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Luminescence Chronology

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1. Introduction

Luminescence dating is a collective term for dating methods that encompass thermoluminescence (TL) and optically stimulated luminescence (OSL) dating techniques. OSL is also less commonly referred to as optical dating [1], photon stimulated luminescence dating or photoluminescence dating [2]. Luminescence dating methods are based on the ability of some mineral grains to absorb and store energy from environmental ionizing radiation emanating from the immediate surroundings of the mineral grains as well as from cosmic radiation. When stimulated these minerals, generally referred to as dosimeters [3], will release the stored energy in the form of visible light; hence the term luminescence. Measuring the energy and determining the rate at which the energy accumulated allows an age representing the time that has elapsed since the energy began accumulating to be determined. Stimulation of energy release using heat is termed TL while stimulation using light is referred to as OSL. The age range of luminescence methods generally spans from a few decades to about 100,000 years, though ages exceeding several hundred thousand years have been reported in some studies [for example, 4, 5]. In addition, there are dating protocols that are currently under investigation that, if successful, could extend the range even further [6]. Thus, the method is useful for dating Late Quaternary events and, not only does it provide chronology beyond the range that can be attained using radiocarbon methods, but it offers an alternative chronometer in settings where no carbon bearing material can be found.

This chapter aims to acquaint readers who are not familiar with luminescence dating methods with the basics of the techniques. It is not intended to be used as a manual but rather as an introductory primer that brings awareness about the principles behind the dating methods, their practical aspects, as well as their applications. Accordingly, the chapter comprises nine sections. Following the introduction in the first section which briefly lays out the historical development of luminescence dating, the second section examines the principles of the dating methods. This is followed by a discussion of sample stimulation mechanisms and basic

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measurement equipment used in luminescence dating in the third section. Luminescence properties of different minerals are examined in the fourth section. In the fifth section, methods used to determine the energy stored within mineral grains (paleodose) as well as the rate at which the energy accumulates (dose rate) are explored. In the sixth section, practical aspects pertaining to sample collection and laboratory preparation for analysis are discussed after which the types of materials that can be dated using luminescence materials are examined in the seventh section. To illustrate the multifaceted character of some luminescence dating studies, the eighth section presents a case study that uses the chronology of postglacial eolian dune deposition in western Canada to constrain the timing of Late Pleistocene deglaciation in the region. The chapter concludes in the ninth section with a look at current and potential future developments in luminescence dating.

1.1. The historical development of luminescence dating

The history of the development of luminescence dating spans the last six decades and it began with experimental applications of the phenomenon of thermoluminescence, which is the emission of light when materials are heated to temperatures below those of incandescence. One of the earliest documented suggestions of the possibility of using thermoluminescence to measure time in archaeology was by Daniels et al. [7] who in 1953 proposed using thermoluminescence observed from ancient pottery artifacts that had previously been fired as a measure of their age [8]. A few years later, the application of thermoluminescence to date pottery was discussed by Kennedy and Knopff [9] and technical aspects of measurements that would be employed for dating were described by Grögler et al. [10]. Notably the application of thermoluminescence in geology to study ages of carbonates [11] and lava flows [12] was already being discussed. An early study that tested the application of thermoluminescence dating on pottery was reported by Aitken et al. [8] when in 1964 they applied the method on pottery sherds ranging in age from around 1,000–8,000 years and collected from sites spread over a large area. Results indicated that the luminescence ages were linearly proportional to radiocarbon chronology from contemporaneous materials. Subsequently, developments throughout the rest of the 1960s and the 1970s saw improvements in dating procedures that used thermoluminescence in archaeological applications [3].

Incidentally, parallel developments in the former Soviet Union during the late 1960s and early 1970s began seeing the tentative application of thermoluminescence dating on unburnt Quaternary sediments [13, 14] when it was noted that older sediments returned higher TL signals than younger ones. Dating of unburnt sediments was based on the recognition that stored energy in sediment grains could also be depleted by exposure to sunlight as opposed to heating that occurs in fired artifacts. In the West, attempts to apply thermoluminescence on unburnt sediments appear to have begun around the end of the 1970s [15] including attempts by Wintle and Huntley [16, 17] to apply the technique on deep sea sediments. Accurate sediment ages from TL dating, however, remained elusive since the optimal conditions for solar resetting were not yet fully understood [18]. Huntley [18] conducted some of the earliest studies investigating the most appropriate conditions for solar bleaching. In a related development, sediment dating using luminescence methods progressed rapidly when it was noted

[1] that light could also be used to stimulate energy release from sediments during measurement as opposed to heating. This led to the birth of OSL dating and throughout the rest of the 1980s [19, 20], the 1990s [21-25] and the early part of the following decade, improved protocols were introduced [26, 27]) and OSL dating equipment refined. All these developments saw luminescence dating emerge as a robust method for dating clastic Quaternary sediments, especially eolian deposits. A comprehensive historical account of the first 50 yrs of luminescence dating is provided by Wintle [28].

2. Principles of luminescence dating

2.1. Luminescent materials

Some dielectric materials (insulators) that include many minerals such as quartz, feldspar, zircon and calcite have the ability to store energy in their crystal lattices that emanates from ionizing radiation. In natural geological settings, such ionizing radiation (for example, alpha, beta, and gamma radiation) occurs naturally within the immediate surroundings of the geological materials while a small component is also contributed by cosmic radiation. When stimulated, the minerals will exhibit luminescence which essentially represents a release of the stored energy. Luminescence dating employs this phenomenon by measuring the energy stored in the mineral, called the paleodose, and dividing it by the rate at which the energy was received by the mineral in question. Hence, the basic age equation for luminescence dating is:

$$Luminescence\ Age = \frac{Paleodose}{Dose\ rate} \tag{1}$$

2.2. Electron trapping mechanisms

The exact mechanisms through which luminescence energy accumulates in the minerals are complex. However, it is thought that the energy is stored when electrons in the mineral crystal lattices are displaced from the valence band of their parent nuclei. Once detached, the electrons diffuse into the surroundings of lattice defects that act as electron traps. Such defects include a missing atom in the crystal lattice of the mineral, an atom out of its rightful place or the occurrence of impurity atoms in the lattice [29]. Importantly for dating purposes, the number of trapped electrons increases with the duration of exposure of the mineral to the ionising radiation. Fig. 1 is a depiction of an energy level diagram that is commonly used to visualise the trapping mechanism involved in luminescence. The depth of the trap (T) below the conduction band, indicated by 'E' [Fig. 1], is an indication of the efficacy of a given trap. Stable traps are those that can withstand perturbations such as lattice vibrations that could dislodge the electrons from their traps. Stimulation of the crystal lattice structure by heating to an appropriate temperature or by optical means using a suitable wavelength will excite the electrons out of the traps. Once expelled from the traps, the electrons diffuse within the crystal lattice until they come across another site that is attractive to electrons and these are referred to as recombination centres [29]. When electrons reach some recombination centres, energy is

emitted in the form of light and these are described as luminescence centres. Thermal stimulation would yield TL whereas optical stimulation would produce OSL. Importantly, the diffusion process is very rapid such that the time between stimulation and recombination can be treated as instantaneous. For a recombination centre to be effective, an electron must be missing from the site in the lattice, creating what is termed a hole. Holes are created in materials by ionising radiation. The intensity of the luminescence given out following stimulation is proportional to the number of trapped electrons and this is assumed to be proportional to the energy absorbed from the nuclear radiation [29]. Significant for dating applications, however, though the energy storage mechanisms might be the same for a given mineral, the sensitivity to radiation will vary between samples; a consideration that has huge implications for methodological approaches as will be shown later.
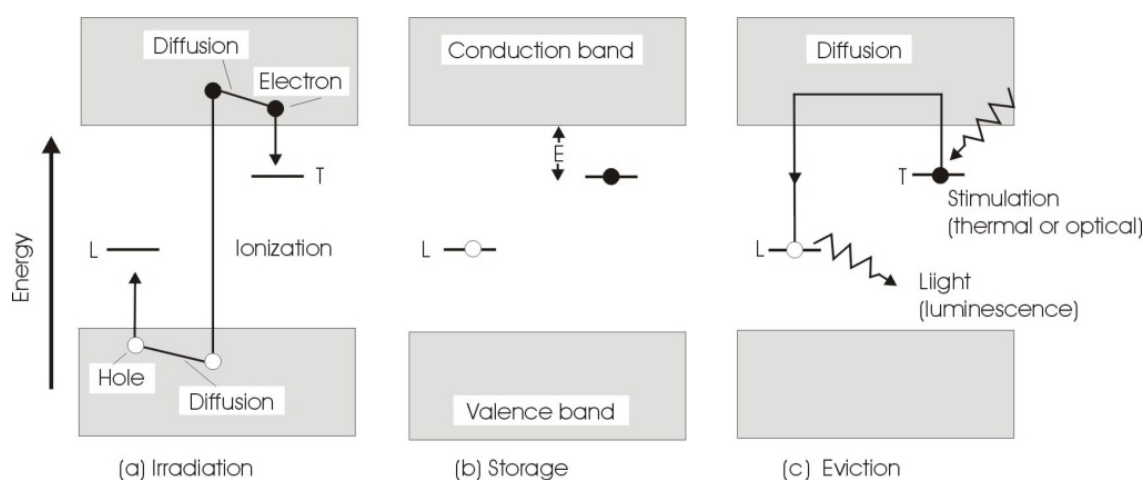

Figure 1. Energy Level diagram illustrating the creation of luminescence centres in crystal lattices through exposure to ionising radiation (redrawn from [29]). (a) Irradiation leads to electrons being expelled from their original site and diffuse within the lattice. (b) Electrons become trapped while holes of missing electrons become localised at particular centres. (c) Stimulation (by heat or light) results in electrons being evicted from the traps and diffusing until they meet a recombination centre. Light (luminescence) is emitted when luminescence centres are encountered by the electrons to give TL or OSL.

It is pertinent to note that the number of traps within the lattice of any dosimeter is not infinite and, hence, they will be exhausted following extended exposure to radiation, beyond which energy will not be stored efficiently. This is referred to as saturation. For dating purposes this is what defines the upper limit on the age beyond which samples cannot be dated using luminescence methods [29]. The exact age representing this upper limit will ultimately depend on the dose rate, with samples experiencing high dose rates having lower age limits.

2.3. Natural sources of ionizing radiation

For purposes of luminescence dating, natural sources of ionising radiation that contribute to the trapped energy in mineral grains are isotopes of uranium (^{238}U and ^{235}U) and thorium (^{232}Th) decay chains, potassium (^{40}K) and rubidium (^{87}Rb). These elements occur in natural materials in very low levels (around 3-10 parts per million for uranium and thorium and generally less

than 5% for potassium, where ^{40}K is one part in 10,000). However, collectively, their radioactive isotopes emit enough radiation to cause detectable luminescence for dating purposes. The radiation emitted includes alpha and beta particles as well as gamma radiation. Alpha particles have penetration ranges of 0.02 mm while beta and gamma rays have ranges of around 0.02 cm and 20 cm respectively [29]. The shorter distance travelled by alpha particles is because they are heavier and much more ionizing, which results in more rapid loss of energy as they knock atoms of materials through which they are traveling out of the way. Beta particles and gamma rays, on the other hand, tend to get scattered.

