Luminescence Dating

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ABSTRACT: Luminescence dating is a unique chronometric tool as it dates sediments and landforms directly by establishing the time elapsed since grains were last exposed to daylight. Sediments from various terrestrial and shallow marine environments, ranging from a few years to over one hundred thousand years in age can be dated using luminescence techniques. Over the last decade, methodological and technological developments have improved the reliability and precision of the technique, thus encouraging widespread application in a variety of environments and helping resolve questions regarding the timing and rate of geomorphic processes. The field of luminescence is continually advancing and ongoing research focuses on extending the age range and exploring new applications. This paper outlines the principles behind luminescence dating and introduces the current and most widely applied methodological approach to using luminescence dating in geomorphology.

KEYWORDS: luminescence, OSL, chronology, Quaternary, environmental change, sediments

Introduction

Constraining the age of sediments and landforms and determining the sequence of events driving landscape development is fundamental in the study of geomorphology and allows geomorphologists to test theoretical models and quantify rates of landscape change. A number of dating tools targeting various materials and spanning different age ranges are available to support geomorphological research (refer to other chapters in this edition). Of these, radiocarbon dating (^{14}C) is historically the most commonly used technique. However, ^{14}C dating is limited to environments where organic matter is preserved in the sediment and as many geomorphic systems are predominantly silicaclastic, its use is often restricted. As an alternative, Luminescence dating uses minerals that are common in most environments making the technique widely applicable in variety of landscapes. Further, it is particularly important in geomorphology as it dates sediments and landforms directly and gives numerical ages without the need for independent age control (see Lian and Roberts, 2006).

This paper provides a brief introduction into the basic principles that allow age determination using luminescence, focusing on common laboratory practices and data analysis techniques in Optically Stimulated Luminescence (OSL) dating. It also acts a guide to assist geomorphologists in selecting the most suitable depositional settings, site locations and sampling methodologies. Overall this technical guide is intended to provide readers with an informed platform of knowledge from which they are able to acquire more information from the published literature or approach individual laboratories for further advice.

Basic Principles

Luminescence dating determines the age since burial of sediment by measuring the total amount of stored signal resulting from exposure of the sediment to a known annual dose of background radiation. The concept of luminescence dating relies on defects in the crystal lattice of dosimeter minerals, most commonly quartz and feldspar, to trap energy produced during the interaction between
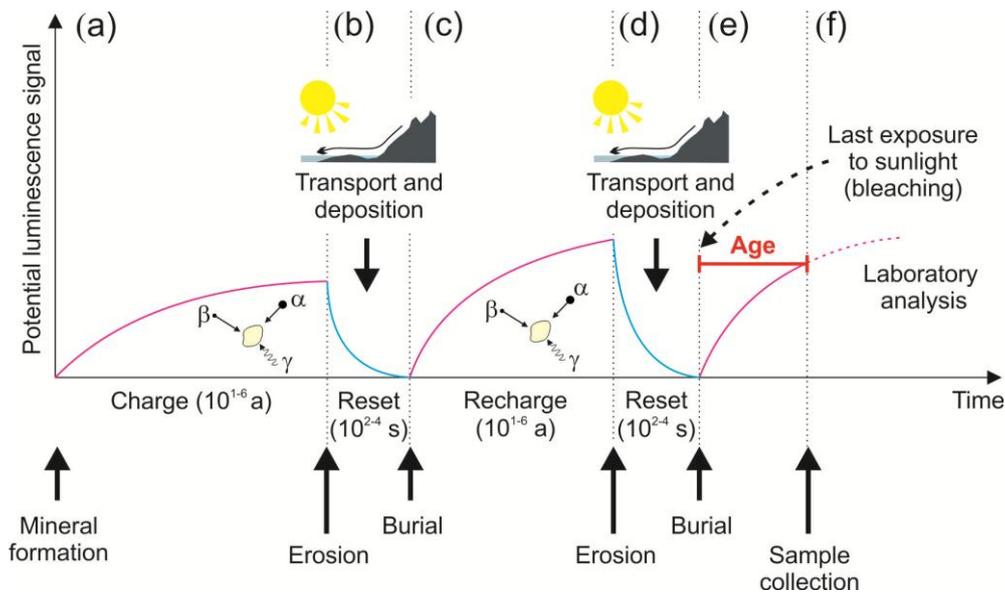
Figure 1: Basic principles of luminescence dating. (a) The luminescence signal builds through time as the sediment is exposed to ionising background radiation. (b) The mineral is eroded, transported and deposited during which time it is exposed to daylight and the energy previously acquired is released as luminescence thus resetting the signal to zero. (c) Upon burial, the luminescence signal builds once more. (d) The luminescence signal is zeroed again. (e) The mineral is again buried and acquires a luminescence signal with time. (f) By sampling without exposure to daylight and then measuring the amount of luminescence given by the mineral (D_e) and the dose-rate it was exposed to, the time elapsed since last exposure to daylight can be determined.

