Luminescence dating in fluvial settings: overcoming the challenge of partial bleaching

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Abstract

Optically stimulated luminescence (OSL) dating is a versatile technique that utilises the two most ubiquitous minerals on Earth (quartz or K-feldspar) for constraining the timing of sediment deposition. It has provided accurate ages in agreement with independent age control in many fluvial settings, but is often characterised by partial bleaching of individual grains. Partial bleaching can occur where sunlight exposure is limited and so only a portion of the grains in the sample were exposed to sunlight prior to burial, especially in sediment-laden, turbulent or deep water columns. OSL analysis on multiple grains can provide accurate ages for partially-bleached sediments where the OSL signal-intensity is dominated by a single brighter grain, but will overestimate the age where the OSL signal-intensity is equally as bright (often typical of K-feldspar) or as dim (sometimes typical of quartz). In such settings, it is important to identify partial bleaching and the minimum dose population by analysing single grains and applying the appropriate statistical age model to the dose population obtained for each sample. To determine accurate OSL ages using these age models, it is important to quantify the amount of scatter (or overdispersion) in the well-bleached part of the partially-bleached dose distribution, which can vary between sediment samples depending upon the bedrock sources and transport histories of grains. Here we discuss how the effects of partial bleaching can be easily identified and overcome to determine accurate ages. This discussion will therefore focus entirely on the burial dose determination for OSL dating, rather than the dose-rate, as only the burial doses are impacted by the effects of partial bleaching.

Keywords: Optically stimulated luminescence dating; OSL; partial bleaching; fluvial; single grains; age models.

1. Introduction

Constraining the timing of sediment deposition in fluvial settings is important for understanding the rates and magnitude of sedimentary processes and events (e.g. floods). A number of geochronological techniques are available to provide ages in fluvial settings, but certain techniques can be restricted by the lack of material preservation and dateable age ranges. Optically stimulated luminescence (OSL) dating is a versatile technique that directly dates the time elapsed since a mineral grain or rock surface was exposed to sunlight and subsequently

buried (Huntley et al. 1985), and has often extended age ranges for sediment burial beyond radiocarbon dating (e.g. Burow et al. 2015). OSL dating is performed on the two most ubiquitous minerals in the Earth's crust (either quartz or K-feldspar), which increases the likelihood of finding material for dating. Moreover, grainsizes from silt up to boulder-sized clasts can be used for dating and the dateable age range for OSL dating extends beyond that of radiocarbon dating, typically up to ~100 ka for quartz and ~500 ka for K-feldspar; however, the age range of the technique is highly dependent upon the characteristics of each sample and the luminescence signal used for analysis. OSL dating is therefore well-suited for constraining the sediment deposition in a fluvial setting and has been extensively used to determine accurate ages in many different environments (e.g. Burow et al. 2015; Colarossi et al. 2015; Giosan et al. in 2012; Kolb and Fuchs, 2018; Lyons et al. 2013, 2014; Thomas et al. 2017); this includes the deposition of young (Shen and Mauz, 2012) and palaeo-delatic sediments (Shen et al. 2013, 2015), and rapidly-deposited sediment during flood events (e.g. He et al. 2019; Medialdea et al. 2014). Novel approaches of the OSL dating technique have also deciphered sediment transport pathways and residence times in fluvial systems (e.g. Chamberlain et al. 2017; Gray et al. 2018; Reimann et al. 2015).

An important consideration for OSL dating in fluvial settings is the extent of sunlight exposure that each individual grain has experienced prior to burial. When grains are exposed to sufficient durations and intensities of sunlight, the OSL signal is reset to zero (or bleached). To determine an accurate age for the deposition of a sedimentary sample, it must contain at least some grains whose OSL signals were fully reset prior to burial. Typically, in depositional setting where there is a greater opportunity for grains to have been exposed to sunlight prior to burial (e.g. aeolian), the OSL signal of all of the individual grains was equally reset; this is often termed well bleached. In contrast, in depositional settings where there is less opportunity for sunlight exposure to all grains (e.g. fluvial, glaciofluvial), the OSL signal of only a portion of the grains in the sample may have been reset to zero prior to burial; this is often termed partial bleaching. To calculate an accurate OSL age for well-bleached sediments, all of the grains in the population can be used; however, for partially-bleached sediments, only those grains in the partially-bleached population that were well-bleached prior to burial can be used to prevent overestimation of the true burial age. Here, we will discuss the process of partial bleaching in a fluvial setting, in addition to the approaches that can be used in OSL dating to identify the effects of partial bleaching and calculate accurate ages for sediment burial.