Besides the radiation from radioactive isotopes, an additional, albeit smaller, component to the energy received by the mineral grains is contributed by cosmic rays from outer space. The cosmic radiation comprises a soft and a hard component and once it reaches the earth's surface, the soft component is absorbed by the upper 50 cm of the near-surface substrate. Only the hard component can penetrate deeper and is of interest to luminescence dating. This hard component comprises muons mostly and at sea level it varies slightly with latitude, increasing in intensity by about 7% from the equator to latitude 40°. Above 40° latitude however, it remains constant up to the poles [29]. At altitudes higher than 1 km, the contribution from the hard component also increases significantly with both latitude and altitude. For dating purposes, special formulae have been developed for evaluating cosmic ray contribution to the dose rate [30].

3. Basic luminescence measurement equipment and sample stimulation mechanisms

The primary objective of TL or OSL measurements in dating studies is to ascertain the amount of energy that has accumulated in the mineral grains since the start of the event being dated. This energy is determined by stimulating the mineral grains using an appropriate mechanism and measuring the amount of light released. As already outlined, trapped electrons in luminescence dating can be evicted from their traps by heating, as is done in TL, or using light, in OSL methods. Fig. 2 illustrates the basic layout of equipment used to measure the luminescence. Typically, samples are placed on discs about 1 cm in diameter and then introduced into the machine in multiples on an appropriate sample holder. A servo-control mechanism moves the sample to the appropriate position for stimulation and measurement. Most modern luminescence measurement systems possess both thermal and optical stimulation capabilities. The luminescence signal from the sample is captured by the photomultiplier tube after going through optical filters.

For TL measurements, the filters help exclude infrared signals from the heating while allowing blue or violet signals. For OSL measurements, the filters reject wavelengths used for stimulation while usually allowing violet and near-ultraviolet wavelengths. The end of the photomultiplier tube closest to the sample is fitted with a photocathode that emits electrons when struck by light photons as a result of the photoelectric effect. Potential differences allow emitted electrons to be attracted by the first dynode in the photomultiplier. For each electron arriving

at the dynode, several are emitted and the process is repeated through the photomultiplier many times such that several millions of electrons reach the anode at the other end of the photomultiplier for each electron leaving the photocathode. These electrons give an easily detectable current pulse at the anode that is commensurate with the arrival of photons at the photocathode. The current pulses are then amplified and the output is presented as photon counts, representing the luminescence signal. As will be shown below, TL output is distinctly different from OSL output.
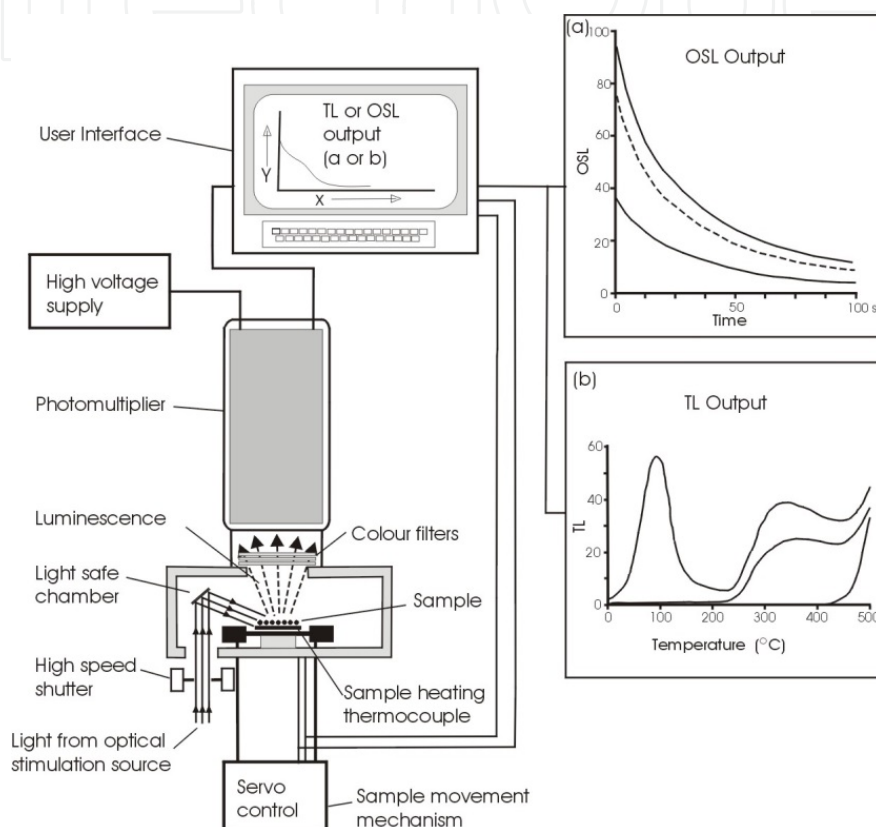

Figure 2. Basic features of a TL/OSL reader that can be used to measure luminescence signals using either heat or optical stimulation (modified after [31]).

3.1. Stimulation by heat (TL)

When stimulating using heat, a sample is heated rapidly at rates in the range of 20°C /s. Once a temperature commensurate with 'E' in Fig.1 and characteristic of a particular trap type is reached, electrons are rapidly evicted from the traps [29]. The temperature is represented by a peak in emission on a plot of the luminescence versus temperature referred to as the TL glow-curve. Continued heating will empty the traps and the luminescence given out will be proportional to the number of electrons trapped in the mineral grains since the beginning of the event being dated. Fig. 3 shows a glow-curve of a sample observed after the first heating. If a sample is heated for a second time immediately after the first heating, a different curve is observed. This second curve is the red-hot glow that is the incandescence given out by any

material when heated to a temperature high enough. Absent from this second heating will be the luminescence emanating from ionizing radiation that accrued since the last event that emptied the electrons from the traps.
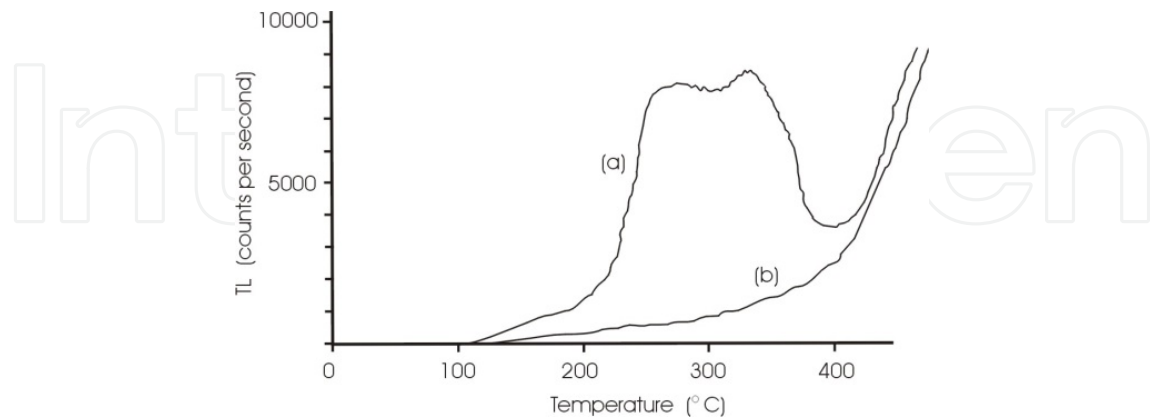

Figure 3. Examples of TL glow-curves showing (a) natural signal obtained from a mineral sample (quartz) during the first heating and (b) red-hot glow-curve obtained from a second heating (incandescence). Note that in curve (a), incandescence is also obtained during the first heating when the sample is heated above 400° C (redrawn from [31].

3.2. Optical Stimulation (OSL)

Optical stimulation of luminescence uses light of a particular wavelength (for example, blue, green, or near-infrared) to expel electrons from their traps. Notably, the rate at which these electrons are evicted directly depends on the rate at which the stimulating photons are received. The sensitivity of a given trap-type to photostimulation is also an important factor influencing the rate at which electrons leave their traps. In essence, the curve that depicts the emission follows an exponential decay (often referred to as a shine-down curve), with high emission rates in the beginning that gradually fall with continued stimulation (Fig. 4). If the process is continued, at a certain point, all electron traps that are sensitive to optical stimulation become exhausted. Integrating the number of photons released over the period of stimulation quantifies the luminescence of the sample and this should be commensurate with the sample's age [29].

Factors that influence the sensitivity of a trap type to electron eviction by light include characteristics of the trap as well as the wavelength of the stimulating light. Generally, shorter wavelengths are associated with faster eviction rates. Stable traps may require more energy than that available from some optical stimulation wavelengths and in such cases, thermal assistance can be used to bridge the gap. This enables longer wavelengths to be used for stimulation in cases where they would not be able to unassisted [29].

When selecting a stimulation wavelength for OSL measurements, it is important to select a wavelength that effectively allows separation to be made between the wavelength of the stimulating source and that of the emitted signal. As described earlier, filters are used to assist this process. Quartz and feldspar for instance, have strong emissions in the near-ultraviolet (365 nm) and violet (410 nm) respectively and filters used in either case are selected because

they have windows in the respective wavelength range. Wavelengths used for stimulation, therefore, should be excluded by the filters [29].
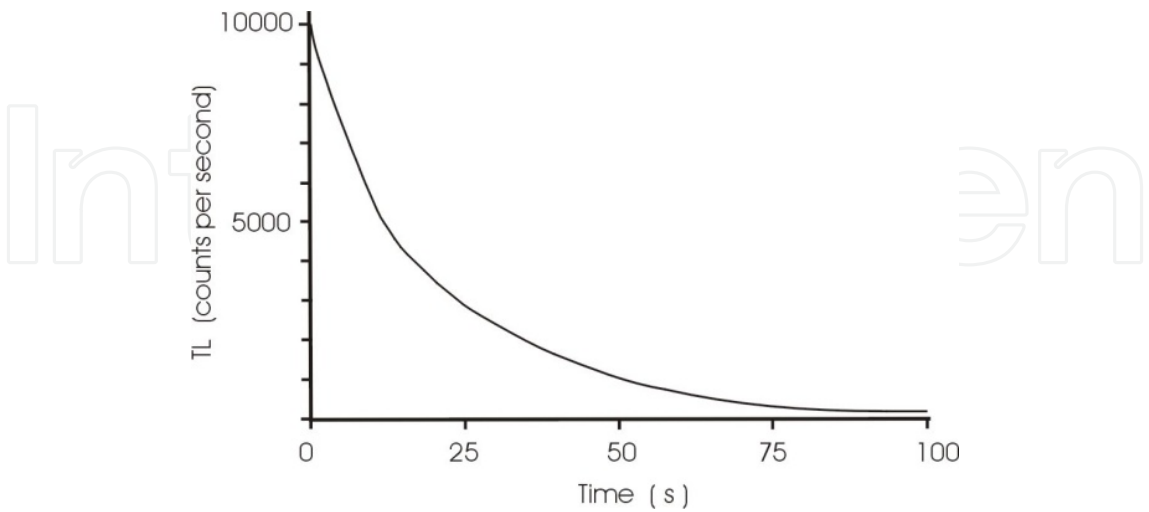

Figure 4. OSL shine-down curve for a hypothetical mineral stimulated using light for about 100 s (modified from [29]).

