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## Challenges involved in obtaining luminescence ages for long records of aridity: Examples from the Arabian Peninsula

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#### ABSTRACT

In the last 35 years luminescence dating has provided a large database of ages for the deposition of dunes in desert environments. Over 380 ages have been generated for dunes from the Arabian Peninsula and demonstrate episodic dune deposition in the late Quaternary, but give a less clear pattern prior to ~50 ka. Interpreting databases of luminescence ages faces two issues. First, the precision of luminescence ages in the last 50 ka is between 5 and 10%, but for older ages the uncertainties may be much larger as luminescence signals reach saturation. Second, different luminescence signals from quartz and from feldspar have been used over the last 35 years as the method has developed and expanded. These different signals have different saturation limits and different rates at which they are reset by exposure to daylight at deposition. Approaches which focus on the most light sensitive signals (e.g. quartz OSL) are better suited to dating recent events, while those which show growth of the luminescence signal over the largest dose range (e.g. the thermally transferred OSL (TT-OSL) signal from quartz and the post-infrared infrared stimulated luminescence (pIR-IRSL) from feldspars) have the potential to date much older events.

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#### 1. Introduction

Luminescence dating methods have revolutionised our understanding of the dynamics of desert dunes by providing numerical chronologies where methods such as radiocarbon are frequently not applicable. Luminescence dating methods have allowed studies of the rates at which dunes form and move (e.g. Bristow et al., 2007), and the timing of periods of stabilisation of dunes (Leighton et al., 2014). Luminescence gives information about the timing of dune stabilisation, though whether such records indicate changes in aridity, sediment supply, or other factors (Kocurek, 1998) is still a matter of debate (Thomas and Wiggs, 2008; Chase, 2009; Thomas and Burrough, this volume). As part of the INQUA Dune Atlas project (Lancaster et al. this volume), 385 luminescence ages (as of 23/11/2015) from the Arabian Peninsula have been compiled.

One of the challenges in compiling a database of ages is that luminescence methods have developed dramatically in the 34 years since Singhvi et al. (1982) working in the Thar Desert of India became the first to apply luminescence dating to desert dunes. Luminescence dating methods rely upon the growth of the trapped charge population within mineral grains as a result of exposure to ionising radiation in the natural environment (Aitken, 1985; Duller, 2008). In the laboratory the concentration of trapped charge can be measured by stimulating the grains either by heat or light to generate luminescence (thermoluminescence (TL) or optically stimulated luminescence (OSL)). The magnitude of the luminescence signal obtained from irradiation during burial is compared in the laboratory with the magnitude of the luminescence signal arising from irradiation using a laboratory radioactive source of known strength. This sequence of measurements yields the equivalent dose  $(D_e)$ , an estimate of the radiation dose the sample was exposed to since the last exposure of the sample to daylight. To calculate an age, it is also necessary to measure the radioactivity of the sample, and this is termed the dose rate  $(D_r)$ . The age is calculated by dividing D<sub>e</sub> by D<sub>r</sub>.

$$Age(ka) = \frac{Equivalent \ Dose \ (D_e)}{Dose \ Rate \ (D_r)}$$
(1)

Although the basis of luminescence dating is the same today as it was in 1982, there have been major changes in both the method of stimulating a luminescence signal (thermoluminescence (TL), infrared stimulated luminescence (IRSL), post-infrared infrared stimulated luminescence (pIR-IRSL) or optically stimulated luminescence (OSL)), and the different methods of equivalent dose

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determination (multiple aliquot methods versus single aliquot methods, Duller (2004)). Thus there is not a single 'luminescence' method but rather a family of different methods that share some common features, but also have their own specific traits. This paper briefly explains the difference between these methods and the impact that these changes may have had upon the quality and reliability of the ages generated. The paper also explores the impact that saturation of these different luminescence signals has upon the accuracy and precision of the ages that are generated. Examples from the INQUA dunes atlas are taken primarily from the Arabian region. Here, as in many of the major deserts of the world, the record of dune activity is likely to be a very long one, beyond the range of current luminescence dating methods. Inevitably samples are collected that push the limits of current luminescence methods, and this paper aims to explore the potential challenges involved in the analysis of such old samples.

#### 2. Methods of luminescence measurement

#### 2.1. Thermoluminescence: TL

The pioneering work of Singhvi et al. (1982) was the first to use luminescence dating methods to determine the period of time since the mineral grains making up the desert dunes had been last exposed to daylight. Singhvi et al. (1982) measured the thermoluminescence (TL) signal from quartz separated from sediments collected from dunes in Rajasthan. The TL signal from quartz had first been used to date the timing of firing of pottery (Wintle, 2008), where the event being dated was heating of the quartz grains. Firing is able to remove all of the TL signal below a specified temperature (typically about 400 °C), but exposure of quartz grains to daylight is only able to remove part of the TL signal. To be useful for dating, the method that was developed had to estimate the amount of the TL signal that was not removed by exposure to daylight and remained at deposition – this is known as the residual signal. This residual signal was then subtracted from the measured signal to allow an age estimate to be obtained (Singhvi et al., 1982; Duller, 1996). The prolonged exposure of mineral grains to daylight prior to deposition made desert dunes an ideal target for this newly developed method, but the need for residual subtraction made it difficult to date very young sediments.

