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Chapter 11

Luminescence Dating: Applications in Earth Sciences and Archaeology

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Abstract

Over the last 60 years, luminescence dating has developed into a robust chronometer for applications in earth sciences and archaeology. The technique is particularly useful for dating materials ranging in age from a few decades to around 100,000–150,000 years. In this chapter, following a brief outline of the historical development of the dating method, basic principles behind the technique are discussed. This is followed by a look at measurement equipment that is employed in determining age and its operation. Luminescence properties of minerals used in dating are then examined after which procedures used in age calculation are looked at. Sample collection methods are also reviewed, as well as types of materials that can be dated. Continuing refinements in both methodology and equipment promise to yield luminescence chronologies with improved accuracy and extended dating range in the future and these are briefly discussed.

Keywords: luminescence dating, thermoluminescence (TL), optically stimulated luminescence (OSL), paleodose, dose rate, earth sciences, archaeology

1. Introduction

Luminescence dating refers to age-dating methods that employ the phenomenon of luminescence to determine the amount of time that has elapsed since the occurrence of a given event. In this chapter, the application of luminescence techniques in dating geological and archaeological events is examined. Generally, the term luminescence dating is a collective reference to numerical age-dating methods that include thermoluminescence (TL) and optically stimulated luminescence (OSL) dating techniques. Other terms used to describe OSL include optical dating [1] and photon-stimulated luminescence dating or photoluminescence dating [2]. Luminescence dating methods are based on the ability of some dielectric and semiconducting

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materials to absorb and store energy from environmental ionizing radiation. In earth sciences and archaeological applications, the dielectric materials are usually minerals such as feldspar and quartz. The materials are also sometimes referred to as dosimeters [3]. Environmental ionizing radiation for earth science and archaeological applications typically comes from radioactive elements within the immediate surroundings of the mineral grains as well as from cosmic radiation. When the minerals are stimulated, they release the stored energy in the form of light, from which the term luminescence is derived. Stimulation of energy release by heating is referred to as TL. When light is used, on the other hand, the technique is described as OSL. Measuring the amount of energy released in conjunction with a determination of the rate at which the energy was accumulated allows an age to be calculated, indicating time that has elapsed since the storage of energy began. Luminescence methods can generally be used to date materials that range in age from a few decades to about 100,000 years. However, ages of up to several hundred thousands of years have been reported in some studies [4]. Therefore, the method can be used for dating events of Late Pleistocene and Holocene age (ca. < 126 000 years). Radiocarbon dating is a technique that can also be used to date some materials of Late Pleistocene or Holocene age if they bear carbon. Hence, when dealing with materials that do not contain organic carbon, luminescence dating can serve as an optional chronometer to radiocarbon dating. Furthermore, the age range that can be dated using luminescence techniques is greater than that of radiocarbon dating, and this provides researchers with a viable alternative dating method.

This chapter presents a brief examination of how luminescence is used in earth sciences and archaeology to measure time. In both fields of study, the imperativeness of assigning a temporal scale to events and processes is an inherent aspect of the discipline. Hence, the role played by dating cannot be overstated. The chapter is not intended to be used as a practice manual. Rather, the aim is to provide a primer that acquaints scholars who may be familiar with the science of luminescence but are not accustomed to its application as a dating method. Thus, the chapter comprises eight sections. Following an introductory look at the development of luminescence dating in Section 1, principles of luminescence dating are examined in Section 2. In Section 3, basic luminescence measurement equipment and sample stimulation mechanisms during measurement are explored after which luminescence properties of some common minerals are examined. The determination of parameters used in the luminescence age equation is discussed in Section 5, and this is followed by a look at materials that can be dated with luminescence methods in Section 6. Section 7 looks at methods used in sample collection and preparation prior to the analysis. In Section 8, the chapter concludes with a look at future developments in luminescence dating.

1.1. Historical development of luminescence dating

The ability of some minerals to luminesce when stimulated is not a recent finding. The earliest recorded observation of the behavior in minerals has been attributed [5] to Robert Boyle, who in 1664 recounted his discovery [6] that a diamond that had been loaned to him could emit light when heated. Subsequently, Boyle would describe the phenomenon as 'self-shining' [7]. Throughout the eighteenth, nineteenth and early parts of the twentieth centuries a lot of activity went into examining the effects of TL such that by the middle of the twentieth century,

physicists were fairly familiar with the phenomenon. The focus of this section, however, is not on the history of the science of luminescence but rather on the history of the emergence of the application of luminescence as a dating technique for geological and archeological materials. That inception can be traced back to approximately seven decades ago, to a period during which experiments were being conducted into applications of TL [8, 9]. Zeller et al. [10] noted that research into applying TL to determine geological ages of rocks using minerals began at the University of Wisconsin around 1950. Progress in dating geological materials, however, was hampered by a limited understanding of the luminescence process in rocks and accurate ages were hard to come by [11]. It is in the dating of heated archaeological artifacts that progress was realized. In 1953, Daniels et al. [9] had proposed that TL from ancient pottery could be used to determine its antiquity [12]. The idea was premised on the recognition that heating the pottery to high temperatures (>500°C) during production had the effect of erasing any previously accumulated energy from constituent mineral grains. Subsequently, Kennedy and Knoff [13] provided some basic aspects of dating heated archaeological materials using TL, though the approach had not been tested on actual samples as yet. Around the same time, Grogler et al. [14] provided additional experimental details on how a dating experiment measuring TL of pottery could be conducted. It was in 1964 that Aitken et al. [12] designed a study that sought to determine the relationship between TL glow of an artifact and its archaeological age. Results demonstrated that the luminescence intensity emitted by the samples was linearly proportional to radiocarbon ages of sediments from which the samples were obtained. The positive results noted in these early studies gave impetus for further refinements in TL protocols, and throughout the rest of the 1960s and 1970s, the dating method gained a foothold in archaeological studies.

A major development in luminescence studies occurred when TL dating was extended to determining burial ages of unheated sediments. The evolution appears to have followed two parallel paths, one in the West and another in former Eastern Bloc countries. However, it seems there was minimal interaction between the two geographical regions, especially in the early stages. In the West, some of the earliest work includes a study [15] that looked at TL signals of deep-sea sediment that mostly comprised foraminiferal shells. The investigators [15] considered the signals to be from calcite and noticed that the TL intensity increased with depth. Later, another study [16] investigated a deep-sea sediment core that comprised predominantly siliceous plankton and reported results similar to those presented earlier [15]. Subsequent studies by Wintle and Huntley [17] provided additional TL data from deep-sea sediments that also showed increasing signal intensities with depth. However, it was later observed [17] that the TL signals from deep-sea cores actually came from detrital minerals that were mixed with the plankton. Significantly, it was also suggested [17] that what was being dated was the last time the ocean sediments had been exposed to sunlight. Though it was some time before researchers fully understood the zeroing mechanism for unheated sediment [18], that discovery was extremely important for sediment dating since it meant that the exposure of sediments to sunlight had the same zeroing effect of accumulated energy in sediment grains as did heating in pottery.

In former Eastern Bloc countries, the analysis of unheated terrestrial sediments using TL appears to have begun sometime during the 1960s. Early published reports from the former

Soviet Union include a study [19] that examined TL signals of Quaternary deposits. A few years later, Morozov [20] presented relative ages of Quaternary sediments from Ukraine that had been dated using TL methods. The study was mainly based on the recognition that luminescence signal intensities increased with depth, which was interpreted as commensurate with age. Morozov [20] also suggested that the signal was coming from quartz in the sediments. Shortly afterwards, Shelkoplyas [21] reported a range of Quaternary TL ages obtained from soils and loess deposits [18]. Throughout the 1970s, researchers in Eastern Bloc countries [22–29] as well in China [30] reported studies in which TL was used to date Quaternary deposits. The accuracy of some of these early ages, however, is questionable [18], not least because zeroing mechanisms were not well-understood at the time. In other studies, efforts were directed at understanding TL characteristics of dosimeters, especially quartz [31–33].

Around the time Wintle and Huntley [17] discovered that TL signals in their deep-sea cores were coming from detrital mineral grains mixed with the plankton, they became aware of the work by Eastern Bloc researchers who had worked extensively with terrestrial sediments. Ultimately, they realized that TL dating could be applied much more broadly to date Quaternary deposits [18]. These developments resulted in the landmark publications by Wintle and Huntley [17, 34, 35] in which TL dating of sediments was outlined. With increased research throughout the early 1980s, dating procedures improved as efforts were made to standardize procedures [18]. However, optimal conditions for solar resetting of sediments remained unclear to researchers and this hampered the accuracy of TL ages. Researchers who examined the problem include Huntley [36] who investigated solar resetting of sediments from various environments and proposed a method to address the issue of inadequate zeroing. By 1985, another monumental step in luminescence dating would be realized when

Authors	Topic	Source
Murray and Olley (2002)	Luminescence dating of sedimentary quartz	[40]
Feathers (2003)	Luminescence dating in archaeology	[41]
Lian (2007)	Optically stimulated luminescence dating	[42]
Duller (2008)	Luminescence dating guidelines for archaeology	[43]
Fuchs and Owen (2008)	Luminescence dating of glacial sediments	[44]
Preusser et al. (2008)	Luminescence dating: principles and applications	[45]
Rittenour (2008)	Luminescence dating of fluvial deposits	[46]
Singhvi and Porat (2008)	Luminescence dating in geomorphology	[47]
Wintle (2008)	History of luminescence dating over the last 50 years	[39]
Wintle (2008)	Luminescence dating: history and prospects	[3]
Rhodes (2011)	Luminescence dating of sediments	[48]
Liritzis et al. (2013)	Luminescence dating in archaeology	[49]

Table 1. Recent review studies examining luminescence dating and its applications.

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Huntley et al. [11] reported that a light source could be used to stimulate energy release from a dosimeter during measurement instead of heating as was used in TL. This led to the development of OSL dating which offered a number of advantages over TL methods when dating unheated sediments. With further equipment and methodological refinements, there was a burgeoning of OSL dating studies of Quaternary sediments throughout the 1990s that saw luminescence dating emerge as a robust dating technique. Over the last two decades, the technique has developed further [37–39], and today, it is the method of choice for dating detrital sediments of Late Pleistocene and Holocene age as well as previously heated archaeological artifacts. **Table 1** presents some recent review studies that have examined various aspects of luminescence dating and its applications.