electrons within the crystal and background radiation from the radioactive decay of uranium (U), thorium (Th) and potassium (K), and cosmic rays. In the case of OSL, this energy is released as luminescence (light) when quartz minerals are exposed to visible light (bleached), i.e. during sediment erosion, transport and deposition, or through stimulation in the laboratory. Feldspar minerals are also bleached by visible light. However, the luminescence produced by feldspar in the laboratory is stimulated using infrared and is referred to as Infrared Stimulated Luminescence (IRSL). Both dating techniques rely on the principles outlined in this section. However, due to differences in inherent mineral properties, the laboratory treatments for each mineral are different. During the last decade, OSL dating of quartz has become routine and will be the focus of this paper.

In its most simplistic form, the amount of luminescence released by quartz and feldspar when exposed to light is a function of the total radiation delivered to the mineral from the surrounding environment and the amount of time elapsed since bleaching (Fig.

1). This principle is expressed in the 'age' equation (Eq. 1) (Aitken, 1998), where, equivalent dose (D_e) is the radiation dose delivered to the mineral grains in the laboratory to stimulate luminescence (i.e. equal to the dose acquired in the natural environment since the last bleaching event), and dose-rate is the rate at which ionising energy is delivered from background radiation. The SI unit for dose is Gray (Gy) which is a measure of how much energy is absorbed by a sample in joules per kilogram ($J\ kg^{-1}$). The methodologies for determining D_e and dose-rate are outlined later in this paper.

(Eq. 1)

$$\text{Age (a)} = \frac{\text{equivalent dose } (D_e)\ (\text{Gy})}{\text{dose-rate (Gy/a)}}$$

Age range

The range of ages that can be obtained using luminescence dating is site specific and depends on the nature of the sediment (inherent mineral properties and sedimentary history) and the dose-rate received in the

environment. The lower age limit of luminescence dating is in the range of years (see Madsen and Murray, 2009 for a review), e.g. 10 ± 3 a for modern dunes in Denmark (Madsen et al., 2007). The upper age limit depends on the dose required to saturate the luminescence signal which is a function of the minerals capacity to store charge and the dose-rate received. Routine OSL dating of quartz can give ages up to 150 ka (Stokes, 1999). Where the dose-rate is low and/or the saturation dose is high, there is the potential to extend this upper age limit towards hundreds of thousands of years (e.g. 107.8 ± 5.2 ka: Mellett et al., 2012, and 507 ± 41 ka: Watanuki et al., 2005). Quartz typically saturates at a much lower dose than feldspar. However, the dose-rate of potassium-rich feldspars can be high due to internal dose contribution from the decay of radioactive isotopes within the crystal lattice (Adamiec and Aitken 1998). Given the variability outlined above, an age range for luminescence dating is difficult to define and often only reveals itself when samples are being analysed in the laboratory.