#### 2.2. Optically stimulated luminescence: OSL and IRSL

In 1985 Huntley and co-workers published details of an alternative method of obtaining a luminescence signal from mineral grains. Instead of heating the grains to obtain a TL signal, the grains were exposed to light in a restricted wavelength range (514 nm in their instrument), and the resulting optically stimulated luminescence (OSL) emission could be used to determine the  $D_e$ . This method had the advantage that it only measured the part of the luminescence signal that was most sensitive to daylight, and thus there was no need to remove a residual signal. For desert dunes this made it possible to generate ages covering very recent periods of time, providing high temporal resolution for events in the last few millennia (Thomas et al., 1997).

The Research Laboratory for Archaeology and the History of Art in Oxford obtained an argon-ion laser like that used by Huntley et al. (1985) and undertook a great deal of research on desert dunes (e.g. Stokes et al., 1997). However, the expense and complexity of these lasers meant that no other laboratories made significant use of them, severely limiting the adoption of this method. Hütt et al. (1988) provided an alternative when they showed that it was possible to stimulate an OSL signal from feldspars using infrared light (~880 nm). Light emitting diodes of the type used in domestic remote controls for televisions provided an affordable and easily controlled source of IR, and many laboratories either built their own instruments or purchased affordable commercial systems. The application of infrared stimulated luminescence (IRSL) methods was also boosted by the development of single aliquot methods of  $D_e$  determination (Duller, 1991).

However, a major challenge for IRSL measurements of feldspars was the phenomenon of anomalous fading (Wintle, 1973). This term describes the loss of trapped charge at a rate that is much faster than would be predicted from a simple interpretation of the physical characteristics of the crystal defects at which charge is trapped. If anomalous fading occurs then it will lead to a measurement of D<sub>e</sub> that is smaller than would be expected, and hence an underestimate of the age. A recurring point of discussion through the 1990's and early 2000's was whether anomalous fading was a universal phenomenon affecting all types of feldspars (e.g. Huntley and Lamothe, 2001), or whether there were some feldspars that were not affected. Some authors chose to attempt to measure the rate of anomalous fading and correct for it, whilst others reported that they did not observe any fading, and thus did not correct. In Arabia, the very long sequence from Oman reported by Preusser et al. (2002; records ARBL0027-98) was dated using IRSL measurements, and they reported that they could not observe any fading. Until recently there has been little standardisation in how measurements have been made to assess the degree to which a sample of feldspar is affected by anomalous fading. In recent years it has become commonplace to measure the extent of fading and characterise it by a g-value (Huntley and Lamothe, 2001). g-values are quoted as a percentage figure and values in excess of ~1.5% per decade are sufficiently large that they require some form of correction (Buylaert et al., 2012). Unfortunately many earlier publications do not express their fading measurements in this way, making it difficult to assess whether fading was significant or not.

While infrared (IR) light emitting diodes are able to stimulate an IRSL signal from feldspars, quartz requires a different light source. The Ar-ion laser originally used by Huntley et al. (1985) emitted light at 514 nm. In the late 1990's green, and then blue, light emitting diodes became commercially available, and these were ideally suited for generating an OSL signal from quartz (Galloway, 1992; Bøtter-Jensen et al., 1999). Commercial instruments based especially upon blue LEDs rapidly became standard in luminescence laboratories, and together with the development of single aliquot methods of  $D_e$  determination (see below), this became a very standard method of analysis for desert dunes. The current database is now dominated by luminescence ages measured on quartz grains using the OSL signal generated using blue light emitting diodes.

# 3. Methods of equivalent dose determination: multiple aliquot and single aliquot

Calculation of the equivalent dose ( $D_e$ ) from a sample involves comparing the intensity of the luminescence signal resulting from trapped charge acquired during burial of the sediment with the luminescence resulting from exposure of the sample to laboratory irradiations from radiation sources that are precisely calibrated (Duller, 2008; Rhodes, 2011). Early luminescence work used many different sub-samples of each sample to determine a single value of  $D_e$  and these are termed multiple aliquot methods. Each subsample, called an aliquot, was given a different treatment: some were used to measure the luminescence arising from irradiation in nature, while others were used to measure the response to laboratory irradiation, or the response to exposure to daylight. All of these measurements on multiple aliquots were then combined together to generate a single value of  $D_e$ . Download English Version:

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