2. Principles of luminescence dating

2.1. Luminescence behavior of dielectric materials used in dating

Many minerals such as quartz, feldspar, calcite and zircon are dielectric materials and, when subjected to ionizing radiation, they are able to store energy in their crystal lattices. In natural geological and archeological settings, the ionizing radiation emanates naturally from the immediate surroundings of the minerals. Cosmic radiation may also contribute a small component. If the minerals used in dating are stimulated, they release the energy by luminescing and, within certain constraints, the energy released is proportional to the stored energy.

2.2. Luminescence dating equation

In luminescence dating, the energy given out by the minerals or dosimeters following stimulation is measured using appropriate instrumentation. This energy is referred to as the paleodose [50]. In order to determine an age, the rate at which the energy was accumulated by the dosimeter, or the dose date, is also ascertained. The quotient of the paleodose and the dose rate, as indicated in Eq. (1), is the luminescence age.

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

If the mineral grains were emptied of all previously accumulated energy prior to the latest energy storage episode, the age obtained will denote time that has elapsed since the start of that episode. Hence, both in geology and archaeology, the luminescence age simply connotes time that has passed since the occurrence of a specific energy zeroing event. In geology, this might be a geomorphic event that exposed sediment to sunlight. Zeroing by sunlight is also sometimes referred to as optical bleaching [3]. In pottery, zeroing would normally occur during a firing event associated with the manufacture.

2.3. Electron trapping mechanisms

Mechanisms by which minerals store energy in their crystal lattices as a result of ionizing radiation are complex [50–52]. However, it is thought that ionizing radiation drives mineral

crystals into a metastable state where electrons are displaced from their parent nuclei. The positions from which the electrons have been evicted act as holes. The electrons and holes then diffuse within the mineral crystals and become trapped separately at lattice defects. Examples of common defects include a negative ion missing from its lattice position, a negative ion positioned in an interstitial site or the presence of impurity atoms in the lattice through substitution [52]. Other more complex trap types exist [52]. **Figure 1** depicts an energy level diagram that is used to visualize the trapping mechanisms involved in luminescence in crystalline materials. The depth of the trap (T) below the conduction band, indicated by 'E' (**Figure 1**) is a reflection 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. If the crystal lattice is stimulated using an appropriate mechanism, for example, by heating to an adequately high temperature or by exposure to an optical source with a suitable wavelength, trapped electrons will be evicted out of the traps. Once evicted, the electrons diffuse within the crystal lattice until they reach a site that is attractive to electrons. Such sites are referred to as recombination centers. Some recombination centers emit energy in the form of light when they capture electrons. Where stimulation is conducted by heating, the effect would be referred to as TL. When stimulation is by optical means, OSL will be obtained. The diffusion of evicted electrons to their recombination centers occurs fairly rapidly to the extent that the time between stimulation and recombination can be viewed as instantaneous. Effective recombination centers are usually those sites in the lattice where electrons are missing. These are the holes created when the materials are exposed to ionizing radiation (**Figure 1a**). The intensity of the luminescence that is obtained when a material is stimulated is proportional to the number of electrons that are trapped in the material which, in turn, is commensurate with the energy absorbed from the ionizing radiation [50, 51]. However, despite the energy

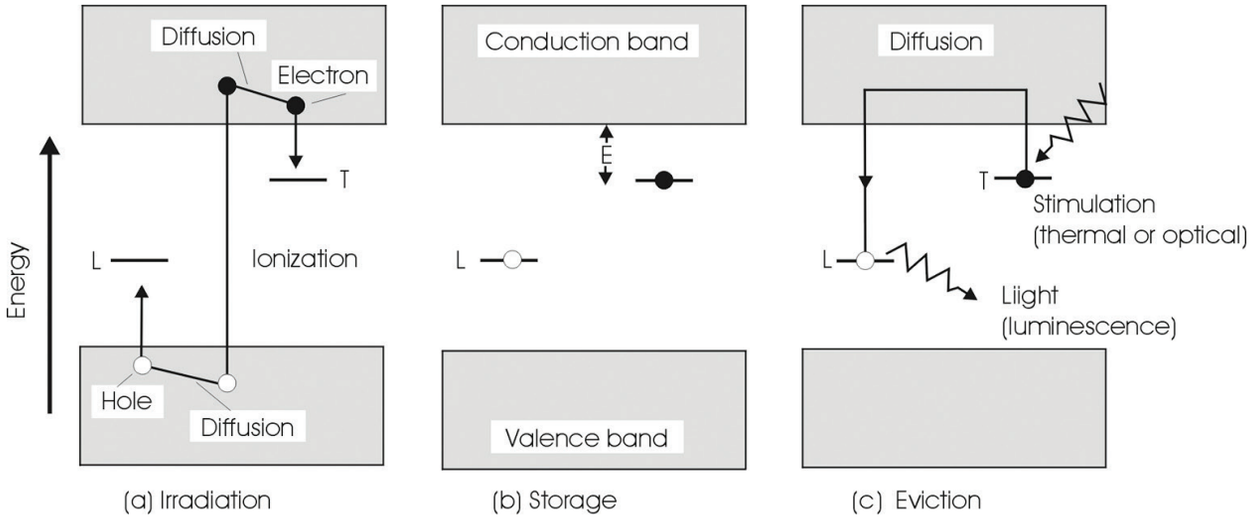


Figure 1. An energy level diagram that illustrates how ionizing radiation creates luminescence centers in crystal lattices (redrawn from Ref. [50]). (a) Following irradiation, electrons are expelled from their original sites leaving holes behind. Both electrons and holes diffuse within the lattice. (b) At appropriate sites, electrons are trapped while holes may become localized. (c) Thermal (TL) or optical (OSL) stimulation of the material results in electrons being evicted from the traps. Evicted electrons that reach luminescence centers result in light being emitted.

storage mechanism being the same for a given mineral, the sensitivity to radiation may vary greatly between samples, an aspect that has important implications for dating procedures as will be shown later.

Importantly, for dating purposes, the number of electron traps within any mineral lattice is finite. As a result, when minerals are exposed to ionizing radiation for an extended period, the traps become exhausted such that energy can no longer be stored efficiently. This effect is referred to as saturation. In dating, saturation determines the upper limit beyond which samples cannot be dated using luminescence techniques. Materials that are subjected to very high dose rates will have the number of traps exhausted more rapidly such that the specific age representing the upper age limit will depend on both the number of traps present as well as the dose rate.

2.4. Natural sources of ionizing radiation

In geological and archaeological dating applications, natural sources of ionizing radiation that contribute to the trapped energy in mineral grains include isotopes of uranium (^{238}U and ^{235}U) and thorium (^{232}Th) decay chains, potassium (^{40}K) and rubidium (^{87}Rb). The elements are found in natural materials in very low concentrations (about 3–10 parts per million for uranium and thorium and less than 5% for potassium, where ^{40}K is one part in 10,000). Despite the low concentrations, these radioactive isotopes collectively emit enough radiation to induce luminescence that is detectable for dating purposes. The radiation emitted includes alpha and beta particles as well as gamma radiation. Beta particles and gamma rays have penetration ranges of about 0.02 cm and 20 cm in earth materials, respectively, whereas alpha particles penetrate about 0.02 mm [51].

An additional though smaller radiation component received by earth materials comes from cosmic radiation. Cosmic rays from outer space consist of a soft and a hard component. On earth, surface substrate absorbs the soft component such that it cannot penetrate deeper than 50 cm. The hard component, however, largely comprising muons, penetrates deeper and is lightly ionizing. Hence, only the hard component is relevant to luminescence dating. On earth, the intensity of the hard component is also influenced by both latitude and altitude. Special formulae for evaluating cosmic ray contribution to dose rate have been developed for luminescence dating [53].

3. Basic luminescence measurement equipment and sample stimulation mechanisms

For dating studies, the primary aim of TL and OSL measurements is to determine the amount of energy that has been stored in the mineral grains of a given material since the start of the event that is being investigated. As outlined above, two main methods are used to stimulate energy release in luminescence studies. Heating allows TL to be measured, whereas stimulation using a light source is used for OSL measurements.

3.1. The luminescence reader

The basic layout of equipment used to measure luminescence in geological and archaeological dating is shown in **Figure 2**. Modern luminescence dating systems commonly possess both TL and OSL measurement capabilities [54, 55]. To conduct a measurement, samples are usually loaded on discs about 1 cm in diameter that sit on an appropriate sample holder in multiples. These are then introduced into the device, commonly referred to as a luminescence reader [44] and selectively moved into position for measurement. The luminescence signal from the sample is captured by a photon detector system [1] for example, photomultiplier tube (PMT) or charge-coupled device (CCD) camera after passing through optical filters. When conducting TL measurements, the filters exclude infrared signals from the heating but permit blue or violet emissions to pass through. In OSL measurements, wavelengths used for stimulation are rejected by the filters, whereas violet and near ultraviolet wavelengths are usually transmitted. Output from a TL measurement is distinctly different from that obtained using OSL stimulation (**Figure 2**).