Sample selection

Choosing the right sediments

The most suitable sediments for luminescence dating are those that have been exposed to sufficient daylight to enable bleaching and resetting of the luminescence signal. This can be achieved in a variety of terrestrial and shallow water environments making luminescence dating one of the most widely applicable chronometric tools used in geomorphology. The first principles of OSL dating were developed using aeolian sediments from low latitude desert environments where insolation levels are high and there is increased likelihood that sediments will undergo repeated erosion-deposition cycles (see Wintle, 1993 for a review). However, improved understanding of mineral characteristics and bleaching regimes in a variety of sedimentary environments (see Rhodes, 2011 pp 468-470) means it is now possible to produce reliable luminescence ages from sediments deposited in fluvial, coastal and shallow marine, glaciofluvial, periglacial and colluvial environments (see Wallinga, 2002; Jacobs, 2008; Thrasher et al., 2009a; Bateman, 2008; Fuchs and Lang, 2009 for reviews

respectively). When making decisions about the most suitable sediments for luminescence dating it is vital to have an independent understanding of depositional history obtained through lithological, stratigraphic and morphological analyses.

Sand or silt sized sediments are required for luminescence dating. Grain size fractions 4-11 µm (silt) and ~63-250 µm (sand) should be targeted to account for differences in the laboratory dose-rate received by a sample according to grain size (Aitken, 1985). Further, it is advisable to avoid sediments that are rich in organic matter or carbonates as they may have received heterogeneous dose-rates.

Quartz vs feldspar?

When quartz or feldspar minerals are exposed to light the luminescence signal is released over a short period of time (typically 10s of seconds). The amount of time required to remove this signal and the style of signal decay is mineral dependent (Fig. 2). Quartz bleaches more easily than feldspar when exposed to natural light (Godfrey-Smith et al., 1988). Therefore, quartz is the preferred mineral when dating environments where insufficient bleaching, and hence age overestimation, may present a problem (e.g. Wallinga et al., 2001; Jain et al., 2004). In environments where quartz has been eroded only recently, low sensitivity can hinder reliable age estimates (Sawakuchi et al., 2011) and feldspar may be better suited. However, feldspars are vulnerable to the effect of anomalous fading (Wintle, 1973) and can underestimate ages as a result. Methodological advancements such as the development of single-grain OSL dating (Duller, 2008a) have improved the reliability of ages obtained from environments where incomplete bleaching is a problem. However, it is pertinent to consider depositional history and local mineralogy when selecting the most suitable mineral for luminescence dating.

Sampling methodology

The number and location of samples for luminescence dating is governed by the geomorphic problem that is being addressed and is therefore site specific. Despite this, there are a number of criteria that must be
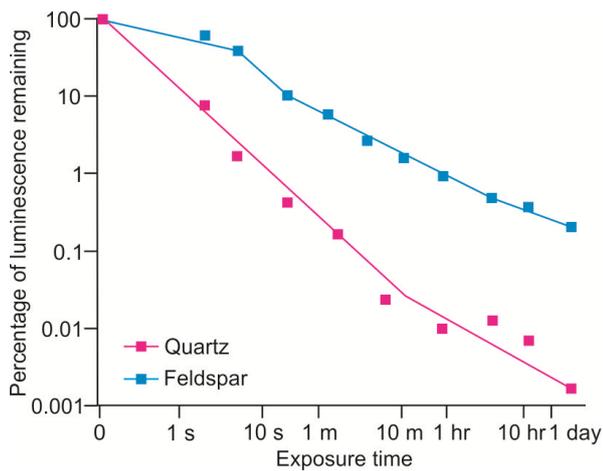
Figure 2: Reduction of Quartz and Feldspar luminescence signals during exposure to light (redrawn from Duller et al., 2008b). Note the luminescence is released much faster in quartz when compared to feldspar.