2. Luminescence dating: basic principles

A fundamental requirement of OSL dating is that a mineral grain can store and release energy (or electrons), almost like a rechargeable battery (after Duller, 2008). The electrons are ionised

by the low-level radiation that is all around us due to the emission of alpha and beta particles, and gamma rays during the radioactive decay of K, Rb, U and Th in radionuclide equilibrium in the surrounding environment, in addition to cosmic rays. In the crystal lattice of minerals, electrons become trapped at defects or impurities, which can then be released when the mineral is excited by stimulation of light (OSL), heat or pressure. When the mineral grains are then buried for a period of time and exposed to natural radiation from the surrounding environment, trapped electrons will re-accumulate at the defects within the grain. The long life-times of radioactive elements in the natural environment mean that the accumulation of energy resulting from the environmental dose-rate is typically constant over time. Therefore, we can determine the time elapsed since a mineral grain was last exposed to light and buried by measuring the OSL signal emitted from the mineral grains and comparing it to OSL signals resulting from known radiation doses delivered in the laboratory to the same sample: this gives the equivalent dose (D_e). To obtain the age, we divide the D_e by the environmental dose-rate (Eq. 1), which is determined for the bulk sediment from its geochemical composition or by emission-counting techniques for alpha, beta and gamma radiation.

$$\text{Age (ka)} = \frac{\text{Equivalent dose (Gy)}}{\text{Dose rate (Gy/ka)}} \quad (1)$$

3. Mineral choice: quartz or feldspar?

The two principle minerals used for OSL dating are quartz and K-feldspar. Since 2000 and the development of the single aliquot regenerative dose (SAR) protocol (Murray and Wintle, 2000), quartz has been the preferred mineral for OSL dating. However, in certain settings very few grains of quartz emit an OSL signal and those signals can be very dim, making OSL analysis to determine the D_e value very difficult and inefficient (e.g. Chiverrell et al. 2018). In comparison, for the same sample a large proportion of K-feldspar grains can emit a detectable OSL signal of K-feldspar, which can be relatively brighter than the OSL signal of the quartz; thus, making OSL analysis more efficient and potentially more precise as brighter signals generate better counting statistics and reduce uncertainties on the D_e values. The relative brightness of K-feldspar grains over quartz has meant that in some settings it is advantageous to analyse K-feldspar grains. However, we need to consider the additional complexities of internal dose-rates, anomalous fading and slower inherent bleaching rates for K-feldspar, which are characteristic of K-feldspar but do not pertain to quartz.

Anomalous fading of K-feldspar is the athermal depletion of trapped charge stored within the grain during burial (Wintle, 1973). Previous studies have suggested that anomalous fading is

ubiquitous to all K-feldspar when the D_e value is measured using the infra-red stimulated luminescence (IRSL) signal at 50 °C (Huntley and Lamothe, 2001), and thus, the ages need fading correction to prevent age underestimations. There are methods that can be used to measure and correct for anomalous fading (e.g. Huntley and Lamothe, 2001; Kars et al. 2008), but this can often introduce additional uncertainty into age calculations. Therefore, it is advantageous to circumvent anomalous fading by using a more stable signal of K-feldspar. In 2008, Thomsen et al. (2008) developed the use of a new signal of K-feldspar that can circumvent the issue of anomalous fading; this is termed the post-IR IRSL signal and has revolutionised the use of K-feldspar for luminescence dating. The post-IR IRSL protocol is a two-step procedure that uses an initial IRSL measurement at a lower temperature (typically 50 °C) to remove the more unstable IRSL signal followed by an IRSL measurement at a higher temperature, typically 225 °C (the $pIRIR_{225}$ signal; Buylaert et al. 2009) or 290 °C (the $pIRIR_{290}$ signal; Thiel et al. 2011), which is used to determine a D_e value. By accessing the higher-temperature, more stable pIRIR signal in K-feldspar, we can circumvent anomalous fading and determine accurate ages, which have been validated against independent age control (e.g. Roberts, 2012).

Another consideration for the mineral choice is the relative differences in bleaching rates inherent to the OSL signal of quartz and the IR_{50} and post-IR IRSL signals of K-feldspar, especially when dating sediments deposited during the last few centuries. It has long been known that in air (not within a water column), the OSL signal of quartz typically bleaches faster than the IR_{50} signal of K-feldspar (Godfrey-Smith et al. 1988). Collarossi et al. (2015) then showed using a solar simulator (typically up to ~6.5 times stronger than direct sunlight) that the OSL signal of quartz was reduced to 5 % of the original signal after only 10 s of bleaching, while the $pIRIR_{225}$ and $pIRIR_{290}$ signals of K-feldspar took four and 14 days respectively to reduce to 5 % of the original signal. Slower inherent bleaching rates of the pIRIR signals mean that small residual doses may be incorporated into the D_e value used for dating because the OSL signal was not fully reset to zero prior to burial. These small residual doses are negligible when dating older samples, but may cause age overestimation for young samples deposited in the last few hundred years, and so lower-temperature signals are often preferred in such cases to minimise the impact upon dating (e.g. Reimann et al. 2011; Reimann and Tsukamoto, 2012). It was thought that small residual doses in K-feldspar would also restrict the use of the pIRIR signals for single-grain analysis, and although Smedley et al. (2015) found that the inherent bleaching rates of the pIRIR signals of K-feldspar varied between grains, the bleaching rates of the $pIRIR_{225}$ signals of most of the single grains were similar and would not restrict single-grain analysis. However, the bleaching rates of the higher-temperature $pIRIR_{290}$ signals of the single grains were highly variable, where very few grains bleached at faster

rates and so single-grain dating of K-feldspar grains using the pIRIR$_{290}$ signal in a partially-bleached setting would likely be characterised by large and variable residual doses in individual grains (Smedley et al. 2015). This is supported by recent findings from modern river systems that showed that some single grains of K-feldspar determined zero dose values using the pIRIR signals (Gliganic et al. 2017).

