed at isolating the most rapidly bleached ‘fast’ component of the OSL signal. The fast component is most likely to be well-bleached, is geologically stable over millions of years, and is less susceptible to thermal transfer of charge (Wintle and Murray, 2006). Singarayer and Bailey (2004) showed that experimental isolation of the fast component is possible through raised-temperature infrared (IR) stimulation. However, subsequent IR dating protocols which use either direct IR stimulated luminescence or IR depletion (e.g. Jain et al., 2005) are only applicable to older samples, since the low IR stimulation energy elicits a very slow OSL response with poor signal-to-noise ratios.

Other approaches to fast-component separation have focused on curve-fitting of the OSL data, in which the data is assumed to be the sum of multiple OSL components (such as the ultrafast, fast, medium and slow components). Such approaches assume the superposition of non-interactive first-order processes, such that their combined signal (i.e. the OSL data) can be separated mathematically.

Several authors (e.g. Rhodes et al., 2005; Li and Li, 2006; Choi et al., 2006) have applied the curve-fitting approach to Linearly Modulated (LM) OSL data, in which the

intensity of stimulation light is ramped linearly throughout the measurement period. Under this stimulation mode, the OSL signal over the measurement time appears as a series of overlapping peaks corresponding to different OSL components. However, LM-OSL gives relatively poor signal-to-noise ratios, because the stimulation intensity is sub-maximum until the final time-interval, while at least part of the instrumental noise is independent of stimulation power. The signal-to-noise ratio is higher under (maximum intensity) Continuous Wave (CW) OSL, in which the stimulation light is held at a constant intensity for the duration of the measurement. Moreover, Wallinga et al. (2008) have shown that LM-OSL actually offers no improvement on CW-OSL in the degree of component separation, because the degree of component separation is independent of stimulation light intensity.

Some attempts at fast component isolation have previously been made through curve-fitting the CW-OSL signal (e.g. Watanuki et al., 2005), which is the method we use here. However, no fitting approach has gained widespread use for dating purposes, perhaps because of computational difficulties; the fitting of multiple exponential functions is an ill-posed problem, in the sense that there are many possible solutions (Istratov and Vyvenko, 1999). Nevertheless, here we make use of the CW-OSL signal so as to maximise the signal-to-noise ratio. Our curve-fitting procedure uses pre-defined decay constants, which reduces complexity and increases the speed of data analysis. A Monte Carlo protocol is employed to generate an appropriate error term for the fast component D_e .

2. Methods

2.1 Sample and measurement details

The curve fitting procedure is tested using two samples from the Netherlands: a well-bleached sample NCL-1108011 from a coastal dune in North Holland, and a poorly bleached sample NCL-3505030 from a fluvial channel bar deposit in the central Netherlands. Both samples are approximately 250 years old. Quartz grains 212-250 μm in diameter were extracted from the samples using standard laboratory procedures. A

SAR protocol was used. For sample NCL-1108011, aliquots were preheated at 180°C for 10 s, followed by OSL measurement at 125°C. The cutheat following the test dose was 180°C, and a 200°C OSL bleach was used at the end of each SAR cycle (Murray and Wintle, 2003). For sample NCL-3505030 the OSL readout was at 125°C, preheat and cutheat were at 200°C, the OSL bleach was at 220°C, and an additional 175°C IR bleach prior to each OSL readout was used to minimise the feldspar contribution to the OSL signal (Wallinga et al., 2002).

Since the OSL signals were relatively weak, our SAR protocol contained two unusual features: we used a single regenerative dose of 2.24 Gy, and a relatively high test dose also of 2.24 Gy. These adaptations were designed to increase the number of aliquots processed, and to reduce the uncertainty of the sensitivity-corrected OSL signal. Assuming that the true dose response curve for our samples conforms to a saturating exponential function with a characteristic dose (D_0) of 80 Gy, then our use of a single regenerative dose implies a systematic overestimate of D_e of ~1% (equation and definitions can be found in Duller (2007)). This value is insignificant compared with other sources of error, and can be corrected for if necessary.

Measurements were carried out on a Risø TL-DA-15 reader, using 470 nm blue diodes with a power at the sample position of ~35 mW cm⁻². Irradiation was with a ⁹⁰Sr/⁹⁰Y beta source providing a dose rate of 0.028 Gy s⁻¹ to quartz grains at the sample position. The IR diodes emitted at a wavelength of 875 nm and power of ~116 mW cm⁻². A 7.5 mm Hoya U340 detection filter was used.