3.3. Advantages of OSL over TL

As will be shown later, advantages of OSL dating versus TL dating mainly apply to dating of sediments that have been zeroed by solar bleaching. Studies have demonstrated that solar bleaching of natural TL occurs much more slowly than OSL [29].
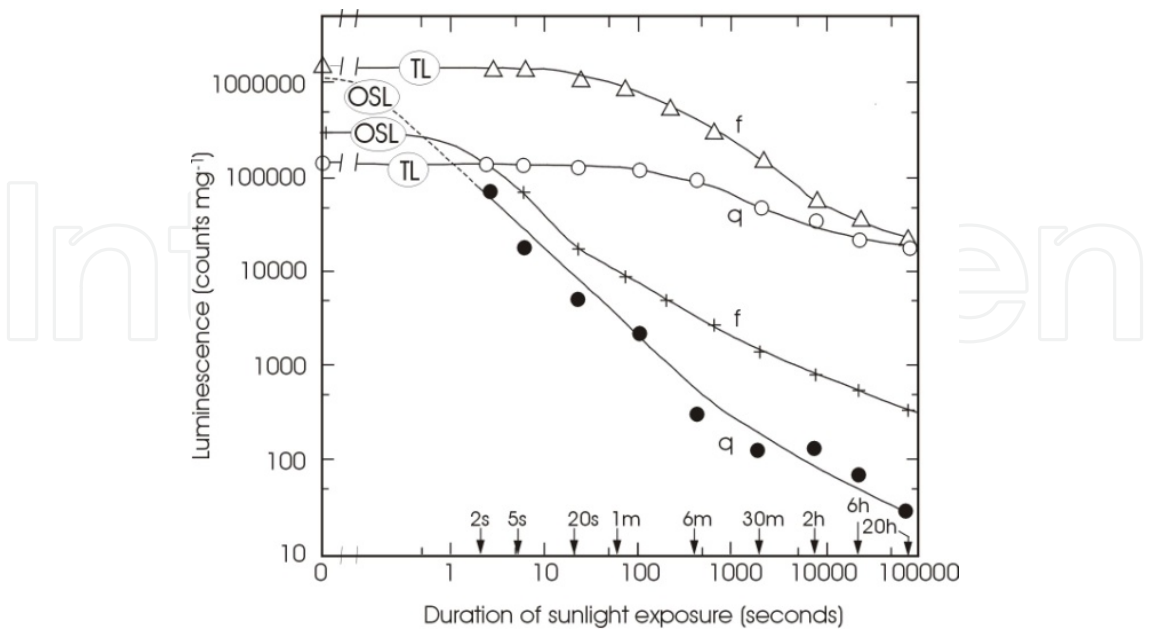

Figure 5. Comparison of bleaching rates of natural TL and OSL (green light) signals of quartz (q) and feldspar (f) conducted by Godfrey-Smith et al. [19] (redrawn from [29]). The slower bleaching curves are from the TL signals.

In a study examining the bleaching rates of quartz and feldspar (Fig. 5), it was shown [19] that after 20 hrs of exposure to sunlight, samples of both minerals had less than 0.1% of their original OSL signal remaining (Fig. 5). Conversely, similar samples had TL signals that were at least 10 times higher remaining after an equal time under solar radiation. This means that the optical signal is zeroed much more rapidly by the sun and the hard to bleach TL traps generally result in a much higher residual signal following solar bleaching compared to the OSL signal. Consequently, it is often difficult to date very young samples using TL [1, 29]), with OSL being preferred for dating sediments in general. However, dating of fired artifacts and baked sediments using TL remains an appropriate methodology since the zeroing mechanism in nature will be similar to the stimulation mechanism at measurement.

4. Luminescence properties of some common minerals

The discovery of luminescence in minerals is not a recent event. Aitken [2, 29, 32] has often cited the case of Robert Boyle who in 1663 reported holding a diamond close to his body and noting that it 'shined in the dark'. This is because many minerals are capable of luminescing in the dark when appropriately irradiated and stimulated. For dating purposes using luminescence methods, however, quartz and feldspar are the dosimeters that have received the most focus. Attempts have also been made to use zircon and calcite but these are not commonly used for dating because of a number of drawbacks. In this section, the luminescence properties of these minerals are briefly examined.

4.1. Quartz

Quartz is a widely used mineral in luminescence dating because of the advantages it offers compared to the alternatives. It is one of the most abundant minerals on the earth's surface, making it ubiquitous in most depositional environments, a feature arising from its high stability at the earth's surface and resistance to abrasion. It also has very stable luminescence properties. Because of its chemistry, quartz itself has no internal source of radiation that is a major element of its composition. As a result, the radiation quartz grains receive in nature usually originates from outside the grain. In some settings, however, quartz may contain some trace amounts of uranium [32].

4.1.1. Quartz TL properties

For TL analysis, natural quartz signals normally display two peaks above 300 °C [33]. One peak is at 325 °C and another at 375 °C and it is the latter that is usually used for dating. Laboratory irradiated quartz also shows a peak at 110 °C. In terms of emissions, when heated above 300 °C, quartz has a TL emission band in the range 460-480 nm (blue) and another in the range 610-630 nm (orange) [34]. Below 300 °C, quartz has an emission in the range 360-420 nm (near UV to violet) when irradiated with a laboratory dose. The 375 °C peak is thought to be from AlO_4 impurities in the quartz lattice serving as holes [35].The peak has a high thermal

stability but it tends to saturate at low doses such that it is of limited use beyond 50-100 ka unless very low dose rates are involved [36].

The 325 °C TL peak bleaches more rapidly than that at 375 °C [36] and its emission peaks at about 380 nm [37]. Hence, by using appropriate filters, the emission can be separated from that of the 375 °C peak. Studies suggest that, because of its relative stability, the 325 °C peak could in theory be used for dating up to 1 million years, which is much older than ages that can be measured using the 375 °C peak [36].

4.1.2. Quartz OSL properties

Irradiated quartz has been shown to emit OSL when stimulated by light from any part of the visible spectrum. For dating purposes using current methods, however, blue light is preferred in most applications because, as indicated earlier, OSL yield is proportional to the wavelength used for stimulation, with higher energies yielding higher OSL intensities [38]. The OSL signal from sedimentary quartz has been demonstrated to comprise at least three or four components and these are referred to as fast medium and slow components with respect to the rates at which they decay [39]. Others have reported up to seven components [40]. These components can only be separated when stimulation uses a constantly increasing stimulating power, referred to as linearly modulated OSL (LM-OSL) [41]. A stimulation source with a constant power (continuous wave), as is used in most regular dating, cannot resolve the components. However by using heat treatments (to eliminate unstable signals) or stimulating for controlled times (to exclude dominant slow components) the appropriate signal can be focused on when using continuous wave stimulation.

4.2. Feldspar

Feldspar has been used extensively in OSL dating. Like quartz, it is a mineral that is widely available at the earth's surface, though it weathers more rapidly. The chemistry of feldspars has important implications with respect to how they are used in luminescence dating. They are aluminosilicates that have potassium (K), calcium (Ca) or sodium (Na) as end members. The presence of potassium in some of the feldspars is critical in that ^{40}K isotopes that form part of the potassium constitute an internal source of radiation, in addition to any external radiation the grains may receive. Feldspars that do not have high potassium as part of their chemistry (for example, Ca and Na-feldspars), however, would not have this additional internal dose. Hence, for dating purposes, as will be discussed later, K-feldspars are normally separated from other feldspars prior to conducting analysis.

For dating applications, feldspar has a number of attractive characteristics. One is that, in terms of emissions, feldspars have a higher brightness compared to quartz which means that it gives strong signals, allowing smaller doses to be measured. Secondly, the internal dose in feldspars that have a high potassium content constitutes a reliable radiation source that is immune from environmental changes that would affect external sources (for example, interstitial water). As a result, dose rates can be determined more accurately. The third advantage of feldspar, which will be discussed below, is that it can be stimulated using infrared stimulation. A major

drawback for feldspar, however, which delayed its application in routine dating, is that it is afflicted by a phenomenon called anomalous fading [42]. In anomalous fading, the measured luminescence intensity decays with increasing time from the time of irradiation because some electrons have much shorter residence times in their traps than predicted by physical models [29, 43]. The ultimate result is that most feldspar grains will return equivalent doses slightly lower than they would if the dose were stable over time. To address this phenomenon in feldspar dating, correction methods for the fading have been devised [43, 44].

4.2.1. Feldspar TL properties

Many K-feldspars of sedimentary origin have been shown to display natural signals with TL peaks at 280 °C and 330 °C [36]. With regards to emissions, some studies [for example, 34] have reported emissions from K-rich feldspars in the 390-440 nm range (violet to blue) while plagioclase feldspar emissions have been reported in the 550-560 nm range (blue-green). However, results from other studies [47] suggest a more complex pattern.

4.2.2. Feldspar OSL properties

Luminescence from feldspars has been investigated using visible light stimulation. Earlier investigations used lasers (for example, 514.5 nm (green) from argon and 633 nm (red) from krypton) and observed the emissions at shorter wavelengths [1, 19]. For plagioclase feldspars, results showed that the spectra observed were similar to those from TL. Another study [48] that used a stimulation wavelength of 633 nm also showed that the emission was centred at 400 nm. OSL applications for dating using green light stimulation have been very limited and this has largely been because infrared stimulation (IRSL), as discussed below, was found to be a much better alternative. However, a study [49] that compared green light stimulation luminescence (GLSL) and IRSL data from feldspars from alluvial sediment showed results that suggested the signals had different thermal stabilities, with GLSL signals being more stable than IRSL signals at 10 °C. Stimulation of feldspars using a wide range of wavelengths in the range 380-1020 nm, apart from green and red, has also been demonstrated by [50].

4.2.3. Feldspar IRSL properties

Feldspars can also be stimulated using the near infrared part of the electromagnetic spectrum (around 880 nm). Since the discovery of this stimulation peak [20], most dating research that uses optical stimulation of feldspars for sediment dating has been focussed on IRSL. A major advantage of this, as stated earlier, is that it leaves the rest of the visible part of the spectrum open for use in emission detection. Other investigations using IRSL include studies on fine-grained sedimentary samples containing both plagioclase and K-feldspar that have also shown a major stimulation peak at 854 nm (1.45 eV) and another weaker one at 775 nm (1.6 eV) at room temperature [51]. Overall, these characteristics allow feldspars to be stimulated by light emitting diodes that have emission peaks at around 880±40 nm and these are widely available and cheap. Emission spectra of the K-feldspars stimulated using IRSL were reported by Huntley et al. [52] to show a dominant peak at 410 nm and another minor peak between 300 and 350 nm. Plagioclase feldspars, on the other hand, showed an emission peak at 570 nm.

Krbetschek et al., [53] reported additional natural emission peaks for K-feldspar at 560 nm as well as at 280 and 700 nm.

Feldspar samples irradiated with a laboratory dose have been shown to display an additional emission peak at 290 nm. For feldspar samples with a natural signal however, this 290 nm peak is absent. In dating studies, the 290 nm peak can be eliminated by preheating [36].

4.3. Calcite

The mineral calcite has been demonstrated to have a TL signal with an emission at 570 nm [36] and attempts have been made to use the mineral for dating. However, calcite which often occurs in carbonate cave deposits has a limited environmental occurrence, which constrains its applicability. The luminescence from calcite is also complicated by the tendency of calcite to preferentially concentrate uranium. Thus, evaluation of the dose rate has to account for the disequilibrium of the decay chain of uranium. Notably, calcite dating using the uranium disequilibrium can be used to establish better chronologies than would be attainable using luminescence methods [36]. Hence, overall, the incentive to use calcite in luminescence studies has been low. Published attempts to use OSL emissions from calcite for dating include studies by Ugumori and Ikeya [54]. Nonetheless, as with the TL efforts, these have not translated into widespread applications.