4. Partial bleaching in fluvial settings

Sedimentary samples from fluvial settings can be partially bleached prior to deposition, especially in higher-energy, deeper river channels. Although there is the potential for the OSL signal of fluvial sediments to be partially bleached prior to burial, a surprising proportion of samples were well bleached, especially in shallow, low-energy fluvial settings (e.g. Durcan et al. in press). For a suite of 72 glaciofluvial samples from the British-Irish Ice Sheet, the single-grain D$_e$ distributions determined using quartz showed that ~30 % of them were well-bleached prior to burial (e.g. Smedley et al. 2017a,b). This demonstrates that there is a greater potential for sunlight exposure prior to burial than we might assume in glaciofluvial settings, which are typically expected to have less opportunity for sunlight exposure than fluvial settings. Little is known about the physical processes of OSL signal resetting in fluvial settings in the natural environment, but it is suggested that sunlight attenuation is greater in deeper, sediment-laden, turbulent water columns. Therefore, implying that there may be variability in the bleaching efficiency of different grainsizes, and potentially different minerals due to preferential attenuation of different wavelengths through water columns. Previous studies have shown how shorter wavelengths that are more efficient at bleaching the OSL signal of quartz are attenuated to greater extents in turbid water columns in comparison to the wavelengths that are more efficient at bleaching the IRSL signals of K-feldspar (e.g. Jerlov, 1970; Krongborg, 1983; Sanderson et al. 2007). However, this still needs to be validated by directly comparing single-grain OSL dating of quartz and K-feldspar of the same sedimentary samples taken from a former river system.

Our understanding of how coarser (sand) and finer (silt) grainsizes bleach in a fluvial setting varies between studies, perhaps reflecting the complex nature of sunlight bleaching in different fluvial settings and processes. Many studies report a difference in the OSL ages obtained from the coarser and finer grainsizes (e.g. Gray and Mahan, 2015; Bailey et al., 2003; Fuchs et al. 2005; Olley et al. 1998; Truelsen and Wallinga 2003), but the reasons for this continue to be unresolved. Some studies have repeatedly observed a general depletion of D$_e$ in fluvial sedimentary samples with increasing transport distances (Gray and Mahan, 2015; Bailey et al., 2003; Stokes et al., 2001); this is attributed to the fact that coarser grainsizes fall out from suspension first, which restricts the distance that the grains are transported and therefore their

potential exposure to sunlight during transportation. This theory is also relevant to vertical grainsize distribution in the water column as a turbid water column with a high suspended load is likely to attenuate the sunlight through the water column and so restrict the bleaching of coarser grainsizes transported across or close to the river bed via saltation (e.g. Gray and Mahan, 2015; Rittenour, 2008). Alternatively, other studies have reported that coarser grainsizes are less prone to partial bleaching and are therefore deemed to determine accurate ages in fluvial settings in comparison to finer grainsizes (e.g. He et al. 2019; Kim et al. 2015; Fuchs et al. 2005; Fan et al. 2010; Olley et al. 1998; Thompson et al. 2018; Truelsen and Wallinga 2003). It has been suggested that this could be caused by greater sunlight exposure during shorter periods of deposition prior to transportation (e.g. in mid-channel bars) could preferentially bleach the coarser grainsizes, while the sunlight bleaching of finer grainsizes may be restricted by mud-coatings (e.g. Truelsen and Wallinga 2003). It may also be because coarser grainsizes are transported at a slower rate and for longer distance in a river system via traction or saltation and so have greater opportunity for sunlight exposure than finer grainsizes which are transported within the potentially turbulent water column via suspension (He et al. 2019; Thompson et al. 2018). It is likely that the contrasting reports of whether the coarser or finer grainsizes bleach preferentially in a river system are related to the complex sedimentary processes occurring prior to deposition and subsequent burial. Further investigations on the physical processes of OSL signal resetting in river channels in the natural environment are required to understand how partial bleaching in the water column impacts upon different grainsizes and minerals.

5. Overcoming partial bleaching with OSL analysis

The effects of partial bleaching in the natural environment may sound challenging for OSL dating in comparison to the well-bleached sediments typical of aeolian settings, but in fact, it can be easily identified and overcome to determine accurate ages by using the appropriate techniques.