2.2 Component characterisation

The presence of multiple optically sensitive electron trap types in quartz has been well established (Bailey et al., 1997; Jain et al., 2003; Singarayer and Bailey, 2003). Assuming first-order kinetics (no re-trapping), then under continuous wave stimulation the OSL response $I_{cw}(t)$ can be described as the sum of N exponentials:

$$I_{cw}(t) = \sum_{i=1}^N n_{0,i} \alpha_i \exp(-\alpha_i t)$$

Each exponential corresponds to a component (fast, medium, slow etc.) with a decay rate under optical stimulation α_i (equal to photoionisation cross-section of component i multiplied by stimulation intensity), and initial trap concentration n_0 at $t = 0$ which can be used to define the D_e . Under CW-OSL, a constant background is also present.

Deconvoluting multiple exponentials is computationally expensive due to the number of free parameters, and is highly sensitive to noise. However, if the number of components is known, and the decay rate parameters α are fixed, then the procedure becomes simple enough for routine analyses.

We have attempted to characterize the decay rates of the fast and medium components in each of our samples. For this, 32 aliquots of each sample were given a relatively large dose of 30 Gy, followed by the preheat and OSL steps described in section 2.1. Each decay curve was then fitted using Origin 7.5, which was able to describe most curves adequately using 3 components plus background. The average decay rate ratios for the 3 components are 1:0.2:0.01, similar to the relative cross-sections of the fast, medium and slow2 components of Jain et al. (2003), and they are henceforth referred to as the fast, medium and slow components. The distribution of decay rates for sample NCL-1108011 is shown in Fig. 1, which plots the fast component decay rate against that of the medium component.

Place figure 1 near here

2.3 Calculation of D_e

Using the measured decay rates of Fig. 1, the average decay rates for the fast and medium components were then used to fit a multiple exponential function to each decay curve of the SAR cycle, using a simple least-squares routine (after subtraction of the separately measured background, see section 2.4.1). The slow component was treated differently, with the decay rate defined separately for each decay curve by fitting a single exponential plus background to the last 25 s of the 40 s OSL decay. This was required because the decay rate of the slow component varied over different OSL steps of the SAR cycle, most likely because it in fact contains several different slow components which bleach at

different rates. D_e (and the repeat point) can be calculated after fitting each OSL decay curve. The OSL signal due to natural irradiation and the dose response curve for one aliquot are shown in Fig. 2a and b.

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2.4 Calculation of error

There are two principal sources of error in the fitting routine described above (assuming the model of quartz OSL stated in section 2.2 is adequate). The first comes from noise in the OSL data. The second comes from the assumptions introduced about the decay rates, i.e. the difference between the true fast and medium component decay rates for an OSL decay curve, and the average decay rate values which are used to fit the data. To take both these factors into account, we used a Monte Carlo error routine based on repeated curve-fitting of simulated data.

2.4.1 Simulation of OSL decay curves

Simulation of decay curves was carried out using the original 3-component fit to the OSL data, which was added with simulated instrumental noise drawn randomly from a measured distribution. This distribution was obtained beforehand, by measuring the OSL of a number of blank discs at the same temperature as the SAR OSL. Each simulated curve was then randomized using the Poisson distribution. Figure 2(c) shows an example simulation of an OSL decay curve due to natural irradiation.

2.4.2 Curve fitting of simulated datasets

Each set of simulated decay curves for an aliquot was put through the fitting routine of section 2.3, but rather than using the average decay constants that were used for the central D_e values, new decay constants for the fast and medium components were drawn from a simulation of the measured distribution of Fig. 1. This distribution was modelled using the (as measured) normal distribution of fast component decay rates, and the relationship between fast and medium component decay rates ($r^2=0.5$). A residual was

then added for the medium component based on the measured normal distribution of residuals.

Once new decay rates had been specified, the simulated decay curves could be fitted to obtain a new value of D_e . Repetition of this process (simulation of decay curves; selecting new decay rates; curve fitting) a large number of times (e.g. 200) leads to a distribution of Monte Carlo D_e values centred on the mean (Fig. 2d), from which the standard deviation provides the error term.