met to ensure luminescence ages from sediments are reliable for dating geomorphic events or processes. Firstly, it is important to take a sample from the centre of a lithofacies or stratigraphic unit (e.g. A and B Fig. 3b.) to avoid heterogeneity in the dose rate (e.g. C Fig. 3b). Disturbed ground that shows evidence of bioturbation or water leaching (e.g. D Fig. 3b) should be avoided to ensure no post-depositional mixing or significant changes in water content as these can influence the accuracy of the age produced. Further, an understanding of the sediment composition surrounding the sample is vital for determining dose rate. Therefore, sampling at the base of sections (or cores) where the underlying geology is unknown should be avoided (e.g. E Fig. 3b). To test the reliability and chronostratigraphic significance of ages where no independent chronological tool is available (e.g. radiocarbon dating), multiple samples within a lithofacies or stratigraphic unit (e.g. B Fig. 3b) may prove useful.

When sampling from cores the above criteria also apply. However, further care must be employed to avoid sampling sediment in close proximity (approximately 1 cm) to the core barrel or casing as this may have been disturbed during the coring process (Fig. 4a). It is advisable to avoid areas in the core where there is visible evidence of disturbance.

After choosing sampling locations, the most important methodological constraint is to recover the sediment and transport it to the laboratory without exposure to daylight. If sampling using coring apparatus, this can be achieved using opaque core liners and subsequent sub-sampling under safe light conditions in the laboratory whilst ensuring exposed sediment from the top and bottom of the core is discarded. When sampling exposed sediment sections (e.g. Fig 3a), an opaque PVC or metal cylinder is hammered into the face of the exposed section until it is completely filled with sediment (N.B. the diameter and length of cylinder depends on the lithofacies being sampled) (Fig. 4b). The cylinder is then excavated and sealed with black opaque plastic liners to ensure minimum exposure to daylight and transported to the laboratory. In some cases, to avoid unnecessary exposure to daylight an opaque fabric or liner can be draped over the sampling location whilst the sediment is being extracted (Fig. 4c).
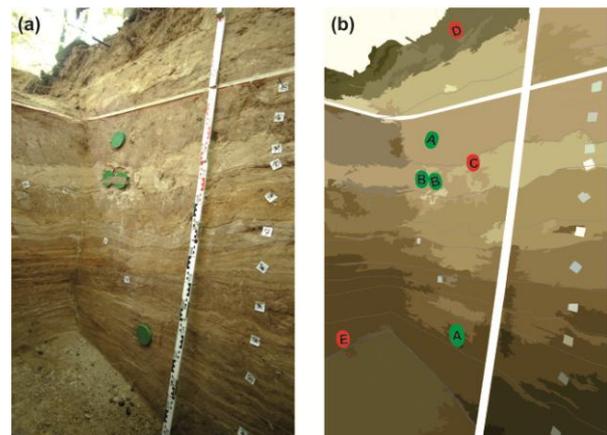

Figure 3: (a) Luminescence sampling of an excavated stratigraphic section. Photograph courtesy of Andreas Lang. (b) schematic stratigraphic section showing ideal sample locations (green) and unsuitable sample locations (red) as discussed in the main text.

The above section outlines procedures for sampling material for D_e determination (one half of the age equation). An additional bulk sample is required to determine dose-rate if in situ gamma spectrometry is not being undertaken (see the next section). This sample can be excavated whilst exposed to daylight (either from the exposed section or a core) and should be taken from the area directly surrounding the original sample to ensure an accurate representation of

environmental dose. Further, a sub-sample of sediment should be extracted to determine water content (an important component of the dose-rate calculation). These samples must be stored in airtight bags and are best kept refrigerated to ensure limited evaporation.
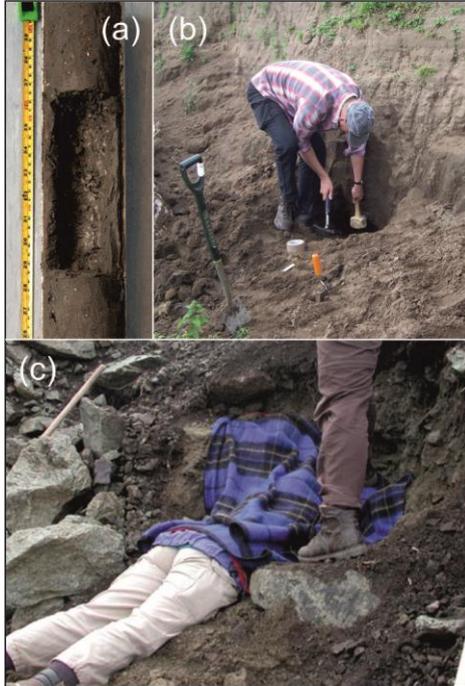