5.1 Identifying partial bleaching

A D_e distribution determined for a well-bleached sediment will form a log-normal distribution around a central D_e value that appears symmetrically distributed when plotted in log-space (e.g. Fig. 1a). In contrast, partially-bleached D_e distributions are scattered asymmetrically in log-space from a minimum dose population that was well-bleached prior to burial, up to larger doses from grains that may never have been exposed to sunlight and have saturated OSL signals (e.g. Fig. 1b). To fully characterise the true nature of a partially-bleached D_e distribution, OSL analysis must be performed on single grains as the extent of sunlight exposure and signal resetting varied between grains prior to burial; some grains were well

bleached while the other grains may never have been exposed to sunlight. The easiest method of ensuring that OSL analysis is performed on the OSL signal emitted from an individual grain is to stimulate single grains at a time using a focussed laser system (Duller et al. 1999). Grains are mounted in purpose-built single-grain discs with a 10 x 10 array of holes drilled at specific diameters so that only a single grain can be in each hole at a time (e.g. Fig. 2a). For example, a grainsize of 150 – 180 μm and 212 – 250 μm would be analysed using 200 μm and 300 μm holes, respectively, to prevent more than one grain being present in each hole (e.g. Fig. 2b). Where more than one grain is present in each hole, the analysis should be termed pseudo-single grain or microhole measurements as they are not truly single grain measurements and it can have an impact upon the D_e value determined (see Arnold and Roberts, 2009). Single-grain OSL measurements can also be obtained by integrating the OSL signal emitted by individual grains measured using an electron-multiplying charged coupled device (EMCCD) (Lapp et al. 2015; Thomsen et al. 2015), but typically requires a bespoke EMCCD attachment for equipment and is less sensitive than the photo-multiplier tubes typically used for OSL analysis; thus, EMCCDs are not currently used on a routine basis for analysis.

In addition to single-grain analysis, it may also be possible to characterise a partially-bleached D_e distribution by analysing multiple-grain aliquots (typically 2 mm in diameter) using light emitting diodes (LEDs), if the OSL signal averaged from all of the grains on the aliquot is dominated by a single grain. This can be the case for some samples (e.g. Fig. 3, Sample B), but in certain settings the OSL signal of quartz can be equally distributed across the individual grains (e.g. Fig. 3, Sample A) and so more than one grain contributes to the multiple-grain OSL signal. This is often the case during OSL analysis of K-feldspar as the grains are often equally as bright as each other (e.g. Fig. 3, Sample B). Where OSL signals are averaged equally across multiple grains and not dominated by a single grain, it can be difficult to characterise the partially-bleached D_e distribution (e.g. Trauerstein et al. 2017). This is especially problematic for the minimum dose population as measurements are weighted towards the larger D_e values because they typically emit brighter natural OSL signals than those grains that characterise the minimum dose population. In such cases, many more measurements may be required to characterise the minimum dose population in the partially-bleached D_e distribution in comparison to single grains. OSL signal averaging across multiple grains can even be a problem for the pseudo-single grain measurements where more than one grain is in each hole of a single-grain disc (e.g. Fig. 2b), and so microhole measurements should be treated with caution and not assumed to be equivalent to true single-grain measurements (e.g. sample T3DOGM01, Chiverrell et al. 2018).

5.2   Calculating accurate ages for partially-bleached sediments

Sophisticated statistical age models are used to determine OSL ages from partially-bleached D_e distributions (see Galbraith and Roberts, 2012 for a review). For the majority of partially-bleached samples from fluvial settings, an accurate OSL age for the last depositional event will be determined from the well-bleached population in a partially-bleached D_e distribution and requires the application of a minimum age model; this includes the Minimum Age Model (MAM; Galbraith and Laslett, 1993; Galbraith et al. 1999) or the Internal External Uncertainty (IEU) model (Thomsen et al. 2007; Smedley, 2015). In cases where the D_e distribution contains multiple, distinct populations (e.g. bimodal), the Finite Mixture Model (FMM; Galbraith and Green, 1990) would need to be applied to determine ages from those discrete populations (e.g. Rodnight et al. 2006). Bailey and Arnold (2006) provide a decision tree which can be used to determine the most appropriate age model to apply for a given D_e distribution. The number of D_e values needed to fully characterise a partially-bleached D_e distribution and calculate an accurate using the MAM or FMM will depend on the extent of bleaching prior to burial. Rodnight (2006) show that a minimum of 50 D_e values should be used as a working population.

Overdispersion quantifies the amount of scatter in a D_e distribution. To determine accurate OSL ages using the MAM, FMM and IEU models, we must be able to quantify the amount of overdispersion that would be expected in the same D_e distribution had the sediment been well bleached prior to burial, instead of partially bleached. This allows us to identify the well-bleached part of the partially-bleached D_e distribution and determine an age based on those grains that were well bleached prior to burial. For the MAM and FMM, this is quantified by the σ_b parameter, and for the IEU model, it is quantified by the relative values of *a* and *b.* Accurately quantifying the values of σ_b (MAM, FMM) and *a* and *b* (IEU model) are important for calculating accurate OSL ages, and even small changes can have a large impact upon the age determined (e.g. Fig. 4).