3.2. Thermal stimulation

When using thermal stimulation, samples are heated at rates approaching 20°C per second. On reaching a temperature that corresponds to the trap depth 'E' (**Figure 1**), usually characteristic

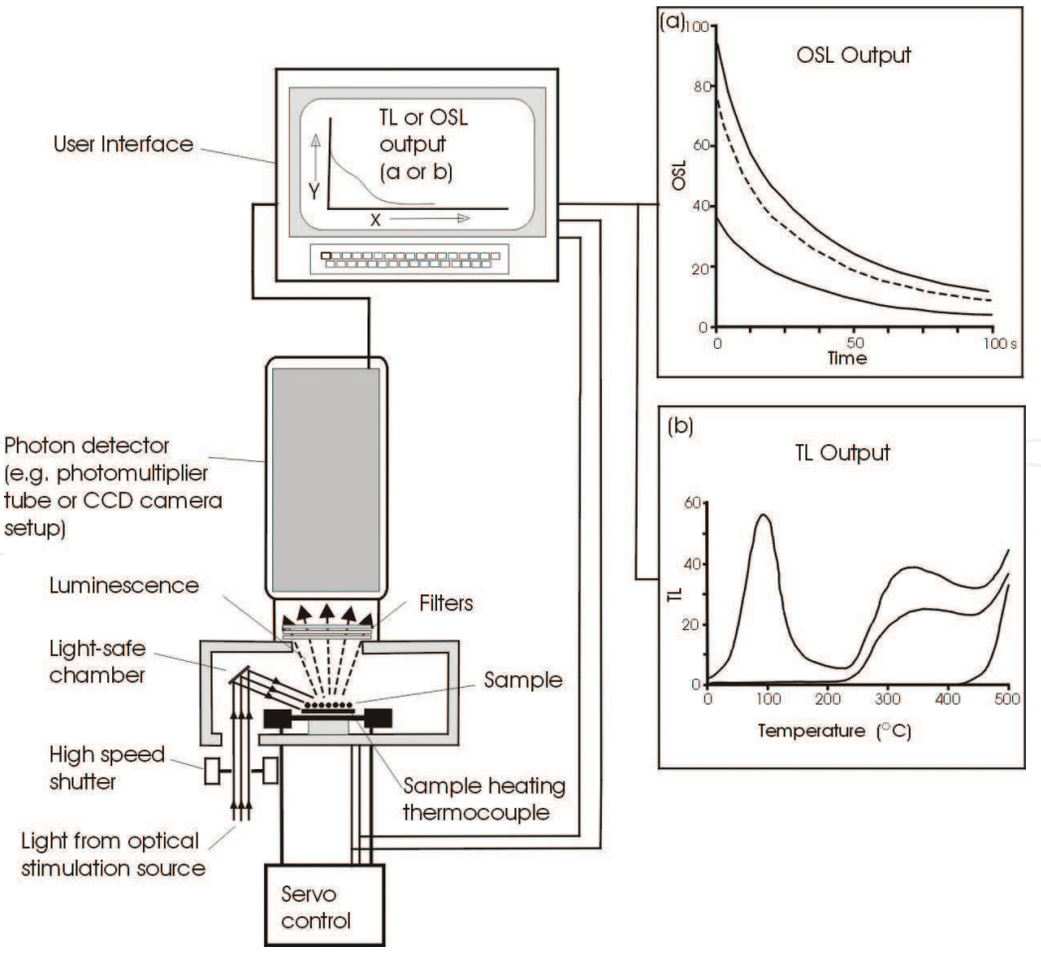


Figure 2. Main components of a luminescence reader that measures both TL and OSL signals (modified after Ref. [56]).

of a given trap type, trapped electrons are rapidly evicted [51] into the conduction band. The equation below gives the probability per second at temperature T that the energy provided is sufficient to evict a trapped electron at depth 'E' into the conduction band:

$$\rho(T) = s(T)\exp(-E/kT) \tag{2}$$

where $s(T)$ is a temperature dependent factor associated with the lattice vibrational frequency and change in entropy and k is Boltzmann's constant [52]. Once evicted, electrons are free to be re-trapped at the same site, be trapped at a different site or get to a recombination site where luminescence occurs. The eviction temperature is depicted by a peak in emission on a plot of the luminescence signal versus temperature which is referred to as a TL glow-curve. If the heating continues, all the electron traps will be emptied. A glow-curve that is obtained after the first heating of a sample is given in **Figure 3**. Heating the sample again soon after the first heating will produce a different curve. The second curve corresponds to incandescence that is usually observed when any material is adequately heated to an elevated temperature. Hence, from this second heating, there will be no luminescence from trapped electrons that had accumulated from ionizing radiation since the last zeroing event.

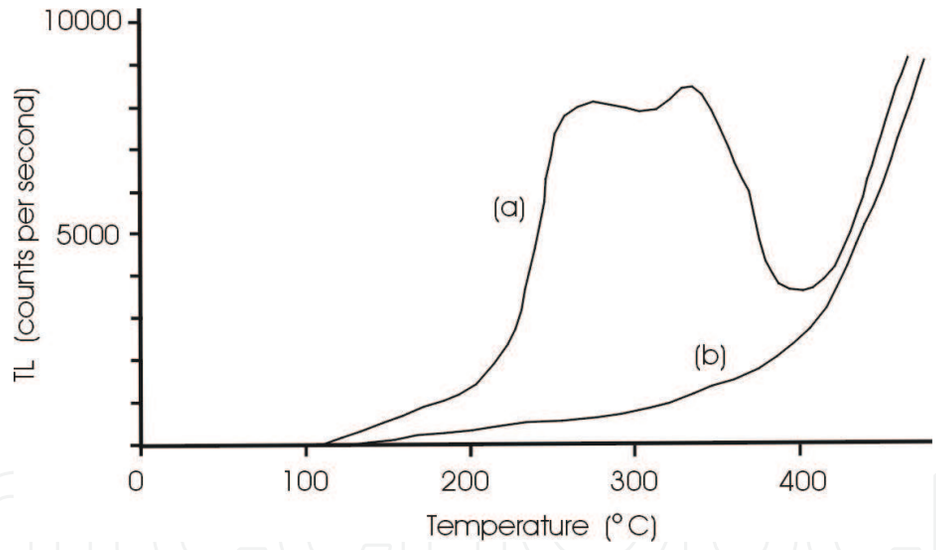


Figure 3. Illustration of glow-curves obtained following thermal stimulation. (a) A signal yielded after the first heating of a quartz sample with trapped electrons (paleodose). (b) A glow-curve from incandescence resulting from the second heating of the sample to a high temperature. It is important to note that incandescence is also realized above 400°C during the first heating (redrawn from Ref. [56]).

3.3. Optical stimulation

In optical stimulation, electrons are expelled from their traps using a source of a chosen wavelength. Commonly used sources in luminescence dating include blue, green or near-infrared wavelengths. The rate at which trapped electrons are evicted is influenced by the rate at which stimulating photons are emitted by the source as well as by the sensitivity of the trap types to optical stimulation. Generally, however, starting with a concentration of 'n' trapped electrons

and if '*p*' is the probability for electrons to be evicted by an optical source to the conduction band per unit time, 'n' will change according to Eq. (3):

$$\frac{dn}{dt} = -np \tag{3}$$

assuming that no re-trapping of freed electron occurs. Resolving Eq. (3) shows that as traps are emptied, the concentration of trapped electrons decays exponentially as given in Eq. (4) [5].

$$n(t) = n_0 e^{-pt} \tag{4}$$

where n_0 is the concentration of trapped electrons prior to the stimulation. If all freed electrons reach recombination sites instantaneously, the luminescence intensity will be proportional to electrons being evicted from the traps. Thus from Eq. (3) and Eq. (4):

$$I_{OSl}(t) \alpha \left| \frac{dn}{dt} \right| = n_0 p e^{-pt} \tag{5}$$

The exponentially decaying emission curve obtained is referred to in OSL dating as a shine-down curve (**Figure 4**). With continued stimulation, a point is reached where all trapped electrons that are susceptible to optical stimulation are depleted. If all the photons released during stimulation are integrated, the total luminescence energy released by the mineral can be ascertained.

Factors that influence the sensitivity of a trap type to optical eviction include characteristics of the trap as well as the wavelength of the optical source. Generally, however, eviction rates are faster when shorter wavelengths are employed. Electron eviction from some traps could require more energy than that provided by an optical source. To circumvent that limitation,

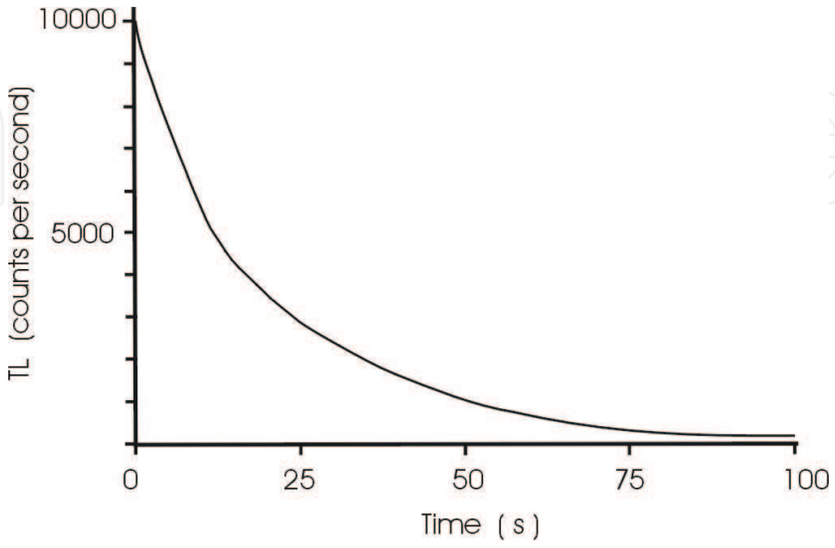


Figure 4. A shine-down curve obtained following the optical stimulation (OSL) of a hypothetical mineral sample for about 100 s (modified after Ref. [51]).

thermal assistance is used to attain the energy threshold required for eviction. This allows longer wavelengths that would not normally be employed for optical stimulation to be used in dating [50].

Generally, sources for optical stimulation are selected such that separation can be made between wavelengths of the source used for stimulation and those of signals emitted by the minerals being analyzed. That separation is usually aided by the use of optical filters. As an illustration, the main emissions for quartz and feldspar are in the near-ultraviolet (356 nm) and violet (410 nm) regions of the electromagnetic spectrum. Thus, filters that are employed when analyzing quartz and feldspars have windows in those respective regions but exclude wavelengths used for stimulation, for example, blue for quartz and near infrared for feldspar.