4.4. Zircon

Zircon also has luminescence properties. Its properties as a dosimeter are particularly interesting because zircons naturally have a high concentration of uranium such that the internal dose that they receive is usually far greater than any radiation originating from the grain's exterior. As a result, the dose rate is very constant because it is not susceptible to variations that may be induced by changes in interstitial water content or burial depth [36]. A major methodological drawback, however, is that the uranium content is variable between grains and, as a result, measurements have to be made on individual grains. Zircon studies using TL include investigations by Huntley at al. [46] and Templer and Smith [55]. OSL analyses on zircons include studies by Smith [56].

In addition to variations in the uranium content between individual grains, zircon grains also have inhomogeneities in their crystal structures that develop during formation. Hence, for dating purposes, the luminescence from the internal dose is not easily comparable to the signal from the artificial dose administered in the laboratory as would be done with quartz or feldspar. To circumvent this problem, zircon dating often employs the autoregeneration method whereby the natural signal of the zircon is measured after which the grains are stored for several months [55, 56]. At the end of this storage period, the grains are measured again to determine the signal that has accrued from the internal dose since the initial measurement. This storage signal is then used to calibrate the natural signal from antiquity to determine an age [2].

5. Paleodose and dose rate determination

As outlined earlier, in order to calculate an age, the basic luminescence age equation divides the dose that has accumulated since the beginning of the event being dated (paleodose) by the rate at which the energy was accumulated (dose rate) (Equation 1). Hence, the two basic parameters that have to be determined are the paleodose and the dose rate. In this section methods used to determine these variables are discussed.

5.1. Paleodose determination

Paleodose determination aims to ascertain the amount of energy that has accumulated in a dosimeter since the event being dated occurred. In luminescence dating this is generally the period that coincides with the time when the mineral grains were emptied of any previously accumulated energy (or zeroed). The dose is generally defined as the energy absorbed per kilogram of material and the unit used to measure it is the gray (Gy) where 1 Gy=1 joule per kg. For fired (or heated) materials, the period corresponding to zeroing would be the time when the samples were last heated to the appropriate temperature whereas, for unheated sediments, it would commonly be the time when they were last adequately exposed to the bleaching effects of the sun. The luminescence signal obtained from a sample from the field is referred to as the natural signal. In principle, in order to determine the natural signal in a given sample in Gy, artificial irradiation that is well calibrated is used to induce luminescence in the sample in a laboratory setting after which the natural signal is compared to the signals from the artificial irradiation. This allows the magnitude of the laboratory dose that induces a signal equivalent to that produced by the natural dose to be ascertained. That laboratory dose is referred to as the equivalent dose (D_e). As depicted in Fig. 6, there are two main methods that have been developed for determining D_e and these are the additive dose and the regenerative dose (or regeneration) methods [3, 29]. Other methods that have been used in the past include the partial bleach method [for example, 17]. However, these are no longer widely used.

5.1.1. Additive dose method

As initially developed, to determine the equivalent dose using the additive dose method, samples from the field are typically separated into multiple aliquots. One set of aliquots would have the natural signal measured after which the other aliquots are irradiated with well calibrated incremental doses and then measured, with multiple aliquots being used for each dose level. The acquired signals are then plotted to give a dose-response curve that shows the luminescence signal against the laboratory irradiation (Fig. 6a). This is referred to as a growth curve and it is essentially a simulation of the evolution of the total dose had the sample experienced similar dose levels in its natural setting over time. Since the method employs multiple aliquots, signal normalisation is performed to correct for inter-aliquot variations by giving the aliquots a small test dose afterwards and then measuring the response. Such variations arise from differences in mass and grain sensitivity. To determine D_e using the additive dose method, the curve is extrapolated backwards to zero signal intensity and the D_e will be where the curve intercepts the horizontal (dose) axis (Fig. 6a). In TL dating, the

residual signal remaining after solar bleaching, would have to be taken into account as well. It is important to note that, for both feldspar and quartz, growth curves usually show a linear relationship between the luminescence signal and the dose in the early part of the curve. If high enough doses are administered, however, the signal tends to level off, indicating saturation or an exhaustion of the luminescence traps.
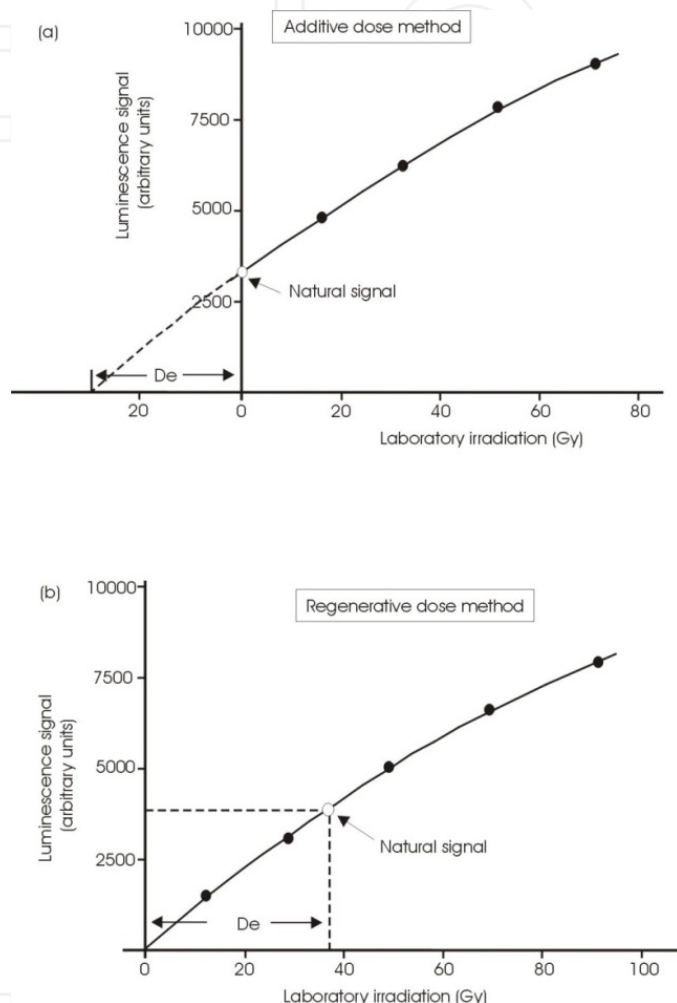

Figure 6. Methods used to determine the equivalent dose (D_e). In the additive dose method (a), incremental doses are given to unbleached samples and measured. In the regenerative dose method (b), on the other hand, the samples are zeroed first before incremental doses are administered and measured (modified after [3]).

5.1.2. Regenerative dose method

The regeneration method differs from the additive dose method in that the samples are zeroed first before any laboratory dose is applied. For TL dating, previously fired artifacts, for example archeological materials or baked sediments, are zeroed by heating. For OSL samples, on the other hand, zeroing is achieved by exposure to sunlight. The zeroed aliquots are then given incremental doses as with the additive dose method, preferably with the doses being chosen to lie above and below the natural signal. Normalisation can also be conducted to correct for

inter-aliquot signal variations. Signals from the doses are then plotted to give a regenerative growth curve. The equivalent dose would be obtained by interpolating the natural signal or the unknown signal into the curve (Fig. 6b).

5.1.3. Single aliquot methods versus multiple aliquot methods

As outlined above, when initially developed for TL dating, both the additive dose and regenerative dose methods involved the use of multiple aliquots. With the introduction of OSL dating, though the possibility of using single aliquots was contemplated early [1], the practice of using multiple aliquots was adopted too [36]. An inherent assumption when using multiple aliquots is that all the aliquots of a given sample respond similarly to the dose received. However, this is not what is observed; inter-aliqout variations arise from a number of sources and this necessitates the implementation of normalisation to try and address the differences. Quartz in particular appears susceptible to sensitivity changes. The different grains within any particular aliquot would have experienced dissimilar histories within the natural environment and these can include differences in erosion and deposition cycles, episodes of heating from wildfires or other extreme conditions [3, 57]. The heating administered in the laboratory as part of the analysis (see further below) also results in sensitivity modifications that are dissimilar between the grains, and ultimately between the aliquots. Variations in sample mass between aliquots may also be a reason for differences in behavior between aliquots. The net result of these disparities is that they give rise to differences in sensitivity that contribute to uncertainties in the calculated ages, even in cases where normalisation is used. Hence, to help address these aspects, there was a desire to develop a method that only utilised a single aliquot. There are a number of advantages associated with the use of single aliquots and these include [36]:

i.   when using a single aliquot, to obtain D_e, the natural signal obtained will be compared to the dose response curve of the same aliquot. Hence, inter-aliquot variations are eliminated resulting in much higher precision for D_e.

ii.   only a very small amount of sample material is required. This is particularly important for archeological samples that may be of limited size.

iii. normalisation to correct for inter-aliquot variations in numbers of grains on different sample discs or variations in sensitivity is not necessary when using a single aliquot.

iv.   the measurement protocol employed with single aliquots which entails preheating, bleaching, and irradiation can all be conducted within most modern readers which are automated and it increases precision in addition to reducing analysis times.

The following section examines procedures used in single aliquot methods.

5.1.4. Single-Aliquot Regenerative-dose (SAR) protocol

The introduction of single aliquot methods began with investigations that employed the additive dose approach. Efforts to use single aliquots for dating quartz had observed that there were sensitivity changes associated with repeated preheating of the aliquot that was required after every successive laboratory dose prior to conducting the luminescence measurements.

Preheating is necessary because it ensures that the distribution of trapped electrons after administering the laboratory dose is similar to that resulting from the natural dose [58]. To correct for the sensitivity changes using the single aliquot additive dose method on feldspars, Duller [21] monitored an additional aliquot for sensitivity changes. Galloway [22] improved on Duller's [21] approach by correcting the luminescence signals using a least squares fitting approach using measurements made on the same feldspar aliquot subsequent to the additive dose irradiation and measurements. This modification by Galloway [22] essentially transformed the method into a bona-fide single aliquot protocol. Alternatively, using the additive dose method on quartz, Murray et al. [27] devised a correction procedure that included additional preheat and stimulation cycles without any additional dose being given, the results of which were used to formulate a decay constant. The constant was then used to correct the data from the additive dose measurements.

Attempts to use single aliquots with the regeneration method on feldspar [21] and on quartz [58] had initially concluded that it would not be possible because of sensitivity changes [58]. However, to address those sensitivity changes, Mejdahl and Bøtter-Jensen [60] proposed the single-aliquot/ regeneration–added dose protocol (SARA) to date previously heated materials and Murray [61] later used the same method on unheated sediments. However, with SARA, at least two aliquots are required [58]. Subsequently, a truly single aliquot regenerative dose (SAR) protocol was introduced by Murray and Roberts [58] using sedimentary quartz from Australia and, with that SAR method, corrections for the sensitivity changes were made by monitoring the 110° C TL signal measured immediately after administering a regeneration dose. A major methodological breakthrough was made when a streamlined version of the method proposed by Murray and Roberts [58] was put forward by Murray and Wintle [26] whereby sensitivity changes were monitored using a test dose whose signal was also measured. As initially proposed by Murray and Wintle [26], this SAR protocol essentially entailed the following sequence:

Step	Treatment[a]	Observed
1	Irradiate sample with dose, D_i	-
2	Preheat sample (160-300°) for 10s	-
3	Stimulate sample for 100s at 125 °C	L_i
4	Irradiate sample with test dose, D_t	-
5	Heat to 160°C	-
6	Stimulate for 100 s at 125°C	T_i
7	[b]Return to 1 and repeat sequence	-

D_i is the regeneration dose which gives signal L_i whereas D_t is the test dose which gives signal T_i. The observed signals, L_i and T_i are then used to plot a curve of L_i/T_i vs. the regeneration dose, D.