The extent of bleaching in nature prior to burial is currently thought to be the most dominant control upon overdispersion in D_e distributions. However, scatter can also be introduced into a D_e distribution from a number of sources, which need to be considered when quantifying σ_b (MAM, FMM) or *a* and *b* (IEU model). The most ideal approach to quantify σ_b is to estimate it from the total overdispersion in the D_e distribution of a well-bleached sample from the same site (Galbriath and Roberts, 2012) sourced from similar bedrock. However, this is not always possible and so alternatively we can quantify and combine in quadrature the overdispersion arising from the intrinsic and extrinsic sources (after Thomsen et al. 2005). For multiple-grain measurements, sources of overdispersion are limited to the intrinsic luminescence characteristics of the grains, which includes a contribution from instrumental reproducibility during OSL analysis (Thomsen et al. 2005). The overdispersion arising from intrinsic

luminescence characteristics is typically determined from dose-recovery experiments, which is performed on grains that have fully reset OSL signals and have been given a known beta dose (Murray and Wintle, 2000). If the known beta dose can be recovered within ± 10 %, the protocol used for analysis is appropriate for the sample, and the scatter in the single-grain D_e values determined provides an estimate of the intrinsic overdispersion. Previous studies have shown that the intrinsic overdispersion of single grains of quartz varies between samples, and so sample-specific dose-recovery measurements were performed to quantify the intrinsic overdispersion for σ_b (e.g. Smedley et al. 2017a,b; Chiverrell et al. 2018).

For single-grain measurements, overdispersion in a D_e distribution determined for a natural sample also includes the effects of microdosimetry, but is difficult to quantify. Environmental dose-rates are routinely determined from bulk and homogenous samples taken from the sediment matrix, whereas D_e values are determined from individual grains from a sub-sample of the bulk material. Thus, the bulk estimation of the environmental dose-rate cannot quantify or account for any microscale heterogeneity in the environmental dose-rate to individual grains throughout burial; this is termed microdosimetry. Etching of the grain surface during sample preparation removes the alpha-influenced outer portion of the grain used for analysis, while the effective ranges of gamma and cosmic rays are greater than the size of an individual quartz grain (~200 µm). Thus, it is only the external beta dose-rate arising from either K, Rb, U and Th that causes microscale heterogeneities in the dose-rate, where beta particles are deposited as a non-linear function of distance from the point source (i.e. a K-feldspar or zircon grain). Autoradiographs of three samples with different extents of beta dose-heterogeneity are shown in Fig. 5 as examples. Beta dose-heterogeneity is likely to have a greater influence on single-grain D_e distributions determined from quartz in comparison to K-feldspar as quartz grains are internally inert (e.g. Jacobs et al. 2006). Therefore, all of the environmental dose-rate for quartz is from external sources of K, Rb, U and Th, whereas K-feldspar have an additional internal dose-rate that typically accounts for ~30 % of the environmental dose-rate (e.g. Smedley et al. 2012, 2016).

Many studies have suggested that the uneven distribution of K within the sediment matrix can cause heterogeneities in the beta dose-rate and results in scatter in D_e distributions determined using single grains of quartz (Mayya et al. 2006; Nathan et al. 2003; Guerin et al. 2015; Jankowski and Jacobs, 2018). Additional overdispersion of 20 % was incorporated into the σ_b value when using the MAM for OSL dating of single grain of quartz from glaciofluvial sediments to account for the effects of microdosimetry (Smedley et al. 2017b; Chiverrell et al. 2018; Glasser et al. 2018); this was based on the amount of overdispersion in related well-bleached sediments after the removal of intrinsic luminescence characteristics (Smedley et al. 2017b). The use of an additional overdispersion of 20 % incorporated into σ_b for determining

accurate OSL ages for these samples was supported by the excellent agreement between the OSL ages and independent age control provided by the cosmogenic nuclide dating (Smedley et al. 2017b).

Single-grain D_e distributions of K-feldspar have two potential sources of overdispersion in addition to intrinsic luminescence characteristics and microdosimetry that are not characteristic of quartz: (1) internal dose-rates and (2) anomalous fading. Studies have used geochemical measurements of single grains of K-feldspar and demonstrated that there is variability between grains in the internal dose-rates of samples caused by internal K-contents (e.g. Smedley et al. 2012; Trauerstein et al. 2012; Gaar et al. 2014) and U and Th (Smedley and Pearce, 2016). The overdispersion that arises from this variability in the internal dose-rates has been estimated at ~10 % (Smedley and Pearce, 2016) and should be incorporated into σ_b to account for the scatter that will be in the well-bleached part of the partially-bleached D_e distribution. Variability in the anomalous fading rates of single grains of K-feldspar also has the potential to introduce scatter into a single-grain D_e distribution, which is reflected by the larger overdispersion values that are often reported for the IR_{50} signal in comparison to the pIRIR signal for the same sample (e.g. Trauerstein et al. 2012; Smedley et al. 2016). Fig. 6 shows an example of a sample where the single-grain D_e distributions determined using the IR_{50} and $pIRIR_{225}$ signal determined overdispersion values of 54 % and 38 %, respectively. This suggests that additional overdispersion of 38 % (when subtracted in quadrature) that is potentially introduced by anomalous fading into single-grain D_e distributions using the IR_{50} signal, and should be considered when quantifying σ_b values (MAM, FMM) and *a* and *b* values (IEU) when performing single-grain dating of K-feldspars using the IR_{50} signal.