3.4. Advantages of OSL over TL

OSL dating has a number of inherent advantages compared with TL when analyzing sediments that have been zeroed by exposure to sunlight. Investigations have shown that solar bleaching of electron traps that are stimulated by TL proceeds more slowly than with traps that are sensitive to OSL [50]. In the study summarized in **Figure 5**, after 20 h of exposure to

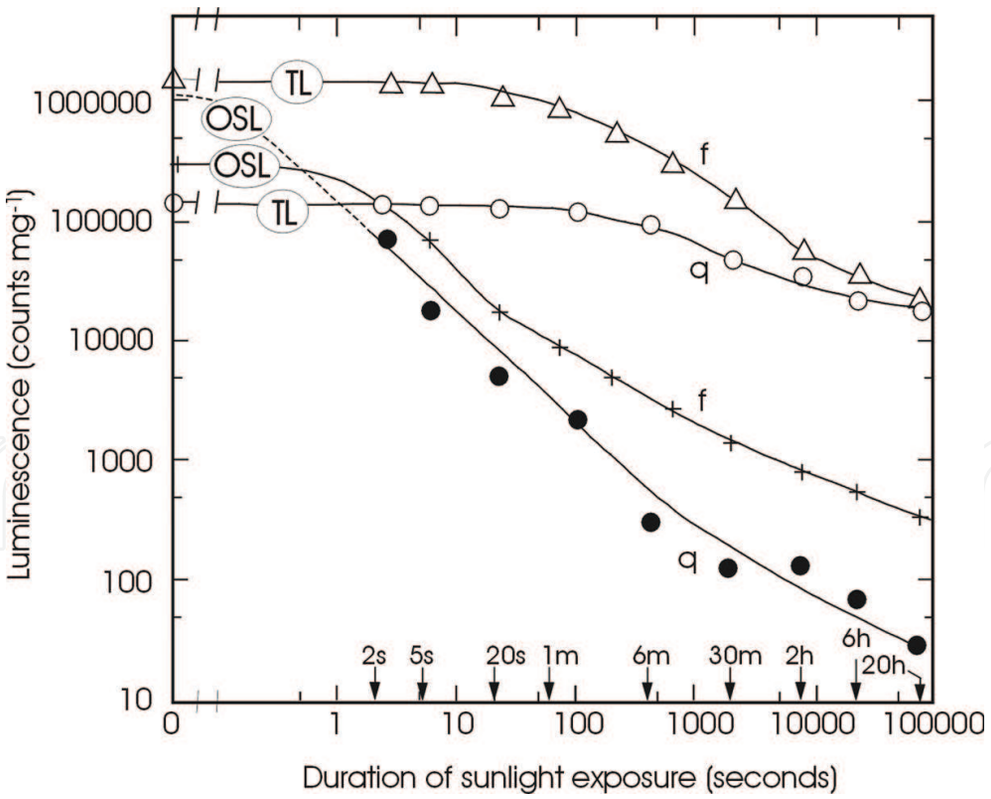


Figure 5. Bleaching rates of quartz (q) and feldspar (f) stimulated using TL and OSL (green light) conducted by Godfrey-Smith et al. [57] (redrawn from Ref. [50]). The slower bleaching curves are from TL signals. For quartz, TL measurement focused on a peak between 320 and 330°C whereas for feldspar, a peak at 310–320°C was analyzed. Detection of TL signals was through a window with a center at 400 nm (violet). For the OSL, both quartz and feldspar used a green laser and a detection window of 380 nm which is violet to near-UV [50].

sunlight, both quartz and feldspar were shown to have less than 0.1% of the signal originally present in the mineral remaining [57]. The TL signal that remained following the same period of bleaching, on the other hand, was a few orders of magnitude higher than the OSL signal. In practical terms, the slower bleaching of TL traps by solar energy means that higher residual signals will be found in unheated sediments analyzed using TL, to the extent that it is difficult to date very young samples using TL [11, 50]. Thus, OSL analysis is generally preferred for dating sediments that were not reset by heating [50]. For dating materials that have previously been zeroed by heating, however, such as archeological artifacts, TL remains an appropriate stimulation mechanism.

4. Luminescence properties of some common minerals

Many minerals will luminesce when stimulated using an appropriate source following a period of exposure to ionizing radiation. However, not all such minerals are suitable for use in luminescence dating. Today, luminescence dating primarily employs quartz and feldspar. Zircon and calcite have been tried in some studies but both minerals are associated with a number of complications. As a result, they are not commonly used in luminescence dating at present. This section examines the luminescence properties of the four minerals. In the discussions below, natural dose refers to energy acquired from natural radiation sources by a mineral grain in its field setting. This is differentiated from an artificial dose that a sample would obtain when irradiated using an artificial source in a laboratory setting.

4.1. Quartz

Quartz is the most commonly used mineral in luminescence dating because it offers a number of advantages when contrasted with alternatives. Due to its resistance to both chemical weathering and mechanical abrasion, it is very stable at the earth's surface. As a result, it is one of the most abundant minerals in clastic depositional environments. Quartz's luminescence properties are also very stable. Additionally, it does not have an internal source of radiation as a major element of its composition. Thus, the ionizing radiation that quartz grains receive in nature is usually from an external source, which simplifies dose rate calculation procedures. Situations exist, however, where quartz grains may contain very low levels of uranium but these are rare [51].

4.1.1. Quartz TL properties

Quartz that has a natural dose displays TL peaks at 325˚C and 375˚C [58]. TL dating usually employs the 375˚C peak which is very stable and is thought to be the result of AlO_4 lattice impurities that serve as hole traps [59]. Under sunlight, the 325˚C peak bleaches much more rapidly than the 375˚C peak [60]. Quartz that has been irradiated artificially also shows a peak at 110˚C [**Figure 6**]. With regards to emissions, heating quartz above 300˚C shows a natural TL emission band around 460–480 nm (blue) and another in the region of 610–630 nm (orange). Laboratory irradiated quartz has a TL emission band below 300˚C in the region of 360–420 nm.

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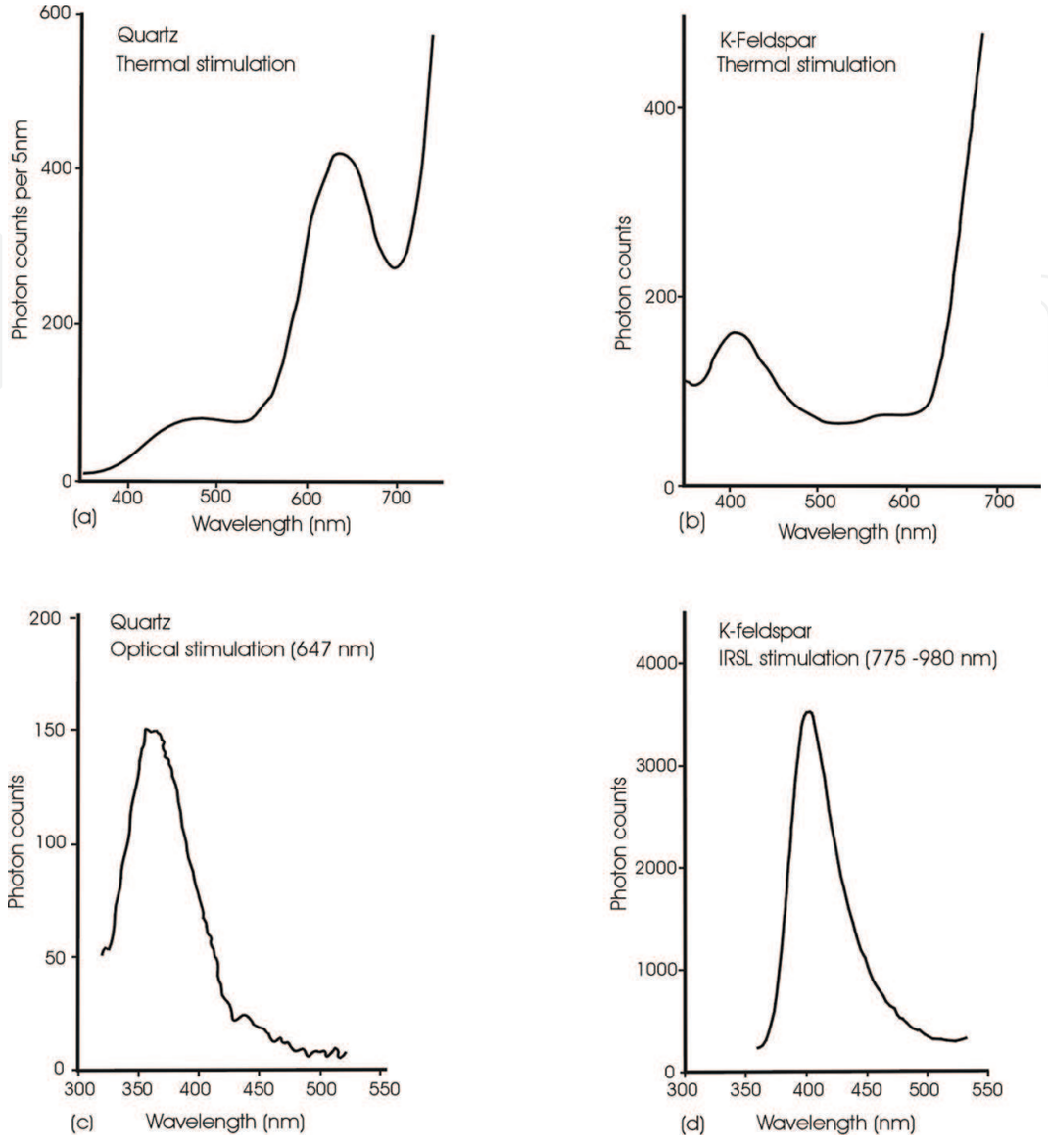


Figure 6. Examples of TL (a, b) and OSL (c, d) emission spectra (adapted with permission from Ref. [60]). For TL spectra, the sharp rise in emissions beyond 650 nm is largely from incandescence (rather than from electrons evicted from traps).

4.1.2. Quartz OSL properties

Quartz has been shown to luminesce when stimulated by wavelengths from any part of the visible spectrum [60]. Most current OSL studies, however, prefer using blue light for stimulation because of the higher OSL intensities it yields [55]. Investigations have demonstrated that the OSL signal of quartz can consist of at least three or more components that are referred to as fast, medium and slow, based on their decay rates [61, 62]. To separate the components during stimulation, constantly increasing power is used to give linearly modulated OSL (LM-OSL) [63]. Most regular dating procedures, however, employ a constant power (continuous wave–CW) and are unable to resolve the components. Through the use of heat treatments or stimulation for limited times (to exclude the slower components), desired signals can be targeted

when using CW stimulation. Emission bands that are observed in quartz OSL are in the range of 360–420 nm (**Figure 6**), which corresponds to emissions that are seen in quartz TL at temperatures below 300˚C [60] (**Figure 7**).