[a]For the natural sample, 1=0 and D_0=0 Gy.

[b]Later modifications stimulate the sample for 40s (instead of 100s) and an additional step to optically stimulate the sample for 40s at a temperature above the preheat temperature is conducted after step 6 to reduce recuperation [see 26, 62].

Table 1. Steps in the SAR protocol as originally proposed by Murray and Wintle [26].

Since its original introduction, the SAR protocol has undergone some minor modifications (Table 1) [26, 27] and, over the last 10-15 years, the protocol has emerged as the preferred method for routine dating of both sedimentary and fired materials using both quartz and feldspar. Further methodological details can be found in [26, 27, 62].

5.1.5. Single grain analysis

Measurement protocols used for analyzing single aliquots can also be adapted for determining paleodoses using individual mineral grains. Special equipment for loading and analyzing single grains have been developed that allow multiple sand-sized grains (up to 250 μm) to be mounted individually in a regular array that permits automated measurement [25]. In this format, thousands of grains can be analysed in a relatively short period of time. Analyzing single grains makes it possible to recognize differences in behaviour between grains from a given sample. For instance, it enables the identification of grains that have been bleached to different levels prior to burial because such grains will yield different paleodoses. For that reason, single grain analysis is commonly used to identify partial bleaching in sediments, especially in fluvial deposits [63].

5.1.6. Presentation of luminescence paleodose data

Once determined, paleodose results can be presented as growth curves as in Fig. 6, with the horizontal axis showing the dose given and the vertical axis showing the luminescence signal (or nor normalised signal). However, for SAR procedures where multiple determinations can be made on the same sample that yield a range of equivalent dose values, individual growth curves do not convey all pertinent statistical information. Alternative means that can be used to provide some statistical information include frequency histograms. However, histograms do not provide information on precision [64]. To address that aspect, paleodose data in luminescence dating are now commonly presented as radial plots that show both the number of D_e determinations made as well as the relative precision associated with each determination [65].

In the example of a radial plot given in Fig. 7, each dot represents an equivalent dose that was determined for a single grain using the SAR approach for a total of 204 grains. Had multiple grain aliquots been used, each data point would denote an aliquot. Any straight line that passes radially through the origin represents a line of constant dose. The horizontal axis at the bottom shows the relative error associated with each paleodose calculation, with the precision increasing from left to right. The shaded area in the plot [Fig. 7] denotes the 2 sigma error band centred at the equivalent dose of 25 Gy, representing the weighted-mean of all 204 data points. Thus, the band represents a 95% confidence level on all aliquots that were analysed. The number of data points that lie outside the shaded area are reflected by the overdispersion of the data and can be calculated [65]. The equivalent dose scale on the right is a logarithmic scale. Overall, radial plots enable investigators to visualize the dose distributions, allowing appropriate data to be targeted for further analysis. For instance, they permit investigators to differentiate between variations in equivalent dose that arise from the bleaching history and local dose rates from those that are caused by intrinsic differ-
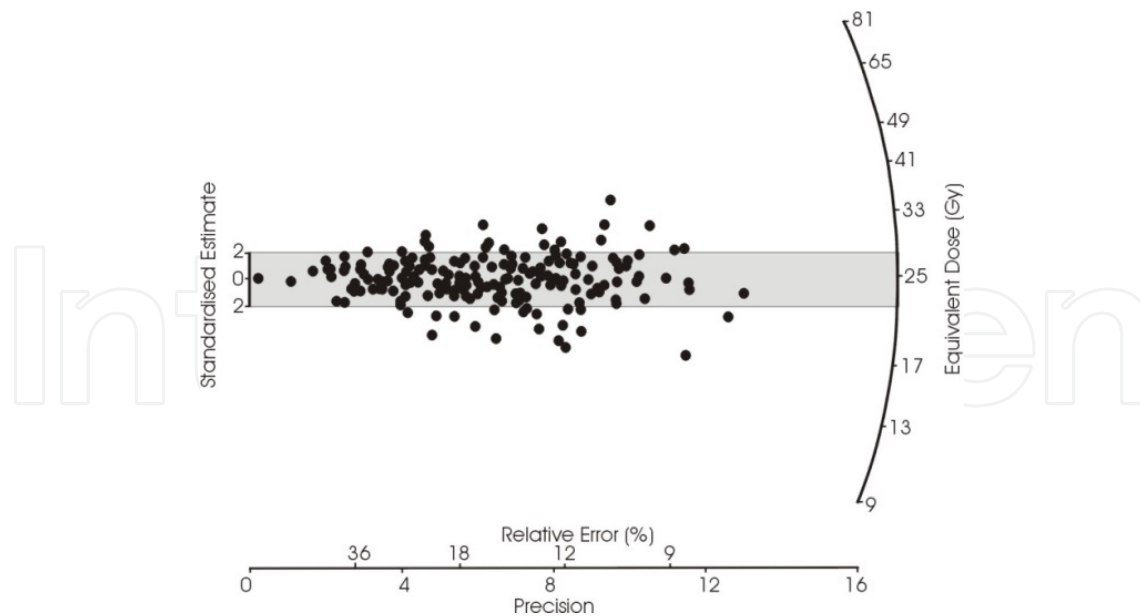
Figure 7. Radial plot of equivalent dose (D_e) estimates for 204 single grains from Sample M3T 61.2 m from Lake Mungo, Australia, obtained using the SAR protocol (redrawn from [66]). Each data point represents a single grain. Precision, shown on the x-axis, is simply the reciprocal of the standard error. Hence, as relative error decreases as one moves to the right, the precision increases.

ences in luminescence sensitivity of the measured aliquots [64]. Accordingly, radial plots have often been used to identify poorly bleached samples, especially when used to analyze individual grain paleodoses. Once the paleodose is determined, statistical models used to calculate the age include the central age model, which calculates the weighted mean equivalent dose from a set of data points taking into account the overdispersion above that associated with measurement errors. Similarly, the minimum age model gives the equivalent dose associated with the population of aliquots (or grains for single grain dating) with the lowest dose. Details on these models can be found in [65].

5.2. Dose rate determination

Apart from the paleodose, an additional element that has to be determined before a luminescence age can be calculated is the dose rate. As outlined earlier, the main isotopes responsible for the accumulation of luminescence energy in natural setting are the isotopes of the uranium and thorium decay chains, potassium and rubidium as well as cosmic radiation. Hence, the total contribution of these effects have to be evaluated. There are several methods that can be used to determine the total dose rate. One is the concentration approach and with this method, concentrations of thorium, uranium, potassium and rubidium in representative samples are measured using an appropriate technique such as neutron activation, atomic absorption spectroscopy (AAS), X-ray fluorescence (XRF), flame photometric detection (FPD) or inductively coupled plasma mass spectrometry (ICP-MS). However, measurement of uranium and thorium using this approach can be inaccurate if there is disequilibrium [29].

High-resolution gamma ray spectrometry and alpha spectrometry are alternative methods that can be used to measure activities of individual radionuclides, including those from the thorium and uranium decay chains. Hence, they can be used to measure dose rates whether there is disequilibrium or not. However, extended measurement times could be required and the equipment can be costly [29].

Another approach that has been employed to minimise effects of disequilibrium of the thorium and uranium chains is to measure the contribution of uranium and thorium using thick source alpha counting (TSAC) and then use AAS, XRF, FPD or ICP-MS to measure potassium. An alternative procedure is to measure the alpha contribution only using TSAC and determine the beta contribution using a beta particle counter. Whenever possible, the gamma dose rate should be measured on site, especially in settings where there is uncertainty about the uniformity of the dose within a 30 cm radius of the sample. The recent development of powerful portable gamma–ray spectrometers has made such field measurements relatively practical [67]. Other field measurement options include the use of very sensitive synthetic dosimeters such as α-Al$_2$O$_3$:C that only need to be buried in the field for a few weeks at the most, as opposed to earlier dosimeters that required burial for up to a year [67].

As described earlier, cosmic-rays also contribute to the dose received by the mineral grains. This contribution is usually minor but in settings where the contributions from the radionuclides are low, cosmic contribution can be significant. Formulae for evaluating cosmic ray contribution have been provided by Prescott and Hutton [30].

The effect of moisture content in the natural setting of the material being dated is to absorb part of the dose that should normally reach the grain. Consequently, when calculating dose rates, the levels of moisture content have to be noted and factored into the determination. In essence, dry sediment will experience a higher dose rate than moist sediment.

5.3. Lower and upper limits for luminescence ages

Improvements in luminescence dating instrumentation and dating protocols have reached a stage where current OSL methods can be used to date samples deposited as recently as the last few decades [for example, 68]. The single grain dating method in particular can yield dates with very high precision. Prerequisites for dating such young samples include appropriate bleaching to remove all previously acquired luminescence energy prior to burial as well as the availability of grains that have high luminescence sensitivity [28]. To optimize the measurements and increase precision, thermal charge transfer is minimized in order to increase the signal size. Such advances mean that luminescence methods can now produce ages from the last 300 years that are more reliable than those attainable using radiocarbon methods. Calibrated radiocarbon ages from the same period have comparatively larger uncertainties due to fluctuations in ^{14}C production [69].

With regards to the upper age limit attainable using luminescence methods, empirical studies have demonstrated that the storage of luminescence energy through the trapping of electrons is not a process that can continue indefinitely within any given material because, eventually, the traps do get exhausted [29, 31]. For that reason, luminescence growth curves are often
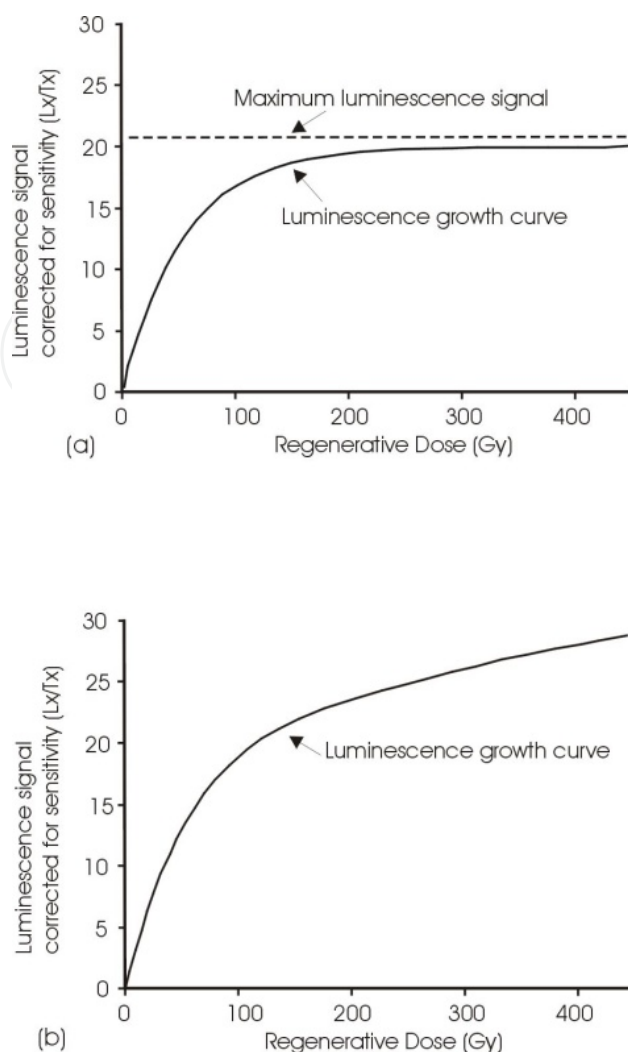
Figure 8. Illustration of quartz growth curves constructed using the SAR protocol (redrawn from [3]). In (a) the curve is constructed using a saturating exponential of the form $I(D) = I_0 \left(I - exp^{-D/D_0}\right)$ where I(D) denotes the OSL signal commensurate with the dose D and I_0 is the maximum OSL intensity that can be obtained. The variable D_0 determines the shape of the curve and in curve (a), D_0 is 55 Gy. Because curve (a) appeared to underestimate some older ages, in (b) the saturating function from (a) is combined with a linear function [3].