6. New techniques: luminescence dating of rocks

New luminescence dating techniques using rock slices from cobbles and boulders have highlighted the potential for constraining sediment deposition in partially-bleached environments such as fluvial systems. Sohbati et al. (2012) developed the use of rock slices taken from boulders for determining surface exposure ages using luminescence dating techniques, which built upon initial research that demonstrated that exposure to sunlight could reset the OSL signal at depths within rocks (e.g. Habermann et al. 2000; Polikreti et al. 2002, 2003; Sohbati et al. 2011; Vafiadou et al. 2007). Depth profiles of the luminescence signal into the rock surface are determined by drilling cores (typically ~2 mm deep) using a water-cooled, diamond-tipped drillbit (typically of diameter ~7 – 10 mm) and then slicing them at intervals with a diamond-tipped blade (typically ~0.4 – 1 mm thick); this method has been used to constrain the surface exposure ages of rockfalls (Sohbati et al. 2012; Chapot et al. 2012), glacial boulders (e.g. Lehmann et al. 2018) and archaeological sites (Freiesleben et al. 2015).

It was shown that the luminescence depth profiles could record multiple exposure/burial cycles within them (Freiesleben et al. 2015), which led workers to develop the use of rocks for burial dating similar to the approach currently used for dating silt and sand grains (Jenkins et al. 2018; Rades et al. in press). Studies have since provided accurate ages in agreement with independent age control to demonstrate the accuracy of these new techniques (e.g. Jenkins et al. 2018; Lehmann et al. 2018). Further studies investigating the optical bleaching properties of different lithologies at depth have shown that the light attenuation into the rock is controlled by the mineral opacity (Ou et al. in press; Meyer et al. in press), where light passes more efficiently through lighter-coloured rocks and should be targeted for OSL dating of rocks (Ou et al. in press). Continued development of this technique has also led to the use of OSL-surface exposure dating paired with cosmogenic nuclide dating for reconstructing erosion rates on centennial-to-millennial scales which is otherwise not possible with current techniques (e.g. Sohbati et al. 2018). The new luminescence dating techniques using rock slices offer excellent potential for expanding the application of OSL dating in sedimentary environments, especially gravel-bed river systems, but also for constraining novel depositional processes (e.g. erosion rates) via surface exposure dating.

7. Conclusion

Optically stimulated luminescence (OSL) dating is a versatile technique that utilises the two most ubiquitous minerals on Earth (quartz or K-feldspar) and is well suited to constraining the timing of sediment deposition in fluvial settings important for understanding the rates and magnitude of sedimentary processes and events. Continuing technical developments facilitate the determination of accurate and precise ages, and provide the potential for analysing new materials, which includes the use of cobbles and boulders for both burial and exposure dating. Although a surprising number of sedimentary samples from fluvial settings are deemed to have been well bleached prior to burial, OSL dating in fluvial settings is often characterised by partial bleaching of the OSL signal of individual grains. Partial bleaching can occur where the potential for sunlight exposure is limited and so only a portion of the grains in the sedimentary sample were exposed to sunlight prior to burial, especially where grains are transported in sediment-laden, turbulent or deep water columns that have greater attenuation of sunlight. Little is known about the physical processes of OSL signal resetting in water in the natural environment, and there are contrasting reports over whether coarser (sand) or finer (silt) grainsizes bleach preferentially in a fluvial system; this likely reflects the complex sedimentary processes occurring in such settings that may lead to the differential bleaching of grainsizes and minerals.

The effects of partial bleaching in the natural environment may seem challenging for OSL dating in comparison to the well-bleached sediments, but in fact, it can be easily identified and overcome to determine accurate ages. OSL analysis on multiple grains can provide accurate ages for partially-bleached sediments where the OSL signal-intensity is dominated by a single brighter grain, but will overestimate the age where the OSL signal-intensity is equally as bright (often typical of K-feldspar grains) or as dim (sometimes typical of quartz grains). In such settings, it is important to identify partial bleaching and the minimum dose population that was well bleached during the last depositional cycle; this is also possible by analysing single grains and applying an appropriate statistical age models e.g. the MAM, FMM or IEU model. To determine accurate OSL ages using these age models, it is important to quantify the amount of scatter (or overdispersion) in the well-bleached part of the partially-bleached D_e distribution, referred to as σ_b for the MAM and FMM, and a and b values for the IEU model. This can be quantified from the total overdispersion in the D_e distribution of a well-bleached sample from the same site sourced from similar bedrock, or where this is not possible, can be combined in quadrature from the overdispersion arising from the intrinsic and extrinsic sources. For quartz grains, these sources are limited to the intrinsic luminescence characteristics, instrument reproducibility and microdosimetry, but for K-feldspar grains, this may also include the overdispersion arising from anomalous fading and internal dose-rates. By using these approaches, OSL dating can provide ages in excellent agreement with independent age control from sedimentary samples across many fluvial settings.