4.2. Feldspar

Feldspar is another widely used mineral in OSL dating. It constitutes about 60% of the earth's crust and even though it weathers more rapidly than quartz, it is also a very common mineral at the earth's surface. In terms of chemistry, feldspars are aluminosilicates that form solid solution series with potassium (K) calcium (Ca) and sodium (Na) as end members of a ternary system. Since potassium has an isotope that contributes ionizing radiation in luminescence dating, the potassium in K-feldspars has to be treated as a source of internal dose, in addition to dose contributions from sources external to the grains. As a result, when dating feldspars, it is necessary to separate K-feldspars from Ca and Na-feldspars and analyze them separately.

Compared with quartz, feldspar has a number of attractive luminescence features. First, feldspar emissions are generally brighter than those from quartz which produces stronger signals. This means that smaller doses can be measured during analysis. Second, the internal dose from potassium is not susceptible to external influences such as variations in pore water and this allows dose rates to be ascertained more accurately. Third, feldspar can be stimulated using infrared radiation which allows effective separation to be made between the stimulation source and emission wavelengths. The main drawback for feldspar, however, is its susceptibility to anomalous fading [64]. Anomalous fading occurs when trapped electrons reside in their traps for shorter periods than what would be predicted by physical models such that the luminescence intensity drops over time from the time of irradiation. Ultimately, the result of anomalous fading is that most feldspar grains yield equivalent doses that are slightly lower than they would in the absence of fading.

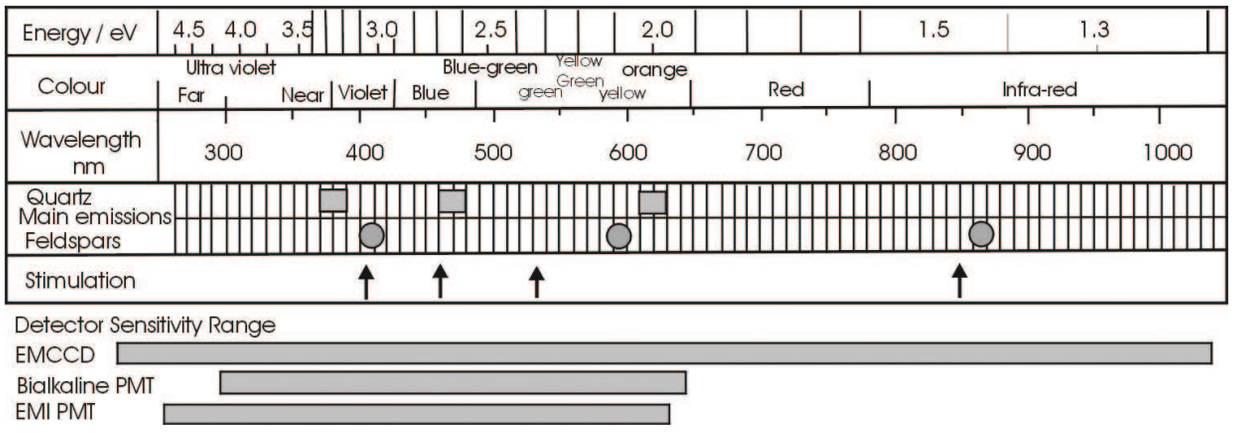


Figure 7. Main emission wavelengths for quartz and feldspars used in luminescence dating as well as wavelengths employed for stimulation. Sensitivity ranges for some detectors are also shown. Standard bialkaline photomultipliers (PMT) and EMI 9235QB PMTs do not detect emissions beyond 650 nm, whereas electron multiplying CCD (EMCCD) cameras can capture the whole range (modified after Ref. [54]).

Correction methods have been developed for dealing with anomalous fading when dating feldspars [65, 66].

4.2.1. Feldspar TL properties

Various studies have shown that K-feldspars extracted from sediments yield natural TL signals that display peaks at 280 and 330°C [60]. In terms of emission wavelengths, K-rich feldspars have been reported [67] to show maximums in the range of 390–440 nm (violet to blue). Conversely, emissions for some plagioclase feldspars have been reported to appear in the range of 550–560 nm (blue-green). Other studies, however, have intimated at a more complex emission pattern for feldspars [68].

4.2.2. Feldspar OSL properties

Optical stimulation of luminescence from feldspars has been investigated using visible light. Early studies employed lasers which included the 514.5 nm wavelength from argon and the 633 nm (red) wavelength from krypton. The emissions were then monitored at shorter wavelengths [1, 57] and shown to be centered around 400 nm [69]. The application of OSL stimulation in dating feldspars, however, has been relatively limited because near-infrared stimulation (discussed below) has been shown to be a more desirable approach. Nonetheless, a study [70] comparing green light stimulation (GLSL) of feldspars with infrared stimulated luminescence (IRSL) reported data suggesting that at 10°C, GLSL signals were more stable than IRSL signals. This would indicate that different trap types might be involved [50]. Apart from green and red stimulation, luminescence in feldspar has been demonstrated using a range of other wavelengths in the region spanning 380–1020 nm [71].

4.2.3. Feldspar IRSL properties

As mentioned above, wavelengths in the near infrared region (peaking around 880 nm) can also be used to induce luminescence in feldspars. Since this effect was first noticed [72], most research in optical dating of feldspars has focused on IRSL stimulation. The main advantage of using IRSL is that the rest of the visible spectrum can then be used for emission detection. Fine-grained sediments containing mixtures of both plagioclase and K-feldspars have also been demonstrated to display a major stimulation peak around 845 nm as well as a weaker one at 775 nm [73]. Today, most studies that use feldspar for optical dating employ light emitting diodes (LEDs) for stimulation that have emission peaks at around 880 ± 40 nm. LEDs are much cheaper than lasers and are widely available, making them a desirable alternative. Sedimentary K-feldspars stimulated using IRSL show a major natural emission peak at 410 nm (**Figure 6d**) and another minor peak in the range of 300–350 nm [74]. Some IRSL studies [75] have reported additional natural emission maxima for K-feldspars at 280, 560 and 700 nm. With plagioclase feldspar, an IRSL emission peak has been identified at 570 nm.

Feldspars stimulated using IRSL following the administration of a laboratory dose also exhibit an emission peak at 290 nm. That peak is not observed in feldspars that have a natural signal. When not required during dating, the peak can be removed by preheating the sample to an appropriate temperature.

4.3. Calcite

Thermally stimulated calcite has an emission maximum at 570 nm [60]. However, efforts to use the mineral in luminescence dating have been encumbered by the limited environmental occurrence of calcite. Calcite also tends to concentrate uranium in its lattice and this complicates dose rate calculations since isotopic disequilibrium of uranium has to be taken into account. Worth noting is that uranium disequilibrium dating can yield ages from calcite that are more reliable than those obtained using luminescence techniques. As a result, the incentive to employ luminescence methods in dating calcite has been small. It should be mentioned that some of the earliest, albeit unsuccessful, TL studies that tried to date rocks employed calcite [10]. Other attempts to use calcite in archaeological dating include a report by Ugumori and Ikeya [76].

4.4. Zircon

Zircon is an attractive dosimeter because it usually has a relatively high concentration of uranium. Consequently, the dose from the mineral grain's interior is far greater than that originating from outside. This yields a dose rate that is relatively constant since it is not susceptible to variations arising from external effects such as changes in water content or burial depth. An associated drawback, however, is that the uranium content of zircon varies between individual grains. Consequently, measurements for dose rate are made on single grains. Also notably, zircon crystal lattices often have natural inhomogeneities that make it difficult to make comparisons between artificial irradiation administered in the laboratory with natural doses originating from within the grain. As outlined in Section 5, such comparisons are the standard approach for determining the paleodose when dating quartz or feldspar. To address that problem, zircon dating uses a technique called autoregeneration. With autoregeneration, after the natural signal from the zircon grains is measured, the grains are stored for a few months to allow a new dose to accrue. Measuring the signal at the end of the storage period and comparing it to the natural signal obtained from the initial measurement allows a calibration to be made that yields an age of the natural signal.

Analysis of zircon using TL includes a study by Huntley et al. [67] and by Templer and Smith [77]. OSL studies using zircon include investigations by Smith [78].

5. Paleodose and dose rate determination

The age equation introduced in Section 1 (Eq. 1) shows that two parameters need to be determined before a luminescence age can be calculated: the paleodose and the dose rate. This section examines methods that are used to determine the two variables.

5.1. Paleodose determination

The start of the accumulation of the paleodose should typically coincide with a geomorphic or archeological event that emptied (or zeroed) any previously accumulated energy in the sample grains. The dose refers to the energy absorbed per kilogram of material, and it is

measured using units of gray (Gy), where 1 Gy = 1 J/kg. For materials that were previously zeroed by heating or firing, the start of the accumulation of the paleodose would correspond with the last time the material was heated to a temperature high enough to expel electrons from their traps. In the case of sediments that were zeroed by exposure to sunlight, the start of the accumulation of the paleodose would correspond to the last time that a sample was subjected to the bleaching effects of the sun for a period long enough to evict all trapped electrons. As indicated earlier, the natural signal refers to the luminescence signal yielded by a sample collected from the field. In order to determine the paleodose of a sample of unknown age, the natural signal is measured first after which the sample is irradiated artificially using a well-calibrated laboratory-based source. The signals from the artificial dose are then measured and compared with the natural dose signals in order to determine an artificial dose that gives a signal similar to that of the natural dose. This is referred to as the equivalent dose (D_e). Investigators use two main methods to determine D_e: the additive dose and the regenerative dose (or regeneration) methods [2, 50].

5.1.1. Additive dose method

When determining luminescence ages using the additive dose method, a sample of unknown age is split into two sets of aliquots. The natural signal for one set is measured first after which the second set is irradiated with incremental doses using an artificial source and also measured. Plotting the artificial signals against the dose administered produces a dose–response curve depicting the luminescence signal against the laboratory dose (**Figure 8a**). The curve is also known as a growth curve. The natural signal is also plotted on the growth curve against zero dose (**Figure 8a**). Extrapolating the curve backwards until it intercepts the horizontal axis at zero signal intensity provides D_e (**Figure 8a**) [51]. Growth curves are unique to each sample because luminescence sensitivity of mineral grains can vary from sample to sample. As a result, a new growth curve has to be constructed for each sample whose age is being determined. When dating materials that were zeroed by the sun using TL, the residual TL signal that is noted following solar bleaching would have to be taken into account when extrapolating the curve backwards. With both feldspar and quartz, the relationship between the luminescence signal and the laboratory radiation portrays a linear trend for low and moderate doses. At elevated doses, however, the growth curve plateaus, indicating that luminescence traps are getting exhausted, also referred to as saturation.