Figure 4: (a) An example of a luminescence sample taken from a core. (b) Sampling an exposed section by hammering a cylinder into the face. Photograph courtesy of Richard Chiverrell. (c) An opaque fabric or liner can be used to minimise expose to daylight. Photograph courtesy of Barbara Mauz.

Dose-rate determination

To obtain an age using luminescence dating, the age equation (Eq. 1.) requires a measure of the radiation received by the sample per year i.e. the dose rate. There are two major sources of ionising radiation in the environment that need to be determined; (i) the radioactive decay of radionuclides U, Th and K and their daughter nuclides, and; (ii) cosmic rays.

Annual dose received from radionuclides

This can be achieved by either; (i) measuring the concentration of radioactive elements in the sediment then calculating the dose-rate using the conversion factors outlined in Adamiec and Aitken (1998), or; (ii) measuring the radioactivity directly by counting the

emissions of alpha, beta and gamma using field or laboratory spectrometers. Both methodologies assume the dose-rate has not changed significantly since burial. See Duller (2008b) for a more thorough review of these techniques.

Measuring dose-rate in the laboratory as outlined above does not account for the presence of water within sediment pore spaces which absorbs radiation. To correct for this effect, water content needs to be determined and incorporated into the dose-rate calculation (Aitken, 1998). To account for fluctuations in water content that may have occurred during burial, an understanding of geological history (e.g. changes in climate or water table) is required to constrain the variability (e.g. Mellett et al., 2012). Finally, it is also important to assess if disequilibrium in the U decay series is present (see Olley et al., 1996) to ensure high degrees of accuracy are met when determining dose rate.

Annual dose received from cosmic rays

The dose received from cosmic rays is relatively low when compared to that received from radionuclides. However, in low dose environments and at high altitudes and latitudes, the cosmic dose contribution can be significant. Cosmic dose varies with longitude, latitude, altitude and burial depth and can be calculated using the equations outlined in Prescott and Hutton (1994).

Equivalent dose (D_e) determination

Determining equivalent dose (D_e) is arguably the most time consuming stage in luminescence dating. At this point in the dating process geomorphologists should be working in close collaboration with a luminescence laboratory that will provide advice and guidance about independent laboratory practises and the most suitable techniques according to individual samples. This section gives a brief introduction into conventional procedures used to determine D_e.

Extracting quartz or feldspar

The procedures for extracting quartz or feldspar from a bulk sample will depend on the individual laboratory. Typically, samples

are separated to the desired grain size (sand or silt) then organic matter and calcium carbonate are removed using H_2O_2 and HCl. Quartz, feldspar and heavy minerals are separated according to density using heavy liquid or magnetic procedures. If using quartz for OSL dating, the resulting fraction is etched using HF (to remove the outer part of the grain affected by alpha radiation). The above are carried out under subdued red-light conditions.