3. Results

The results of the CW-OSL fitting routine can be seen in Fig. 3, where it is compared with more standard methods of generating D_e for the two samples used. In Fig. 3a, the curve-fitting approach to D_e is plotted against that derived from the Late Background (LBG) method for poorly bleached sample NCL-3505030. LBG has been the standard approach used in the literature, in which the first part of the OSL decay curve (e.g. 0.00-0.30 s) is used to define the dose, after subtracting a background taken from the last 4 seconds of stimulation. It can be seen that the curve-fitted fast component tends to give a lower D_e , especially for aliquots which yield imprecise estimates (Fig. 3a), and it always returns a larger error term (Fig. 3b). This relationship is to be expected, since the signal in the LBG method contains significant amounts of medium and slow components which are more likely to be partially bleached. Consequently, the LBG method returns a precise (but possibly inaccurate) D_e for each aliquot even when there is little or no fast component.

Figure 3d compares the curve fitting approach (for the same sample) to an improved method of OSL decay curve integrated time-interval (channel) selection suggested by Ballarini et al. (2007), in which the initial OSL signal is subtracted with a background taken from the channels immediately following. Here we have extended the initial signal length (0.00-0.80 s with background 0.80-1.60 s) to overcome weak signals. The early background (EBG) modification is designed to maximize the proportion of fast component contained in the net OSL signal, and indeed correlates well with the curve-

fitted fast component estimate (Fig. 3d). The error term using EBG also correlates very well with the curve-fitting approach for aliquots returning a relatively precise D_e (Fig. 3e), although still gives smaller errors for aliquots returning a relatively imprecise D_e .

For the well-bleached sample NCL-1108011, estimated D_e is very similar whether obtained by curve-fitting or EBG (Fig. 3g) although there is perhaps a tendency for curve-fitting to increase D_e for aliquots which give less precise estimates. The error terms are also closely related, principally because this sample has a dominant (and well-bleached) fast component. Since this sample is well-bleached, there is little difference between the EBG and LBG methods, so no comparison between the curve-fitted fast component D_e and LBG D_e is shown.

Place figure 3 near here

4. Discussion

The curve-fitting procedure outlined above appears to give the results which could be expected if:

- a) The assumed model of the OSL process (section 2.2) provides an adequate reflection of reality.
- b) The curve-fitting procedure is performing as intended.

After testing the curve-fitting procedure on a well-bleached sample and a partially bleached sample, there seem to be potential benefits of using it to date young quartz. Firstly, the D_e obtained using this method is approximately that of the fast component, so is more likely to indicate the true burial dose than usual background subtraction methods, which may yield D_e values from mixes of the fast and the slower components. Secondly, the error term is directly related to the signal intensity of the fast component, and has little relation to the signal intensities of other components. This contrasts with background subtraction methods, in which a strong medium or slow component signal can lead to a precise but inaccurate D_e estimate, even when there is no fast component present (although this is less of a problem with EBG).

Besides assessing the influence of background noise on D_e , the Monte Carlo error term also includes uncertainty derived from changing the decay constants. This has an agreeable implication for dating of partially bleached samples. For aliquots where all components indicate the same burial dose, artificially altering the decay rate parameters has little or no effect on D_e . By contrast, with aliquots for which the fast and medium component have different true palaeodoses (e.g. partially bleached aliquots), the D_e will be sensitive to a change in the decay rates. In other words, partially bleached aliquots will tend to generate a larger error term than well-bleached aliquots. While the occurrence of identical D_e values, calculated using the fast or the medium components, is not necessarily the harbinger of a well-bleached aliquot, it may nonetheless offer an indication that sufficient bleaching occurred (e.g. Bailey and Arnold, 2006). Because the error term is affected by the difference between the fast component D_e and the medium component D_e , the resulting curve-fitted D_e distribution should have an inbuilt bias towards well-bleached aliquots. Nonetheless, the effect is small in comparison to the error derived from the signal-to-noise ratio, at least for young samples such as those examined here.

Overall, the error term derived from the curve-fitting procedure is larger than standard methods for almost all aliquots measured. However, this need not imply a less precise palaeodose estimate, since in contrast to channel-selection methods, the curve-fitted fast component D_e distribution is dominated by the most suitable aliquots for dating. This may be true even for well-bleached samples; for sample NCL-1108011 used here (Fig. 3g-i), overdispersion of D_e was reduced from 7.0 ± 2.4 % using EBG to 2.2 ± 4.0 % using the curve-fitted fast component. A reduction in overdispersion may reduce the number of apparent dose populations in a D_e distribution, making it simpler to apply and interpret the results of a statistical age model. Furthermore, since the Monte Carlo error routine provides a histogram of the spread in D_e for each aliquot, there is no need to assume that the spread is normally distributed. The histograms of each aliquot could potentially be added together to form a PDF, which could then be used as the input for an age model.