represented by saturating exponential functions and the form of the curve determines the maximum luminescence signal that can be stored in the mineral grain beyond which no dose can accumulate efficiently. For quartz for instance, Fig. 8a shows the fast component expressed as a single saturating exponential and, beyond a certain dose, the curve flattens. Dating of quartz samples close to saturation (>100 Gy) has been demonstrated in some studies to yield OSL ages that underestimate the true age by up to 10% [for example, 70]. In reality, the absolute age limit will be determined by the dose rate, with low dose rates having higher age limits. The curve in Fig. 8a also shows that the growth curve is relatively linear for low doses, and ages from the linear part of the growth curve have been shown in numerous studies to be comparable to those obtained using other dating methods, for example, radiocarbon. Generally, however, these results indicate that the storage of luminescence dose in mineral grains

has an upper limit beyond which the traps become saturated. This dose level places a limit beyond which the method cannot be applied. For quartz this dose is about 100-150 Gy. The solution adopted in some studies when working with older ages [4, 5] is to model the luminescence growth using a combination of the saturating function and a linear function to give a curve as in Fig. 8b [28]. Ages in excess of 200-400 ka have been reported from quartz using this method [for example, 5]. Feldspar ages in excess of 100 ka obtained using IRSL signals have also been reported in a number of studies [71]. However, at such dose levels (>100-200 Gy) the growth curve is no longer linear such that correction for anomalous fading using standard procedures [43] becomes problematic.

Overall, however, the indication is that for both feldspars and quartz, there is an upper age limit beyond which accurate OSL ages cannot be obtained. The exact limit will ultimately depend on the dose rate.

6. Sample collection and preparation: some practical aspects

Having looked at the principles of luminescence dating and the methodological aspects in the preceding sections, a topic that needs to be discussed are the types of materials that can be dated using luminescence methods. Prior to exploring that topic, however, it is imperative to examine aspects of the dating method that have a bearing on the types of materials one can date using luminescence techniques. Accordingly, this section examines the importance of grain size after which sample collection and preparation methods are discussed.

6.1. Importance of grain size

For technical reasons, luminescence dating is usually conducted on mineral grains in two broad grain size categories: coarse grains and fine grains. In the coarse grain method, grains in the fine sand category are separated and analyzed. Such grains would normally receive dose from alpha, beta, and gamma radiation. However, because alpha particles can only penetrate the outer 25 μm of the grain, the coarse grains are typically etched using hydrofluoric acid (HF) to remove the outer ring of the grain that experienced alpha radiation. As a result, methods that date coarse grains are often referred to as inclusion dating methods [33]. For quartz inclusion dating of pottery, particles in the size range 90-125 μm are usually extracted [2]. For sediment dating, on the other hand, coarse grains representing the modal grain size are normally extracted. For eolian dunes for instance, grains in the size range 150-180 μm are usually preferred [3]. In feldspar inclusion dating, similar procedures are used to extract grains in the fine to medium sand size range and etching can also be used to remove alpha particle effects. As a result, when dating coarse grains, the annual dose is calculated by evaluating contributions from beta, gamma and cosmic radiation and the age equation is modified to:

$$Luminescence\ Age = \frac{Paleodose}{0.90D_\beta\ +\ D_\gamma\ +\ D_C} \qquad (2)$$

where D_β, D_γ and D_c refer to the beta, gamma and cosmic ray dose contributions respectively [29, 31]. When the dose rates are expressed as annual rates, the age will be given in years. The beta contribution in Equation 2 is factored by 0.9 to account for the attenuation due to grain size as well as the etching that removes some parts of the grain that received the beta dose [29, 31]. Exceptions to Equation 2 are only those cases where the quartz itself has some uranium and thorium within it. Also for feldspar dating, where K-feldspars are usually isolated from Na-feldspars and dated separately, an additional parameter would also have to be included in the denominator in Equation 2 to account for the internal dose from potassium.

In fine-grain dating procedures, dated materials are often not separated into mineral specific concentrations. Rather, polymineral grains with diameter in the range 4-11μm (fine silt) are extracted and analysed. Because of their size, alpha particles can penetrate these particles completely and alpha contribution has to be taken into account when calculating the dose rate. As a result, general age equation is modified to:

$$Luminescence\ Age = \frac{Paleodose}{kD_\alpha\ +\ D_\beta + D_\gamma\ +\ D_C} \tag{3}$$

where D_α, D_β, D_γ and D_c refer to the alpha, beta, gamma and cosmic ray dose contributions respectively. Alternatively, in some studies, fine grained quartz is extracted from the polymineral mixture of fine grains using procedures outlined below and analyzed separately. There are some studies that have used intermediate size grains in the range 35-63 μm, which is coarse silt [for example, 72]. For such studies, alpha particle contribution also has to be taken into account when calculating the dose rate.

6.2. Sample collection and laboratory procedures

6.2.1. Sample collection

When collecting samples for dating using luminescence methods, a primary requirement that has to be fulfilled is that the mineral grains to be analyzed should not be exposed to light from the time they are initially buried up until the point they are exposed to the stimulating source during measurement. This restriction necessitates the adoption of special precautions during sample collection and a number of procedures have been devised over the years. For archaeological artifacts this may entail extracting material from the interior of the artifact using a drill under safe light conditions. For sediments, however, measures taken include sampling at night [29, 64]. Not only is this an inconvenient method because of the need to work in the dark, but there is a greater risk of accidentally exposing the sample to light during the collection [73]. For sediments that are adequately firm, an alternative approach is to cut out a block of sample from the depositional unit being investigated and transfer it to the laboratory where a portion for analysis is extracted from the sample's interior [29, 64]. A sampling approach that has become a method of choice because of its ease and relative guarantee for retrieval of an unadulterated sample is to insert an opaque pipe made of metal, PVC or ABS plastic into a freshly prepared profile face [29]. Once retrieved, the pipe is immediately capped on both ends with an opaque and

preferably airtight seal. At the laboratory, sediment at the ends of the pipe is removed and the sample for luminescence measurements is taken from the central portion of the pipe. In places where depositional units are not directly accessible, drilling has also been used to reach targeted units and methods that can be used for such sampling are reviewed in [73].

6.2.2. Sample preparation prior to measurement

Once the collected sample has reached the laboratory, it has to be prepared for analysis and a number of procedures have been established depending on the material targeted for analysis. As outlined in Section 6.1, samples for luminescence analysis are either measured as fine or coarse grains. It was also indicated that for coarse grains, analysis is usually made on pure mineral separates (for example quartz, or K-feldspars) whereas for fine grains, either poly-mineral fractions or fine grained quartz extracts can be used. When dating fine grained sediments such as loess, for instance it would be preferable to use the fine grain procedure whereas for eolian dune sands, the coarse grain approach would be more appropriate. In sediments that have equal components of fine grains and coarse grains, dating both fractions would provide a good mechanism for comparison as a cross-checking method.

For coarse grained materials, quartz or feldspar are typically separated using a heavy liquid such as sodium polytungstate solution. Fig. 9 is a flowchart of a separating procedure for quartz and feldspar using heavy liquids with successively lighter or heavier specific densities. Prior to separating minerals using the heavy liquid, carbonates and organic materials that often occur in sediments, and are usually introduced during the postdepositional phase of the deposit, are removed using hydrochloric acid and hydrogen peroxide respectively. Additional information on separation procedures of coarse grains can be obtained from [74, 75].

For fine grains, carbonates and organic materials are also removed using dilute hydrochloric acid and hydrogen peroxide respectively. Dilute sodium oxalate solution is then added to prevent flocculation of the particles after which appropriate grain sizes are separated using a sedimentation column. Sedimentation columns employ Stoke's law which states that the velocity of a particle's sedimentation in a fluid also depends on the size of the particle. Hence, by extracting sediment from the column after a predetermined time following agitation permits a desired grain size to be isolated. As indicated earlier, while fine grains can be analysed as polymineralic fractions, some studies extract pure quartz from the fine grains by digesting the feldspars using fluorosilicic acid [for example, 76]. Detailed information on separation procedures of fine grains for luminescence dating have been provided by [77].

Once separated, coarse grains (quartz or feldspar) or the fine grains (mixed polymineralic or pure quartz) are mounted on appropriate sample discs prior to analysis. Typically, the measurement discs are made of stainless steel or aluminum and measure around 10 mm in diameter and 0.5 mm thick [29]. To mount coarse grains, a monolayer of the sample is deposited on the disc in dry form. Silicone oil can be used to help the sample grains adhere to the disc. For fine grain particles, on the other hand, once the desired grain sizes have been isolated, these are mounted on the discs in a solution of acetone, ethanol or water which is then allowed to evaporate, leaving the sediment deposited on the disc. In either case, the disc will now be ready for analysis.
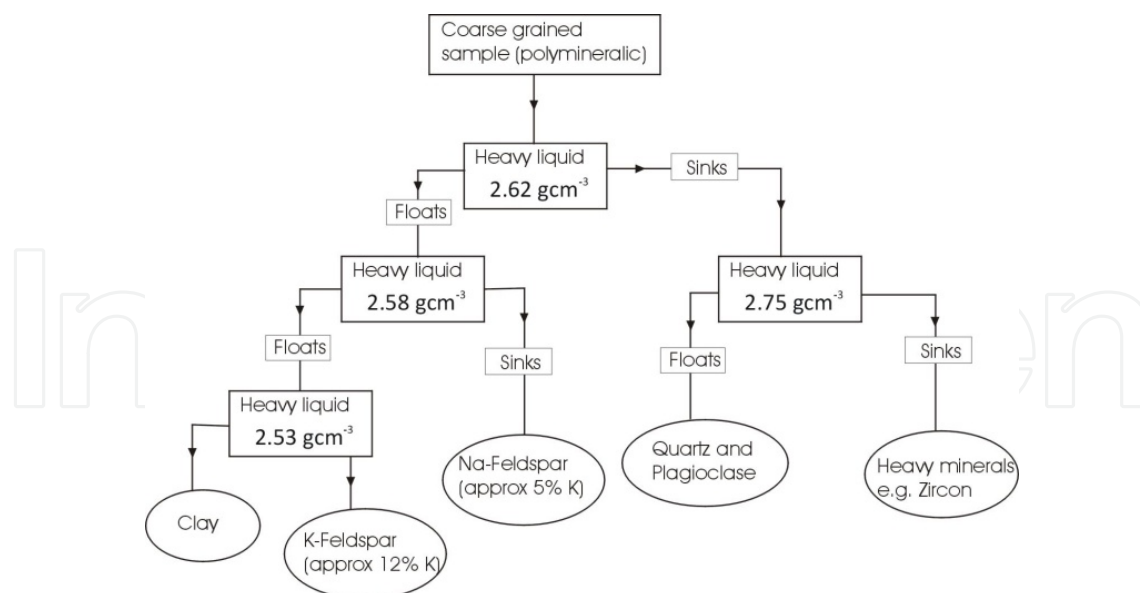
Figure 9. Separation procedure for coarse grains using a heavy liquid. Starting off with a liquid of specific gravity (s.g.) 2.62 gcm^{-3}, the sample is separated by centrifuge into a fraction that is lighter (floats) and one that is heavier (sinks). The fraction that sinks is passed through a liquid of s.g. 2.75 gcm^{-3} and the fraction that floats will comprise mostly quartz or plagioclase while the sinking fraction will comprise heavy minerals, for example, zircon. The fraction that floats in the liquid of density 2.62 gcm^{-3} is passed through another liquid of s.g. 2.58 gcm^{-3}. The fraction that sinks will be Na-feldspar whereas the lighter fraction is further separated using a liquid of s.g. 2.53 gcm^{-3} into K-feldspar and clay.