The single-aliquot-regenerative-dose (SAR) protocol

D_e represents the amount of dose accumulated in the environment since the last exposure of the mineral to daylight. The SAR protocol (Murray and Roberts, 1998; Murray and Wintle, 2000) is a procedure to measure D_e whilst accounting for changes in the mineral (sensitivity change) that can occur due to irradiation, heating or light stimulation in the laboratory. The SAR protocol is widely used to determine D_e in quartz (Wintle and Murray 2006). The protocol measures the natural luminescence (L_N) produced by the mineral when stimulated by light (i.e. it completely bleaches the signal) and then effectively tries to recreate its dose history by giving incremental doses of radiation (Dose 1, Dose 2, Dose 3 Fig. 5) and regenerating the luminescence accumulated. After each given dose, the luminescence is measured $(L_1 \, L_2 \, L_3$ Fig. 5). With the exception of the first cycle (i.e. L_N), each time luminescence is measured during the SAR protocol, it is corrected for sensitivity changes by taking the ratio of the luminescence signal (L_X) to the response to a fixed test dose (T_X). A dose response curve (Fig. 5) is constructed by plotting the sensitivity corrected luminescence (e.g. L_X/T_X). D_e is determined at the point on the X axis where the natural luminescence (L_N) intercepts the dose response curve (Fig. 5). Determining ages using the SAR protocol is considered both accurate and precise when compared to independent age controls (e.g. Rittenour, 2008; Rhodes, 2011). However, it is important to ensure the assumptions inherent to the protocol (Murray and Wintle 2000; Wintle and Murray 2006) are met which can be achieved by carrying out a number of tests in the laboratory (see Duller et al., 2008b for a review).
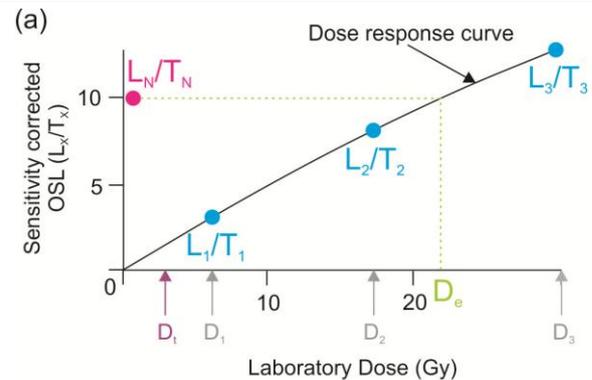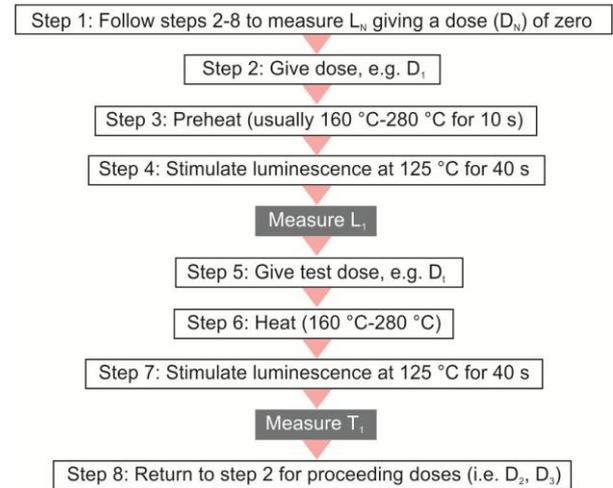

Figure 5: (a) Determination of D_e using the SAR protocol where the natural dose (L_N) and regenerative dose (L_X) are corrected for sensitivity changes using a small test dose (T_X). (b) Generalized quartz SAR protocol (after Murray and Wintle, 2000).

Single aliquot or single grain?

An aliquot is a subsample of grains taken from the bulk sample. In luminescence dating aliquots are mounted on 1 cm diameter discs for measurement. The number of grains present on these discs depends on the area of the aliquot covered (e.g. 1 mm to 10 mm) and the grain size of the subsample (Duller et al., 2008a). For example, a 1 mm aliquot of grain size 100 µm will comprise on average 10 individual grains whilst a 1 mm aliquot of grain size 300 µm will hold only 1 or 2 grains (Duller, 2008a). When using single aliquots, D_e is the sum of the luminescence emitted by all grains present on the disc (Fig. 6a). However, not all grains within the aliquot produce the same, if any, luminescence in response to the same dose (e.g. Duller et al., 2000), thus reducing the precision of D_e values. Therefore, understanding the number of grains present within an aliquot and how

many of those grains produce luminescence is important.