5.1.2. Regenerative dose method

The procedure used in the regeneration method is similar to that employed in the additive dose method apart from that, before the laboratory dose is applied, the sample aliquots in the regeneration method are first zeroed to remove any previously acquired dose. Incremental doses are then applied to the zeroed aliquots and measured. The acquired signals are plotted against the administered dose to give a regenerative dose growth curve. To get the equivalent dose, the signal from the sample of unknown age is interpolated into the growth curve (**Figure 8b**) [51]. Hence, when constructing the growth curve, the laboratory irradiation doses are selected such that the signals they produce lie above and below the signal obtained from the natural dose.

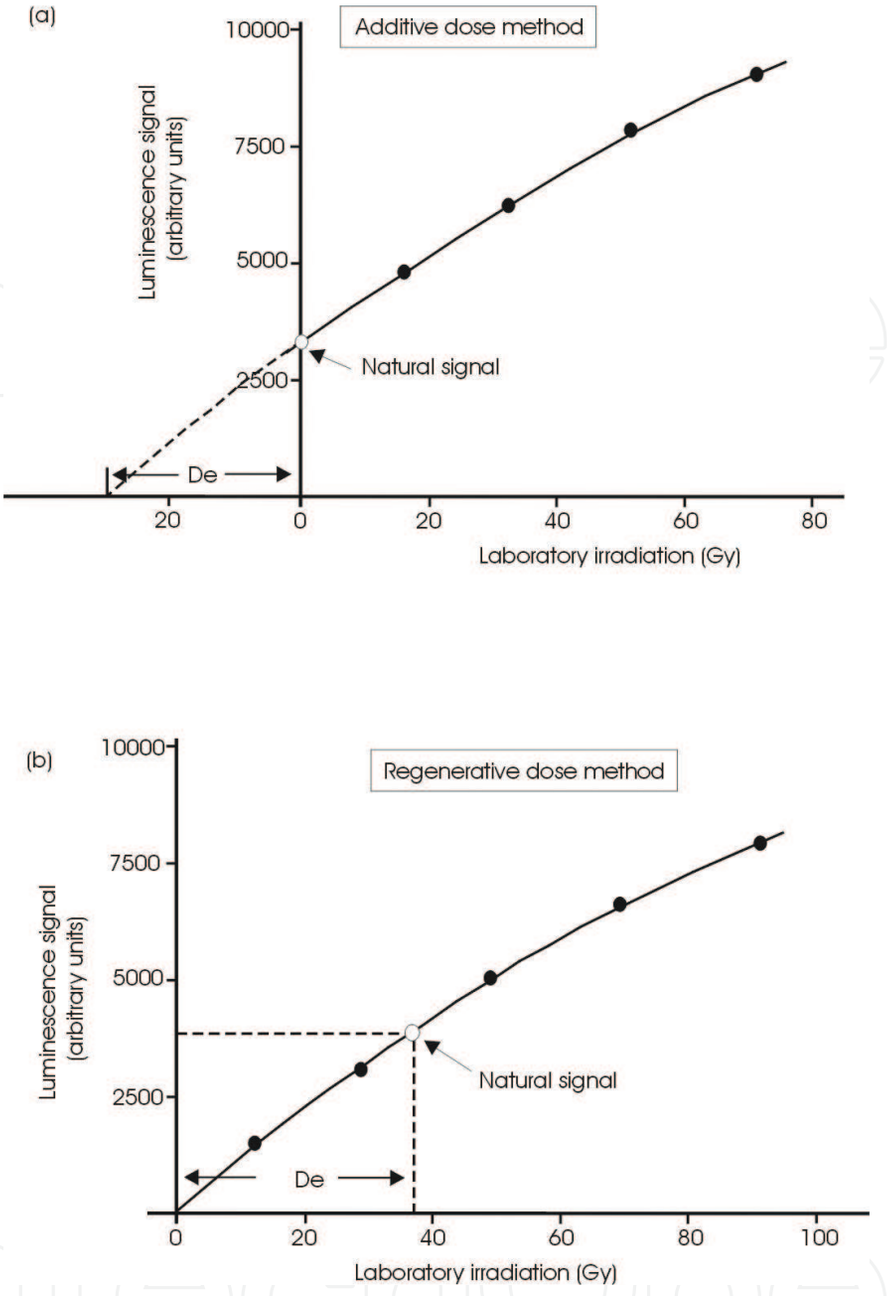


Figure 8. Main methods employed in determining the equivalent dose. (a) When using the additive dose method, incremental doses are administered on top of the natural dose and then measured. (b) With the regenerative dose approach, samples are first zeroed to remove any previously acquired dose and then given incremental doses, followed by measuring. Growth curves are unique to each sample being analyzed such that new measurements have to be made and a new curve constructed for every sample being dated.

5.1.3. Single aliquot methods versus multiple aliquot methods

Both the additive dose and regenerative dose methods employed multiple aliquots when they were originally developed for TL dating. Later, when OSL dating emerged, the possibility of using single aliquots only was brought up [1]. However, the concept did not take hold initially and multiple aliquots were also adopted for OSL dating. Generally, the use of multiple aliquots assumes that all aliquots of a given sample behave similarly to the dose administered.

However, in reality, inter-aliquot variations occur for a range of reasons that include changes in sensitivity [3, 79]. Normalization is used in some cases to try and reduce the effects of the variations. Nonetheless, the effects cannot be eliminated entirely such that uncertainties are contributed to the calculated ages. Ultimately, there was an incentive to devise an approach that only employed a single aliquot.

5.1.4. Single-aliquot regenerative-dose (SAR) protocol

When initially introduced for dating, single aliquot methods employed the additive dose method on feldspars [80, 81]. Quartz had not been used because sensitivity changes that it displays during repeat measurement cycles rendered single aliquot data unworkable. A few years later, however, following work carried out by Mejdhal and Bøtter-Jensen [82], a single aliquot regenerative (SAR) protocol for quartz that incorporated correction steps to address the changes in sensitivity was proposed. Subsequently, Murray and Wintle [37] presented an enhanced version of the SAR approach introduced by Murray and Roberts [83] in which a test dose was used to monitor sensitivity changes in the quartz. As initially proposed by Murray and Wintle [37], their SAR method entailed steps outlined in **Table 2**.

Minor modifications have been made to the SAR procedure presented in **Table 2** since its original inception [37, 38]. However, over the last two decades, the protocol has been widely adopted for routine dating of both sediments and heated materials using quartz and feldspar [84].

Step	Treatment[a]	Observed signal
1	Irradiate sample with artificial dose, D_i	–
2	Sample preheated (160–300°C) for 10s	–
3	Sample stimulated for 100 s at 125°C	L_i
4	Sample irradiated with test dose, D_t	–
5	Sample heated to 160°C	–
6	Sample stimulated for 100 s at 125°C[b]	T_i
7	Return to step 1 and repeat sequence[c]	–

D_i is the dose that yields the signal L_i while D_t is the test dose that produces the signal T_i. These values are used to plot a curve of Li/Ti vs. the regeneration dose D.
[a]For the natural sample, $i = 0$ and $D_0 = 0$ Gy.
[b]Stimulation time varies with the intensity of the light used for stimulation.
[c]Subsequent modifications to steps above include an additional step to help attenuate recuperation.

Table 2. Sequence of steps in the SAR procedure as initially proposed by Murray and Wintle [37].

5.1.5. Single grain analysis

SAR protocols have also been extended to determining paleodoses using individual mineral grains. The transition has been facilitated by the development of special equipment for loading and analyzing thousands of grains of sand size (<250 μm) relatively quickly [85]. The analysis of individual grains from the same sample is particularly useful for identifying differences in paleodose between grains [86]. Examples of cases where this may be expedient is

when studying sediments deposited by rivers (fluvial) where well-bleached grains might be mixed with partially bleached fractions [87].

5.2. Determination of the dose rate

As outlined above, once the paleodose has been ascertained, the dose rate needs to be evaluated before an age can be determined. In Section 1, it was mentioned that ionizing radiation responsible for the energy accumulation in mineral grains in natural settings emanates from uranium and thorium decay chains as well as from potassium and rubidium isotopes. Cosmic radiation also contributes a minor component. A number of methods can be used to evaluate the total contributions of all these components. Using the concentration approach, levels of uranium, potassium and rubidium in a given sample are quantified with the help of an analytical procedure such as atomic absorption spectroscopy (AAS), neutron activation analysis (NAA), flame photometric detection (FPD), X-ray fluorescence (XRF) and inductively coupled plasma spectroscopy (ICPS). Once the concentrations have been measured, dose rate is determined using special conversion tables prepared for the purpose [50]. For uranium and thorium, however, isotopic disequilibrium could render the measurements using these analytical techniques unreliable [50].

As opposed to measuring elemental concentrations "using methods outlined" above, direct measurements of the activities of specific radionuclides can be achieved using methods such as alpha or gamma ray spectrometry. Despite being costly, these methods can provide accurate measurements, including from uranium and thorium decay chains and in cases where disequilibrium exists. Nonetheless, lengthy measurement times may be necessary [50].

Alternatively, errors from isotopic disequilibrium in the uranium and thorium decay chains can be reduced by measuring the contributions of uranium and thorium using thick source alpha counting (TSAC) after which potassium is measured using an analytical technique such as AAS, FPM, XRF or ICPMS. Yet another approach to determine the dose rate is to use TSAC to determine the alpha particle contribution after which a beta particle counter is employed to determine the beta contribution. The gamma dose rate is best determined in the field whenever possible. Highly sensitive portable gamma-ray spectrometers that make such onsite measurements possible are now available [44].

Measurement of the dose rate can also be conducted in the field using synthetic dosimeters such as α-Al_2O_3:C [44]. These are highly sensitive materials that are left in the field for a few weeks after which they are retrieved and analyzed.

The dose contribution from cosmic rays is usually minor. However, in settings where the radionuclide concentrations are low, the proportion from cosmic rays becomes significant. A methodology for calculating cosmic ray contribution to the luminescence dose rate was formulated by Prescott and Hutton [53].