7. What materials can be dated using luminescence methods?

For all practical purposes, an important aspect about luminescence dating is knowing what type of materials or sediments can be dated using luminescence methods. Discussions in the preceding sections have touched on this topic albeit indirectly. As detailed earlier, luminescence methods date materials by measuring energy that has accumulated in materials called dosimeters. Hence, one prerequisite for dating using the method is that the material to be dated must contain a dosimeter. The second prerequisite is that there has to be an event that reset the energy previously stored by the dosimeter to provide a starting point for counting the time. In essence, all energy stored in the dosimeter will be assumed to have accumulated since that point. A third requirement for material to be datable using luminescence methods is that the electron traps in the dosimeter should not be exhausted at the time of dating because once energy storage has reached a point of saturation, the relationship between time and dose rate breaks down. Hence, the sample for dating has to be younger than the upper age limit that can be attained for that particular dosimeter and, as indicated earlier, the exact limit ultimately depends on the dose rate too. With these considerations in mind, materials datable with luminescence techniques broadly fall into two main categories: materials that have been heated and clastic sediments of sand and silt size that have been reset (zeroed) through exposure to solar radiation.

7.1. Heated materials

As discussed in an earlier section, luminescence dating initially began as an archaeological technique that was used for dating materials that had been heated to temperatures adequately high (for example, > 500 °C) to expel all electrons from their traps. Hence, the heating process provides a starting point that can serve as year zero when dating such artifacts. Fired artifacts such as ancient pottery, tiles, bricks or terracotta figures are all examples of archeological materials that can be dated using luminescence methods, especially TL, because these materials usually contain dosimeters such as quartz and feldspar. By separating the quartz and dating quartz inclusions, or by using the fine grained components to date polymineralic fractions or quartz separates, ages can be obtained. Humans are generally believed to have discovered the art of making pottery during the Neolithic period, which dates back to about 30 000 yrs ago in some places [for example, 78], but the art only became widespread about 10 000 years ago [32]. Hence, unless dose rates are very high, most ancient pottery artifacts are not yet saturated and should be datable using luminescence methods.

Apart from pottery, other heated materials of archaeological significance that can be dated using luminescence methods include burnt flint and burnt stones that were associated with human settlements and may have been heated to high temperatures. Flint is a sedimentary form of quartz and hence possesses dosimetric properties [32]. Paleolithic humans used flint extensively as a tool for scraping and for cutting as well as for projectile points. Chips or debitage left over from the manufacture of such implements can be found associated with ancient settlements. If any of these tools or chips were at some stage heated, either deliberately or accidentally during the occupation of the site by humans, dating the objects using luminescence methods will provide a chronology commensurate with the timing of human habitation. Thus, heated flint can be a useful chronometer especially for timescales beyond those commonly covered by other methods such as radiocarbon [32]. Other heated geological materials include stones that were used as 'pot-boilers' by some societies prior to the discovery of pottery. In some settings stones were also used for constructing fireplaces. In both instances, where these previously heated stones contain appropriate minerals they can be dated using luminescence methods [for example, 79].

A different class of heated materials that can be dated using luminescence methods are geological materials that have been heated to appropriate temperatures to have zeroed them during the last approximately 100,000 years. Such materials include contact-baked sediment that is heated to high temperatures following a volcanic eruption. In such cases, the soil can be collected and grains extracted for dating using either coarse grain or fine grain methods [80]. If the sediment contains larger clasts such as gravel, constituent quartz or feldspar grains can be extracted from the pebbles for analysis. The age obtained would be congruent with the timing of the volcanic eruption [32].

Also associated with volcanic eruptions are the products of the eruption itself such as lava and ash. The heat associated with the volcanic eruption is sufficient to zero these products of any previously acquired dose, if at all, since the lava would mostly be in liquid form rather than crystalline state. Fine grained glass (4-11μm) extracted from volcanic ash has been used in

some studies to date the eruption [81]. Other studies have attempted to use minerals from the lava instead of volcanic ash [for example, 82]

Finally, also related to the effects of heating to reset the luminescence signal are materials that have been zeroed by heat emanating from the impact of a meteorite. The thermal shock associated with such impacts can reach temperatures high enough to zero constituent grains of the affected geological material [for example, 83].

7.2. Dating of sediments reset by sunlight

A class of materials that has fostered the rapid development of luminescence dating methods over the last three decades are clastic sediments that have been zeroed by solar bleaching. Since the confirmation by Huntley et al. [1] that energy from sunlight was capable of adequately erasing previously accumulated energy from dosimeters, there have been many applications of the method to obtain chronologies from sedimentary materials, initially using TL and later using OSL. As outlined above, the prerequisites for dating using luminescence methods which include a presence of a dosimeter in the material, the occurrence of a bleaching episode that erases any previously accumulated energy, and the absence of saturation in the dosimeters (sediment grains) need to be satisfied if a material is to be dated. A number of sedimentary materials satisfy these criteria and have been dated using luminescence methods, with some presently constituting formidable chronological archives of environmental change. These include sediments deposited by wind (eolian sediments), water-laid sediment, glacial deposits, and earthquake related sediments. These sediment classes are discussed below under the respective headings. In all instances, reference to ages obtained from sediments bleached by solar resetting denotes burial ages or time that has elapsed since the last time the sediments were exposed to light from the sun.

7.2.1. Eolian deposits

Wind deposited (eolian) sediments are the sediment of choice for dating using luminescence methods. This is because the subaerial transport that the sediment experiences during transportation is expected to provide adequate time to bleach the sediment grains of any previously accumulated energy [29]. While this may not be true in some cases, results generally show that in most settings, that assumption is valid [84]. As a result, this class of sediments has provided the majority of luminescence ages reported to date. Eolian sediments generally fall into one of two main classes. One class comprises sand grains in the fine to medium size range (63-250 μm) that are generally transported by wind through a series of low jumps along the surface of a sedimentary bed in a process referred to as saltation [85]. These grains are usually deposited as dunes. The other sediment category is silt size grains (2-63 μm) that are transported by wind in suspension. Fine silt grains can remain airborne for extended periods of time [85]. For both sand size and silt size grains, the particle transport at the surface is often adequate to zero the grains.

Quartz and feldspar grains extracted from eolian sediments have been dated in numerous studies which have compared the chronologies obtained to those from radiocarbon ages from associated sediments and have provided results that are congruent, validating luminescence dating of eolian sediments as reliable chronometers [for example, 86]. In many cases, because eolian deposits are proxy indicators of dry conditions from the past, luminescence ages from the eolian deposits have been used to provide a temporal framework for environmental changes from the past. Fossil dunes from inland deserts of Australia [for example 87, 88], southern Africa [for example 88, 89], Mongolia [for example, 90], United States [for example, 91, 92], Canadian Prairies [for example, 93, 94], the coversands of northern Europe [for example, 95], South America [for example, 96] and many other regions have all been dated using luminescence methods. Reported ages range from a few decades to over 100,000 years and, in many ways, luminescence dating has revolutionised the study of the geomorphology and paleoclimates of arid regions over the last three decades [84].

Apart from inland deserts, luminescence dating has also been used to date deposits from coastal dune deposits [for example, 97] where the ages obtained provide a chronological framework for processes in the coastal environment, including sea level change [for example, 98].

Sequences of fine grained eolian sediments (silt size) give rise to loess deposits which can reach hundreds of meters in thickness [85]. Fine grain dating methods have been used to provide chronological frameworks for the deposition of such sediments from places such as the Loess Plateau of China [for example, 99, 100] to the North American Great Plains [101]. Ultimately the results in such studies are used for paleoenvironmental reconstruction too. As indicated earlier, methods that use fine grains can employ IRSL stimulation of polymineral aliquots, which targets the feldspars. Alternatively, quartz from the loess can be extracted and dated using blue OSL stimulation [for example, 102].

7.2.2. Water-lain deposits

Sediments deposited by water, either fine grain or coarse grain, have been dated using luminescence methods. The abundance of sand in fluvial systems makes luminescence dating an attractive dating method in such settings. However, it is the case that sediments transported by fluvial processes are not always completely zeroed, such that the grains are often partially bleached [29, 103, 104]. Statistical approaches for dealing with the partial bleaching have been proposed but there is no consensus on how these should be applied. This has led Cunningham and Wallinga [104] to propose a protocol for analysing OSL data from fluvial sediment using a Bayesian approach. Nonetheless, in studies that have investigated fluvial sediments, luminescence chronology has provided information on modern and ancient sedimentation rates [104]. It has also enabled investigators to assess response patterns of river systems to climatic and tectonic forcing [104]. Results from dating of fluvial sediments can similarly be applied to paleoseismic and archaeological studies [103]. Comprehensive reviews of luminescence dating of fluvial sediments can be found in [103, 104].

7.2.3. Sediments of glacial origin

For luminescence dating purposes, sediments of glacial origin can generally be classified into three broad categories with regards to solar bleaching possibilities [67]. One class of sediments is transported below the glacier where no bleaching occurs at all. Hence this class would not be appropriate for luminescence dating. The second class is transported within the glacier itself where limited bleaching could occur under some circumstances but often does not. The third class of sediment is transported above the glacier and, for such sediment, some bleaching could occur. The greatest opportunity for bleaching of sediment associated with glaciers, however, is noticed in sediment transported from the glacier as outwash materials by meltwaters [67, 105]. As a result, the likelihood of the luminescence signal being zeroed increases appreciably with distance of transportation away from the glacier [105, 106, 107]. In practice, however, investigators have noted that, even for outwash deposits, partial bleaching is a problem that is encountered frequently [106]. It is thought that this occurs because the transport of sand and fine grained sediment in the proglacial environment occurs in meltwaters that are both deep and turbid such that penetration by sunlight is impeded [105]). Methods that can be used to date such sediments include single grain methods that try to identify individual grains that were adequately bleached. Current research looks at identifying mineral characteristics such as rapidly bleaching components of the luminescence signal that can be used for dating such sediments [105]. Varying dose rates during the burial history of glacigenic deposits can also be of concern and these have to be reconstructed carefully if accurate ages are to be determined [67]. Additional information on dating of glaciofluvial deposits can be found in [67, 105].