Measurement of multiple single aliquots can result in a large spread (overdispersion) of D_e values (Fig. 6b) which can be a result of (i) variations in the degree of bleaching, or; (ii) mixing of grains during or post-deposition. Typically, using the SAR protocol large numbers of aliquots are measured and the D_e is determined using a variety of statistical analyses (see the section below). However, in some cases the spread in D_e values is too large due poor bleaching and single-grain techniques are better suited (e.g. Olley et al., 2004; Duller, 2006).

Advancements in instrumentation have made it feasible to measure the OSL signal from single grains thus demonstrating the variability in dose distribution between grains from the same sample. This technique is most suitable for samples where exposure of grains to daylight is variable (e.g. fluvial and glaciogenic). However, single grain measurements are time intensive and typically <5% of grains dominate the luminescence signal giving results that are similar to those obtained through single aliquots (Duller, 2008b). The decision to use small aliquots or single grains is sample specific and should be undertaken in collaboration with a laboratory.

Statistical analyses

There is typically a degree of variability in the D_e values from different aliquots or different grains within the same sample. A number of statistical methods exist to identify a dominant component related to the last bleaching event within complex distributions (e.g. Fig. 6c) and hence improve the reliability of age estimates. These include the Central Age Model (CAM) and The Minimum Age Model (MAM) (Galbraith et al., 1999), and the Finite Mixture Model (Galbraith and Green, 1990). The most appropriate age model is sample dependent and can be assessed according to a number of statistical criteria (see Bailey and Arnold, 2006; Rodnight et al., 2006; Rowan et al., 2012; Thrasher et al., 2009b for examples). However, these criteria are not inclusive and statistical treatment can be governed by the geological context of individual samples (Galbraith and Roberts, 2012). After statistical assessment of D_e

distributions, the most appropriate D_e has been established and is available for input into the age equation (Eq. 1).
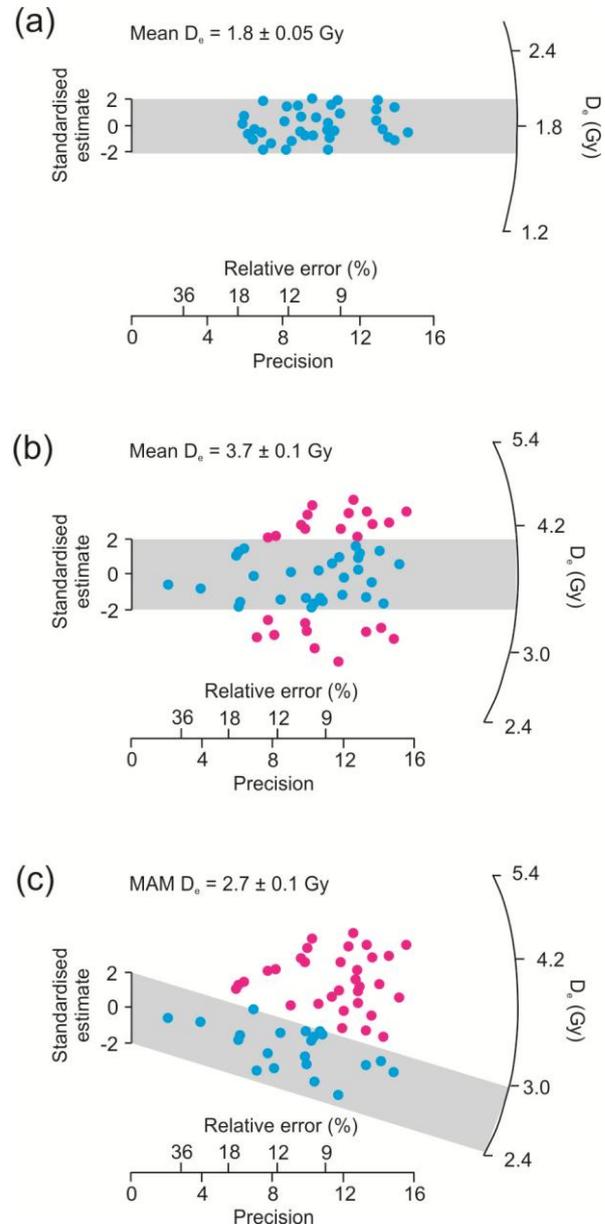