While scripting large parts of the procedure reduces much of the usual tedium of curve fitting, there are some additional steps required prior to analysis. The first is to identify the distribution of decay rates for the fast and medium components by deconvoluting the OSL decay after a high dose (section 2.2). Optical decay rates may vary due to a number of factors, such as grain size and grain opacity, even if we assume the fast and medium components are common in all samples. Furthermore, if the stimulation light intensity is non-uniform across the disc, then the number of bright grains on an aliquot, and their position, may also contribute to variability. It is therefore difficult to define the number of aliquots necessary to get a representative distribution of decay rates. We found at least 30 were required. This is a slightly tedious number of deconvolutions, but the resulting distribution can be applied to many samples with the same source material and grain size (i.e. calculated on a site-by-site basis).

The distribution of instrumental background was also pre-determined. Like the component characterization, it is machine-specific, but can also change over time due to disturbance and settling of the photomultiplier tube. It is also possible that instrumental background is different with grains on the disc, although Li (2007) found otherwise. Even so, it would require a large difference to significantly affect D_e .

There is room to improve some aspects of the program. For example, fitting could be streamlined by using only the first 10 seconds of the decay curve; curve simulation might be better achieved using a smoothing algorithm rather than fitting. For a rigorous assessment of the method, testing will be required on full sedimentary sequences with tight age-control. Nevertheless, the program in its current form has given results which would be expected from a true fast-component protocol, while simultaneously permitting the swift analyses of a large number of aliquots. The combination of these aspects is likely to be of great benefit for quartz OSL dating.

Although the procedure outlined here was specifically designed for young samples, it may be advantageous to use a similar method to date older samples too. For example, there are doubts over the stability of a medium and/or slow component, giving a further reason (besides partial bleaching) for obtaining a fast component signal. In fact, for older samples the procedure could be simplified, since the initial high dose step of section 2.2

would already have been carried out during construction of the dose response curve. It would then be simple enough to tailor the decay constants to each aliquot. Applying this method to single grains would be equally possible, with the added advantage that numerous decay curves could be obtained by giving a high dose to just a few aliquots.

5. Conclusion

We have presented a curve fitting procedure for CW-OSL data for dating young quartz. By pre-defining key fitting parameters, the algorithm constructed is simple enough for processing the large number of aliquots necessary for dating young samples. There are three key improvements over standard channel selection methods:

1. The calculated D_e is approximately that of the fast component.
2. The error term is closely related to the intensity of the fast component signal.
3. The error term contains a bias towards well-bleached aliquots. Partially bleached aliquots return a larger error term if the equivalent doses of the fast and medium component differ.

The method is potentially beneficial for dating both well-bleached and partially bleached samples. While it has been designed with young quartz in mind, it could also be used with little modification to improve the accuracy of dating older samples.

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Figure Captions:

Fig. 1. Fast versus medium component decay rates for 32 aliquots of sample NCL-1108011 (open squares), based on curve-fitting the OSL signal after a 30 Gy dose. The weighted average (filled square) is used to calculate the D_e for all aliquots (section 2.3). The (un-weighted) linear fit (solid line) is used in generating the error term (section 2.4.2).

Fig. 2. D_e and error term calculation for one aliquot of partially bleached sample NCL-3505030: (a) The OSL signal following natural irradiation, fitted with 3 exponentials with pre-defined decay rates, plus a background (not shown). (b) The dose response curve, with dashed line indicating D_e , and with the recycle point also shown (open triangle). (c) An example simulation of the OSL signal due to natural irradiation. (d) Histogram of D_e values calculated using the Monte Carlo error routine.

Fig. 3. Results of the curve-fitted fast component D_e compared to standard channel selection methods. The top row of graphs (a, b, and c) show the fast component versus the late background (LBG) method for partially bleached sample NCL-3505030; the middle row (graphs d, e and f) compare the fast component of the same sample with the early background (EBG) method; the bottom row (graphs g, h and i) show the fast component versus EBG for well-bleached sample NCL-1108011. Each row contains in the left hand column: a D_e comparison between the two methods indicated in the graph, with markers and 1σ error bars shaded in proportion to the relative error on their fast component D_e ; in the middle column: a likewise comparison of the relative error; in the right hand column: probability density functions (PDFs), created using only those aliquots which passed acceptance criteria (recuperation of less than 0.05 Gy, recycling point within 10% of unity). For channel selection methods (LBG and EBG), D_e was calculated as outlined by Duller (2007) with Monte Carlo error terms, after measurement errors were estimated using the method of Li (2007).

Figure 1

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Figure 2

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Figure 3
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