7.2.4. Earthquake related studies

A class of sediments that is of relevance to the category of earthquake related luminescence dating studies are sandy deposits emplaced during a tsunami event. Tsunami events following an earthquake commonly transport sandy materials inland to deposit them in proximal tidal marshes as well as in bogs and lakes. The sand is subsequently overlain by other materials such as peat or mud to form part of the coastal depositional sequence. Dating such sands would allow one to reconstruct the recurrence rate of the tsunami events and, by inference the earthquakes themselves, which is pertinent information for evaluating environmental hazards within the coastal zone [29]. Tsunami events, however, occur abruptly and the turbidity associated with such an event would not normally provide adequate time for effective bleaching of the sands before they are deposited. This problem is avoided by targeting sands that were previously part of the tidal flat and tidal channel environment where they lay exposed at the surface prior to the tsunami [29]. Working on the west coast of North America, close to Washington and Vancouver Island, Huntley and Clague [108] were able to date two tsunami events using this approach. Other more recent studies that have attempted to date tsunami events include [109, 110]. It should be noted, however, that the identification of tsunami deposits within the coastal zone remains a subject of debate [for example, 111].

Another class of earthquake related sediments that have been dated using luminescence methods are deposits emplaced on horizontal surfaces that have been vertically displaced by

faulting associated with earthquakes [29]. Contemporaneous deposition across the displaced surfaces will result in units of similar age on surfaces that differ in height, allowing the units to be correlated.

8. Case study: Luminescence chronology of postglacial eolian dunes from Alberta, Canada: Constraining the timing of Late Pleistocene deglaciation

To demonstrate the diverse aspects that can be encountered in a luminescence dating study, this section presents a brief outline of an investigation carried out in Alberta, western Canada [94] to constrain the timing of the retreat of the Laurentide Ice Sheet from the region. The landscape of Alberta features in excess of sixty discreet eolian dune fields that are believed to have been deposited after the retreat of the Laurentide Ice Sheet that once covered the most of Canada, east of the Rocky Mountains, about 20,000 years ago (Fig.10) [94]. Source sediments for the eolian deposits are thought to be sandy glaciolacustrine and outwash deposits associated with the retreating ice sheet. The eolian dunes in central and northern Alberta are currently stable, with many of them supporting boreal vegetation. It is thought that the eolian deposition was initiated in the immediate aftermath of the retreat of the Laurentide Ice Sheet but before the climate conditions ameliorated enough to allow vegetation to flourish. Once the climate improved, the landscape was stabilized by vegetation and the dunes have remained largely intact such that the depositional sequences they contain can be used as indicators of past environmental conditions. The exact chronology of the retreat of the Laurentide Ice Sheet from the region, however, still has to be firmly established [94, 112]. Efforts to constrain the ice sheet's retreat using radiocarbon chronology has been hampered by the scarcity of contemporaneous radiocarbon bearing material from the region [94, 113, 114] which contrasts with areas further to the east [112, 113], to the west [115] or to the south [116; 117] where radiocarbon ages have provided a sound chronological framework for Late Pleistocene deglaciation. To help address this lack of age controls for the postglacial period, a study was conceived to collect eolian dune sands from central and northern Alberta and date them using luminescence methods [94]. Ages from the eolian dunes would have a number of contributions. Because the transport and deposition of eolian sediment can only take place in ice-free conditions, ages from the dune sands would make it possible to determine by when the Laurentide Ice Sheet had retreated from the landscape. It would also be possible to construct a chronological framework for the environmental evolution of the region by putting maximum age constrains on the colonisation of the region by vegetation. A third important aspect of establishing the chronology of deglaciation of the region was that it would allow investigators to evaluate whether western Canada served as an inland migration route used by the first humans to reach the Americas. This is because the path followed by the first Americans remains a subject of contention. Evidence from central USA shows that humans had settled there by about 15,000-16,000 years ago [118]. These humans are thought to have migrated south from Beringia, having arrived from Eurasia earlier during a low sea-level phase. Around 15,000-16,000 years ago, however, some researchers believe that the Laurentide Ice Sheet still covered large parts of western Canada, including Alberta, making the region unnavigable for

humans [119]. Hence, an alternative route must have been used, possibly a coastal route [120]. By accurately constraining the timing of the retreat of the ice sheet using the chronology of dune deposition, however, it would be possible to ascertain if the ice sheets retreated early enough to allow humans to trek from Beringia through western Canada to reach central USA by around 15,000-16,000 years ago using an inland route.

Accordingly, samples for luminescence dating were collected from eolian dunes in central and northern Alberta from excavation pits as well from vertical profiles and these were sent to luminescence dating labs at the University of Washington and Utah State University for analysis [94]. At both labs the sample preparation methods followed standard procedures that included sieving, heavy liquid separation and etching with HF to separate quartz. The SAR protocol [26, 27] was used determine the paleodose and luminescence measurements were conducted on a Risø DA-20 instrument. For the dose rate, concentrations of U, Th and K were determined using ICP-MS and inductively coupled plasma atomic emission spectroscopy (ICP-AES). Alternatively TSAC in conjunction with flame photometry and beta counting were also used. More experimental details can be found in [94].
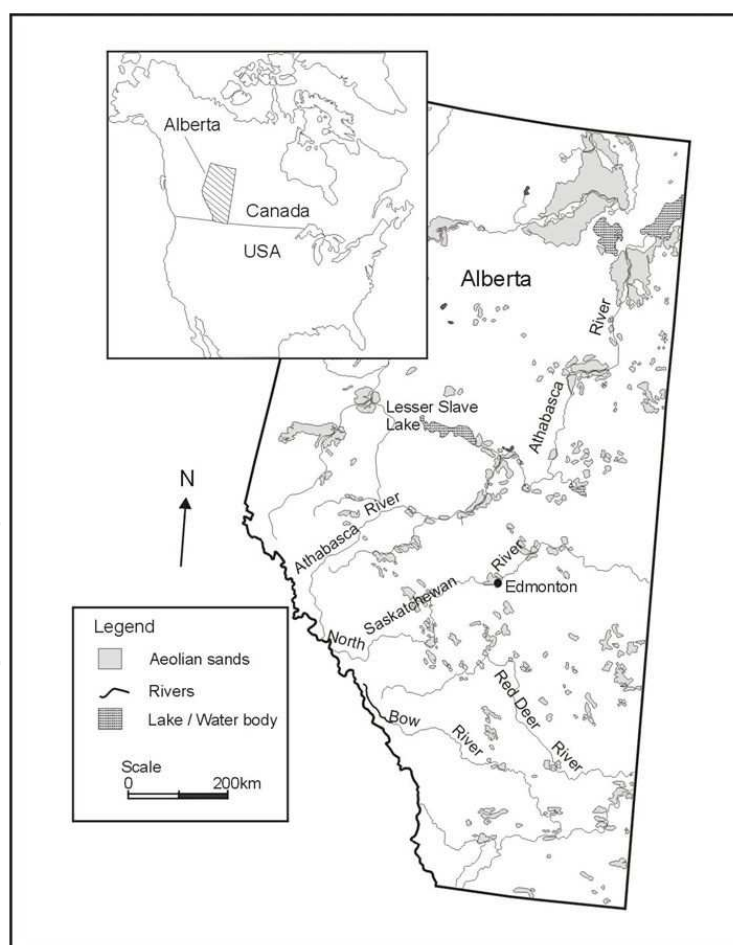

Figure 10. Eolian sands in Alberta, Canada. Samples were collected from dunes in central and northern Alberta from which quartz was extracted for dating using the SAR protocol [94].

Results of the study showed that eolian deposition in central Alberta started at least 15,000-16,000 years ago and that by around 11,000-12,000 years ago, many of the dunes had become stable [94]. A separate investigation [93] that had dated feldspar separates from dune sands in the area using the additive dose protocol obtained similar ages. The age of 15,000-16,000 years ago for eolian deposition in the study area is important because it shows that, by that time, the Laurentide Ice Sheet had retreated from the region, allowing eolian processes to operate. However, it is not yet possible to establish when exactly the eolian conditions began but, with the arrival of humans in central USA at around 15,000-16,000 ka, it is possible that the ice sheet retreated from western Canada early enough to avail the first Americans an inland route to trek southwards [94]. With regards to the subsequent colonisation of the region by boreal vegetation, the termination of eolian deposition around 11,00-12,000 years ago is consistent with other records which point to the proliferation of vegetal communities in the region associated with the postglacial climate amelioration of the Early Holocene [94]. Continuing work in the study area focuses on establishing a higher resolution chronology of eolian deposition, with emphasis on obtaining a more accurate framework for the initiation of eolian processes in the region [94].

9. Current and future trends in luminescence dating

Over the last forty years, luminescence dating has matured into a full-fledged and robust technique with many practising laboratories established across the world. Every three years, international practitioners gather for the Luminescence and Electron Spin Resonance Dating (LED) Conference where research in luminescence dating is presented. In the intervening years between the LED conferences, national and regional meetings such as the UK Luminescence Dating Conference, the German Luminescence and ESR Conference, and the New World Luminescence Dating Workshop in North America are also held. Such meetings feature fundamental research into luminescence dating methods as well as their applications in the environmental, geological and archaeological sciences. Advances in instrumentation are also always an important component at the meetings.

Prominent topics in current fundamental research include efforts to better understand the luminescence signal characteristics of both quartz and feldspar, which should allow for more accurate ages to be produced as well as for the dating ranges to be extended. For instance, ongoing studies are trying to characterise the behaviour of quartz at high doses. As indicated earlier, a number of studies have shown that at high doses (>150Gy), quartz OSL SAR protocols produce ages that underestimate the real ages of dated sedimentary units [for example, 70, 102, 121]. As a result, ongoing studies aim to identify the causes of these underestimations, particularly by looking at the characteristics of the individual components of the luminescence signal (for example, fast, medium and slow). Success in this quest could lead to the development of appropriate protocols that would make it possible to extend the dating range of quartz beyond what is currently attainable.

For feldspar, researchers have been looking at identifying IRSL signals that are less susceptible to anomalous fading which would also allow much older ages to be determined [for example,

6, 122, 123, 124]. As indicated earlier, though quartz is usually favoured for dating because it does not exhibit fading problems like those observed in feldspar, its drawback is that it saturates at much lower doses (<150 Gy). The advantage that feldspar has over quartz, however, is encumbered by the lack of fading correction methods that can be used on older ages (>100 Gy) because fading correction methods proposed to date are normally only effective for low doses [43, 44]. Hence, any methodology that would allow feldspar grains with high doses to yield accurate ages would be desirable. Accordingly, recent studies have shown that IRSL signals obtained by stimulating feldspars at a low temperature (for example, 50 °C) immediately followed by another IRSL measurement at an elevated temperature (for example, 290 °C) yields a signal that has a lower fading rate. This measurement protocol is referred to as post-IR IRSL [122] and results provided to date suggest this is a promising approach that has the potential to extend the dating range of feldspars significantly [6, 125].

Developments in instrumentation are also keeping pace, with new luminescence measurement systems being developed [for example, 126]. Also worth mentioning is the recent development of portable OSL devices capable of conducting rapid measurements in the field [for example, 127]. Though limited in capability compared to regular OSL readers, such portable devices could, with additions such as internal X-ray radiation sources, introduce more options in the sphere of luminescence signal collection.

Overall, the field of luminescence dating is a vibrant research area and, if the recent past is any indication of what the future holds, it is a discipline guaranteed to witness innovative developments in the coming years. With the continual refinement of both the laboratory procedures as well as the equipment, we should see chronologies being reported with greater precision and accuracy.

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