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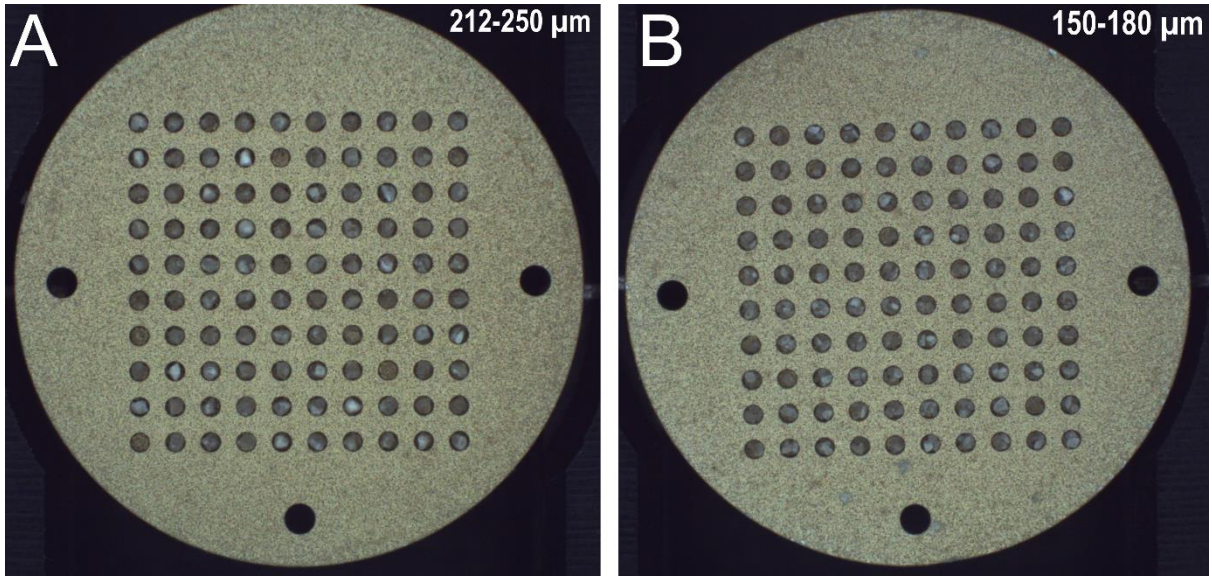Wintle, A.G., 1973. Anomalous fading of thermoluminescence in mineral samples. Nature, 245, 143-144.

Fig. 1. Single-grain discs with hole diameters of 300 μm, but loaded with a grainsize of 212-250 μm so that only a single grain is present in each hole i.e. truly single-grain measurements (A), or loaded with a grainsize of 150-180 μm so that up to 4 grains may be present in each hole i.e. microhole measurements (B).
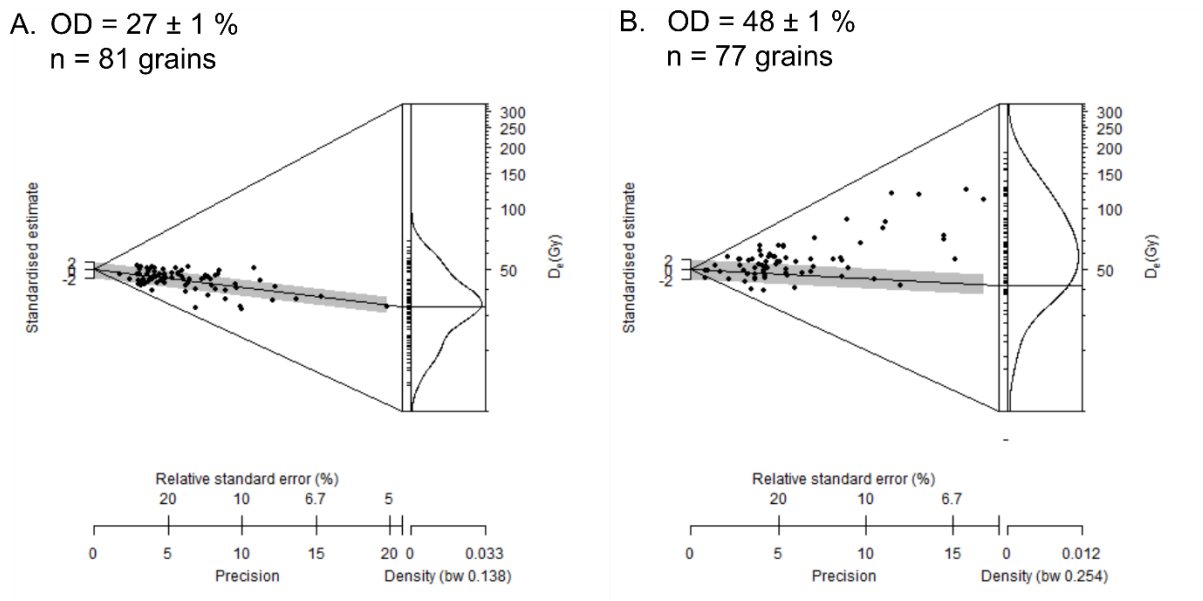