Lastly, it is imperative to take the *in situ* moisture content of the material that is being dated into account when calculating the dose rate. This is because interstitial water absorbs part of the dose that should otherwise reach the dosimeter, with the attenuation of the dose rate intensifying as the moisture content increases.

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5.3. Lower and upper age limits in luminescence dating

At present, luminescence dating methods can be used to date samples that are as young as a few decades [88]. Dating using the single grain approach can produce young ages that are relatively precise. When dating such young samples, it is desirable to use mineral grains characterized by a high luminescence sensitivity and for the grains to have been completely bleached prior to the burial [39]. Minimizing thermal charge transfer during measurement also improves the accuracy of the results.

Maximum ages that can be obtained using luminescence dating methods are ultimately controlled by the fact that the population of electron traps within any given dosimeter is fixed. As

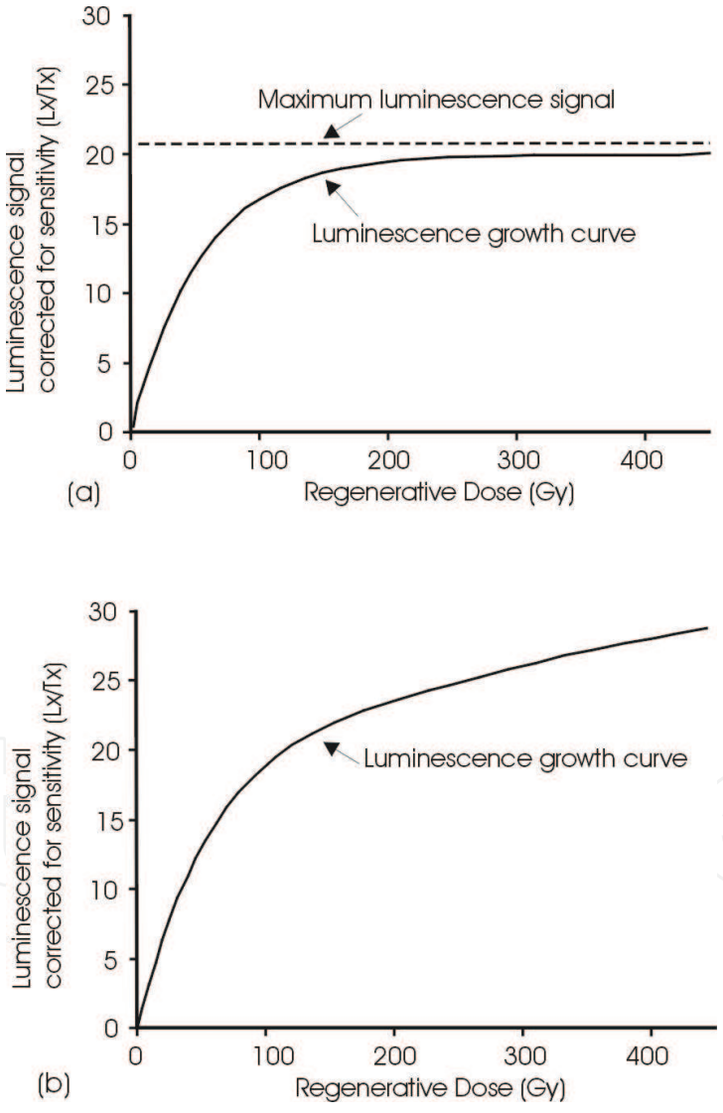


Figure 9. Examples of growth curves for quartz taken to saturation (redrawn after [3]). (a) Curve constructed using a saturating exponential function of the form $I(D) = I_0 (I - exp^{-D/D_0})$ where $I(D)$ is the luminescence signal produced by the dose D and I_0 is the maximum luminescence intensity produced. The parameter D_0 determines the shape of the curve. (b) Since curve (a) appeared to underestimate some older ages, the saturating function from (a) is combined with a linear function in (b) [3].

a result, the number electrons stored by trapping cannot increase indefinitely [50, 51, 56]. This is depicted in luminescence growth curves by a flattening of the signal obtained as the dose increases and is often expressed using a saturating exponential function. **Figure 9a** shows such a function expressing the fast component of a quartz signal. It shows that, once a certain dose is reached, the curve flattens. That dose is the upper limit above which the proportionality between the dose received and luminescence signal obtained breaks down. Quartz usually saturates with a dose of around 100–150 Gy. An approach used in some studies [4, 89] working with high doses is to model the growth curve by combining a linear function and a saturating function as shown in **Figure 9b** [3]. This approach has been used to report quartz ages in excess of 200 000–400 000 years. It should be noted that the limits of the ages that can be obtained are ultimately determined by the magnitude of the dose rate, with low dose rates giving higher age limits and vice versa. With feldspar, several studies have reported ages that exceed 100 000 years using IRSL [90]. Correction for anomalous fading of the feldspar at such high doses (>100–200 Gy) using standard methods [66], however, becomes problematic.

In essence, with both feldspar and quartz dating, there are maximum dose limits above which reliable ages cannot be produced as a result of electron trap exhaustion.

6. Sample collection and preparation for analysis

Preceding sections explored the basics of luminescence dating. A subject that now needs to be addressed is the nature of materials on which luminescence dating methods can be applied. Before that can be looked into, however, it is pertinent to examine the topic of sample grain size, since protocols employed in luminescence analysis are contingent upon the granulometry of the material being analyzed.

6.1. Sample grain size

There are generally two broad mineral grain size ranges that are employed in luminescence dating: coarse grains and fine grains. When using the coarse grain method, sand-sized particles in the diameter range 90–250 μm are extracted and analyzed. Grains in this size range normally receive ionizing radiation from alpha and beta particles as well as gamma and cosmic rays. Because of their size, alpha particles would only be able to penetrate the mineral grain's exterior 25 μm. When working with coarse grains, the outer rim that is affected by the alpha particles is removed by etching using hydrofluoric acid (HF). For this reason, coarse grain luminescence dating is sometimes described as inclusion dating [58, 91, 92]. When dating pottery using quartz inclusions, grains in the size range 90–125 μm are commonly selected. In sediment dating, the usual practice is to extract grain sizes that represent the modal size. Thus, for eolian dunes, grains in the size range 150–180 μm are often used [3]. Feldspar inclusion dating employs procedures similar to those used for quartz dating, including etching to remove the outer rim penetrated by alpha particles.

An important aspect of inclusion dating is that, since parts of the grain affected by alpha particles are removed prior to the performing luminescence measurements, only the contributions of

beta, gamma and cosmic rays are taken into account when determining the dose rate. If Eq. (**1**) is modified to take those analytical considerations into account, the age equation becomes:

$$Luminescence\ Age = \frac{Paleodose}{0.90D_{\beta} + D_{\gamma} + D_{C}} \tag{6}$$

where D_{β}, D_{γ} and D_{c} are the doses from beta, gamma and cosmic ray contributions, respectively [50, 51]. If the dose rates are given as annual values, the equation yields the age in calendar years. The beta dose rate is factored by 0.9 as a correction for the grain size and the etching that disposes of the outer rim which also received a beta dose [50, 51].

Worth noting is that, on rare occasions, quartz may contain some uranium and thorium within its lattice. In such cases, Eq. (**6**) may need to be modified if coarse grains are used. Similarly, when dating feldspar using coarse grains, potassium-40 isotopes in K-feldspar constitute an additional source of beta particles that has to be factored into the dose rate calculation. As a result, feldspars are often separated into K-feldspar and low-potassium fractions that are then analyzed separately.

For fine grains, mineral grains in the size range 4–11 μm are usually extracted and mounted for analysis. When calculating the dose rate for such mineral grains, since their diameters are such that alpha particles can penetrate them entirely, contribution from alpha particles has to be included in the dose rate. Consequently, the age equation becomes:

$$Luminescence\ Age = \frac{Paleodose}{\mathrm{k}D_{\alpha} + D_{\beta} + D_{\gamma} + D_{C}} \tag{7}$$

where D_{α}, D_{β}, D_{γ} and D_{c} are the doses from alpha, beta, gamma and cosmic ray contributions, respectively.

Mineral grains in the diameter range 33–63 μm have been dated in other studies [93]. Because the size range is intermediate between coarse grain and fine grain categories, the analysis is sometimes referred to as middle-grain dating [50]. Etching with HF to remove the outer rim of the grains is not usually employed with middle-grain dating. Thus, the alpha particle contribution is also included when calculating the dose rate.

6.2. Sample collection and preparation

6.2.1. Sample collection

As outlined earlier, exposure of luminescence dating dosimeters to sunlight results in the zeroing of any previously acquired dose. Thus, a basic constraint when collecting samples is that they should be shielded from light until the time of analysis. Accordingly, when dealing with archaeological artifacts, sampling usually entails using a power drill in safe-light conditions to retrieve a representative sample from the interior of the artifact. When collecting sediments from their natural settings, on the other hand, sampling could be conducted at night [50]. However, working in the dark is inconvenient and chances of accidentally exposing the sample to light are also greater. Hence, night-time sampling is not the most popular

approach. More often than not, sampling is conducted during the day. In cases where sediments have a firm consistency, it may be possible to cut out a block of sediment and transport it to the lab where the sample for dating is extracted from the block's interior. A more commonly used approach is to insert an opaque cylindrical pipe into a targeted geological unit. Both ends of the pipe are capped once it has been retrieved. At the laboratory a sample is analysis is removed from the center of the pipe. Samples for luminescence analysis can also be collected at depth by drilling vertically using sampling modules specially designed to avoid exposure of the sample to sunlight [94].

6.2.2. Sample preparation prior to the measurement

Collected samples have to be pretreated before luminescence analysis can be conducted. There are a number of standard procedures that have been developed for pretreating samples, depending on whether the grains targeted for analysis are fine or coarse. Analysis of coarse grains is usually performed on pure separates of either quartz or feldspar. Fine grains, however, are often analyzed as polymineralic fractions. The choice of what mineral or grain size to analyze will generally depend on the sample type. For instance, when dating loess, which is a silt-sized deposit, it is more appropriate to extract fine grains whereas using coarse grains would be more suitable for dating wind deposited dune sands. Where sediments have equal proportions of fine and coarse components, it is possible to validate results by dating both fractions.