Figure 6: Radial plots of D_e values determined using SAR of quartz (modified from Mauz et al., 2010). Refer to Galbraith and Roberts (2012) for an explanation on how to interpret radial plots. (a) D_e is determined by taking the arithmetic mean of values shown in blue, those in pink are rejected. (b) For this sample, due to a broad distribution of values the arithmetic mean is not an appropriate representation of D_e (c) Statistical analysis of the distribution shown in (b) led to the application of the Minimum Age Model (MAM) to determine D_e for the age calculation. Note the difference in D_e determined from the same sample using arithmetic mean (b) and MAM (c).

Recent advances and applications

Age range extension

Age range extension, particularly the upper limit has been a priority amongst researchers over recent years. The development of experimental procedures that remove the effect of anomalous fading in feldspar minerals (post IR-IRSL) (Thomsen et al., 2008; Buylaert et al., 2009) means it may be possible to target the luminescence signal corresponding to the dose received during burial without incorporating less stable components of the signal. As the saturation level of feldspar is much higher than quartz, this methodological development is pushing the upper limit with the potential to extend the age range by a factor of 4-5 (Buylaert et al., 2012).

Dating sediments from challenging environments

The range of environments that can be dated using luminescense dating and examples of the sucessful application of the technique are extensive (see Rhodes, 2011 for a summary). Aeolian and fluvial sediments are commonly dated as bleaching regimes in these environments are relativley well understood (e.g. Fujioka et al., 2009; Wallinga et al., 2010). However, in many marine, coastal, colluvial and glacigenic environments complete zeroing of the luminescsene signal can be problamatic. Despite this, ongoing developments in luminescsense dating techniques are allowing the production of reliable ages from these more challenging environments (e.g. Murari et al., 2007; Mauz et al., 2010; Thrasher et al., 2009b).

Dating gravel rich sediment is problematic as it is difficult to confidently estimate dose-rate in hetrogeneous sediments. However, it has been demonstrated that when gravels are dominated by inert lithologies (such as chalk), sand within the gravel matrix can provide reliable age estimates (e.g. Mellett et al., 2012). A further advancement has been made in dating gravel clasts directly (Simms et al., 2012). Whilst this application is in its pilot stage, if successful it has the potential to extend the range of landforms that can be dated using OSL.

Thermochronology

A limitation of luminescense dating is that as it dates sediment directly, it provides an age of depositional processes only. In geomorphology, erosional regimes are equally important in shaping the landscape and there is a need to date bedrock surfaces. The development of OSL-thermochronology, a technique that measures the exhumation rate of bedrock according to its cooling history, has been used to measure denudation rates in New Zealand (Herman et al., 2010). However, the use of OSL dating in determining the timing of event scale erosion processes directly i.e. fluvial incision of bedrock, has yet to be developed.

Rapid age determination

Luminescense dating is a time and labour intensive technique. The time taken between sampling in the field and obtaining an age can be up to 6 months depending on the individual laboratory and sample characteristics. In geomorphology, this can be problemtatic when undertaking restricted field campaigns. With this in mind, development of the 'range finder' protocol has enabled rapid approximations of OSL ages which can be used to guide sampling strategies and establish the resolution of dating capaigns (Roberts et al., 2009; Durcan et al., 2010).

Finally, the use of portable OSL readers, whilst they do not give an absolute age, can improve the speed of luminescense dating by establishing luminescence charactersitics in the field (e.g. Munyikwa et al., 2012). This procedure identifies what sediments are dateable in the feild helping laboartories avoid intensive analyses on samples that utimatley do not produce any data.

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