Fig. 2. Abanico plots showing example single-grain D_e distributions determined for samples deemed to have been well bleached (A) and partially bleached (A) prior to burial in a fluvial setting. Abanico plots (Dietze et al. 2016) are composed of multiple axes and give an indication of the amount of scatter in a D_e distribution. Each datapoint is a D_e value and the value can be read by drawing a line from 0 on the y-axis (standardised estimate, ± 2 σ) through the datapoint to the z-axis (D_e). The positioning of the datapoint along the x-axis (precision) shows how precisely known the D_e value is (i.e. scale of its uncertainty), where the more precisely known points are towards the right of the graph. The same data is also presented as a probability density function in the plot on the right hand side of the figure. Note that the grey bar shows the D_e value determine for the well-bleached sample using the CAM (A) and the partially-bleached sample using the MAM (B).
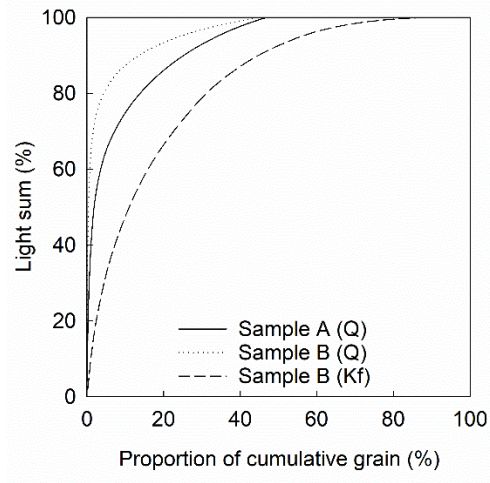
Fig. 3. Distribution of luminescence signal intensity emitted by single grains of quartz for samples where the signal-intensity distribution between quartz (Q) grains was even (Sample A); where the signal-intensity distribution between quartz grains is dominated by a few brighter grains (Sample B). Also shown is the corresponding signal-intensity distribution for single grains of K-feldspar for Sample B.
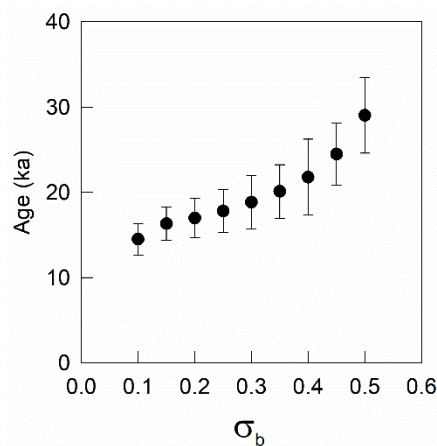

Fig. 4. OSL ages calculated using the MAM D_e value plotted as a function of the σ_b used for the D_e calculations for the partially-bleached sample shown in Fig. 1b, which is an example of a partially-bleached sediment from a fluvial setting.
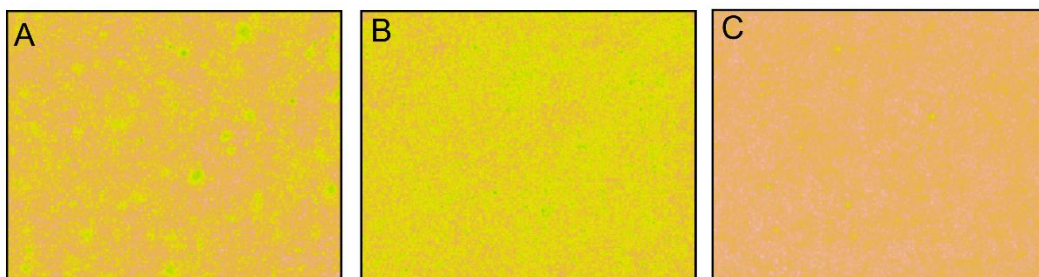

Fig. 5. Autoradiography images showing the beta dose heterogeneity in three different samples: (A) high, heterogeneous beta dose-rate; (B) high, homogeneous beta dose-rate; and a (C) low, heterogeneous beta dose-rate. The autoradiographs shown here were corrected for the background signal, normalised to 1 h exposure time and aggregated to 200 μm^2 pixels.
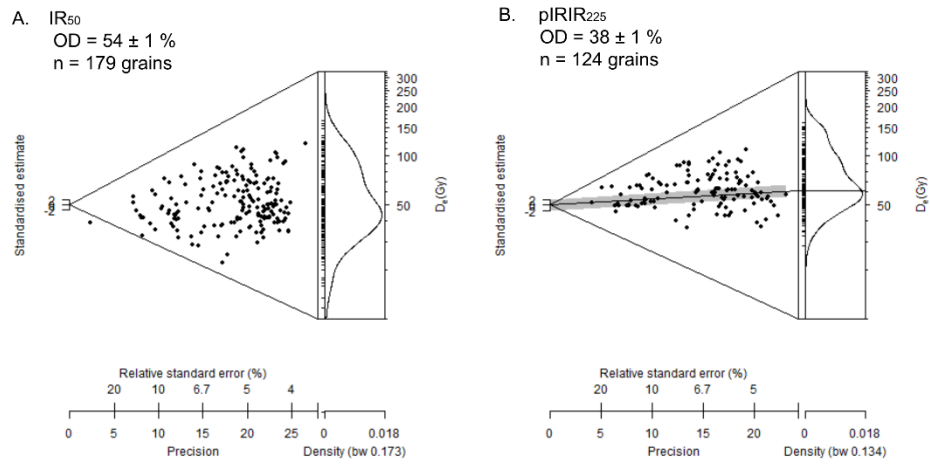
Fig. 6. Abanico plots of single-grains of K-feldspar determined using the IR_{50} and $pIRIR_{225}$ signal for the same sample.