In coarse grain dating, sample pretreatment entails first removing carbonates and organic material using dilute hydrochloric acid (HCl) and hydrogen peroxide (H_2O_2), respectively. Pure separates of quartz or feldspar are then extracted from the sample using a heavy liquid such as sodium polytungstate solution. Finally, the separates are mounted on discs (about 10 mm in diameter) before being analyzed. More details on sample extraction are given in [95].

When extracting fine grains for analysis, carbonates and organic materials are also first eliminated using dilute HCl and H_2O_2, respectively. To avoid flocculation of the grains, dilute sodium oxalate is added. After thorough mixing, a sedimentation column is then used to extract grains of the desired size range (usually 4–11 μm). If no further separation is required, the sample will be ready for mounting on discs. Occasionally, however, some studies do extract pure quartz from fine-grained samples. This is achieved by digesting feldspar from the polymineralic sample using fluorosilisic acid [96]. Additional details on separating and mounting fine grains can be found in [97].

7. On what materials can luminescence dating be applied?

Materials of geological or archaeological origin that are dated using luminescence methods have to satisfy a number of characteristics before they can be successfully analyzed. First, the material must be composed of a substance that behaves as a dosimeter. Second, the luminescence energy contained in the material must have been zeroed at some point that now serves as the starting point for counting time. Third, energy storage in the materials should not have reached saturation at the time of analysis, otherwise stored energy would cease to be

an accurate measure of time that elapsed. Within the context of these requirements, materials from which ages can be obtained using luminescence methods can be classified into two main categories: substances whose accrued energy has been reset by heating and sediment grains of sand and silt size whose previously acquired energy has been zeroed by exposure to sunlight while being transported.

7.1. Heated materials

The heating process employed in pottery production commonly fires earthenware to temperatures in excess of 800°C. Such elevated heat effectively evicts trapped electrons in constituent materials, providing a starting event for a luminescence age. As a result, archeological materials such as ancient pottery, bricks, terracotta figures and tiles are artifacts whose ages can be ascertained using luminescence methods, especially TL. This is because the artifacts commonly contain mineral grains such as quartz and feldspar. If the quartz or feldspar grains from the artifacts are extracted and dated using the inclusion dating approach (coarse grain technique), ages can be obtained. Alternatively, the fine-grained components can be extracted from the same materials and polymineralic fractions dated. Fine-grained quartz separates can also be isolated and dated similarly. It is thought that humans began making pottery about 30,000 years ago, during the Neolithic period [98]. By 10,000 years ago, the skill had spread globally [51]. Thus, most ancient pottery artifacts should be amenable to dating using luminescence methods.

In addition to pottery, other materials of archaeological context that could experience episodes of heating include flint and any other stones that may have been heated to high temperatures by fires associated with human settlements. These include residual rock chips from the production of tools by ancient humans that may have been heated either intentionally or otherwise. Dating any of these artifacts using luminescence techniques provides a timeline that approximates the age of the occupation of the archaeological site by humans [99].

Geological materials that have undergone heating to high temperatures at any point during the last about 100,000 years should also be datable using TL methods if they contain appropriate dosimeters. Materials that could fall into this category include sediments that border volcanic eruptions, as well as products of the volcanic eruptions themselves including lava and ash. In all instances, dating the materials using luminescence methods allows the age of the eruption to be approximated [100].

A final category of substances that can be dated using luminescence methods after being zeroed by elevated temperatures are materials associated with meteorite impacts. Such impacts create thermal shocks that erase any previously acquired dose in mineral grains contained in proximal geological structures, providing a convenient bleaching mechanism for dating the impact using luminescence methods [101].

7.2. Dating of sediments reset by sunlight

Granular sediments that contain dosimeters that have been bleached of all previously acquired energy by exposure to sunlight before being buried form an important class of materials that

can be dated using luminescence methods. Stemming from studies [36] that showed that the exposure of some minerals to sunlight could evict electrons from traps in mineral lattices, luminescence methods have been successfully used to date sediment from a range of environments. While TL was used in the beginning, the subsequent development of OSL techniques was nurtured by the dating of this category of materials. The types of sediments that fall into this category include sediments transported by wind (eolian sediments), water-lain sediments, sediments associated with glaciers, as well as sediment related to earthquakes. The section below examines each of these sediment classes. In each case, luminescence ages denote time that has passed since the sediments were last zeroed by sunlight. Similarly, the ages correspond to time that has passed since the sediment was last shielded from sunlight (or buried) by overlying geological units.

7.2.1. Eolian deposits

Of all classes of geological materials that can be dated using luminescence methods, wind deposited (eolian) sediments are the most ideal. This is because the sediment grains are transported at the earth's surface or in mid-air, affording them adequate exposure to the sun to cause complete zeroing of any dose the sediment may have [50]. Consequently, the largest share of luminescence ages that have been published to date are from deposits of eolian origin. Eolian deposits generally fall into two main categories: sand-sized and silt-sized grains. Sands comprise sediment grains in the range 63–250 μm. The wind transports such particles via a process called saltation [102], which is a sequence of low altitude jumps, less than a foot from the surface of the depositional bed. Such grains usually form dunes. Silt-sized grains comprise particles in the size range 2–63 μm. These are transported in suspension and typically stay airborne for long periods of time [102]. In either case, eolian sands and silt grains are usually well-zeroed by the time they are buried by other sediments.

Eolian dune sediments have been dated in multiple studies [103] using quartz or feldspar extracts (mostly employing blue OSL and IRSL for stimulation, respectively) and produced ages that have been compared with radiocarbon ages from related sediments. In all cases, the results have validated luminescence dating as a competent dating method that provides reliable ages. Since eolian dune deposits are often treated as indicators of dry conditions, chronologies from eolian sands have been used to reconstruct environmental changes from the past [104–107]. Published ages span the last ca. 100,000 years. Hence, the method has emerged as an indispensable tool for the study of Late Pleistocene and Holocene paleoenvironments.

In addition to studying environmental change in inland deserts, luminescence chronologies from coarse-grained eolian sands have also been used to investigate depositional chronologies of coastal dune deposits [108]. Results from such studies have been used to develop temporal frameworks for processes in coastal environments, including sea level change.

Silt-sized eolian sediments can form extensive sequences of deposits referred to as loess [102]. Fine grain dating has been used to provide depositional chronologies of loess in numerous studies [107]. As in coarse grain dating, the chronologies have invariably been used to reconstruct paleoenvironments from the late Quaternary period. Luminescence analysis of fine grains

can be conducted by targeting feldspar in polymineral fractions using IRSL. Alternatively, if quartz fines are extracted as outlined above, blue OSL is employed for stimulation [109].

7.2.2. Water-lain deposits

Coarse as well as fine-grained sediments that have been deposited by water have also been investigated using luminescence methods. However, studies have shown that sediments transported by rivers (fluvial deposits) are not always well-zeroed at deposition [46, 110]. As a result, single grain dating methods have been used to identify well-zeroed grains from such settings [46, 110]. Useful information on modern and ancient depositional rates of river systems have been acquired through luminescence studies of fluvial sediments [110]. Influences of tectonic and climatic effects on river systems have also been investigated using luminescence chronologies [110]. Other applications of luminescence ages from fluvial deposits include paleoseismic and archaeological studies. Additional information on dating fluvial deposits using luminescence methods can be found in [46, 110].

7.2.3. Sediments of glacial origin

As with water-lain deposits in fluvial settings, the greatest concern with deposits from glacial environments is the degree of bleaching that sediments experience prior to the burial. Sediments with the highest probability of being well-zeroed are deposits that meltwaters transport away from the glacier as outwash [44, 111]. Hence, chances for complete zeroing increases with distance from the glacier's front [111–113]. Nonetheless, even with outwash deposits, studies have shown that sediments are often partially zeroed [112]. As with fluvial deposits, single grain dating methods can be used to identify well-bleached grains in such instances. Some investigators have tried to identify signal components that bleach more rapidly for use when dating partially bleached glacial deposits [111]. More detailed discussions on luminescence dating of glaciofluvial deposits are found in [44, 111].

7.2.4. Earthquake-related studies

Tsunami episodes generated by earthquakes often displace sandy deposits from beach areas, depositing them further inland in tidal marshes, bogs and lakes that rim ocean margins. Afterwards, as part of the coastal sequence, other deposits that can include peat or mud may be emplaced atop the displaced sands, shielding them from sunlight. If the sands are dated using luminescence methods, the chronology they yield can be used to approximate recurrence rates of the tsunamis and, by extrapolation, of the earthquakes that cause the tsunamis. Such information would be critical when evaluating environmental hazards within the region [50]. Worth noting, however, is that tsunami events are rapid and often do not provide time for adequate bleaching of the sediments they mobilize. As a result, investigators target sediments that were exposed at the surface before the tsunami event and hence were already zeroed [50, 114]. Other deposits associated with earthquakes on which luminescence dating has been applied are sediments that occur on horizontal surfaces that have experienced differential vertical movement through earthquake-related faulting [49]. Sediments on the uplifted surfaces could be correlated by dating them using luminescence methods.

8. Current and future trends in luminescence dating

Luminescence dating has evolved into a full-fledged and robust chronometer over the last six decades. Concurrent with and driving this development has been the establishment of numerous luminescence dating laboratories throughout the world. To foster interaction between scholars, the Luminescence and Electron Spin Resonance Dating (LED) Conference is held triennially to examine a range of topics that encompass luminescence dating. In the years between the LED conferences, regional and national meetings are also held, including the UK Luminescence Dating Conference, the German Luminescence and ESR Conference or the North American New World Luminescence Dating Workshop. Topics explored at the meetings include fundamental research into luminescence dating as well as applications of luminescence chronologies in environmental, geological and archaeological sciences. Improvements in instrumentation for luminescence dating also constitute an important element at the meetings.

Current topics in fundamental research include investigations that aim to improve knowledge on luminescence characteristics of dosimeters such as feldspar and quartz. Ultimately, this should lead to the production of ages that are more accurate as well as the extension of maximum and minimum age limits that can be obtained using luminescence methods. Such efforts include studies that aim to understand the behavior of quartz at high doses [115, 116]. The information should lead to the formulation of protocols that extend the upper limit of ages obtainable using quartz [116].

Research on feldspar has included efforts to identify IRSL signals that are less inclined to experience anomalous fading [66]. Employing such signals would allow older ages to be determined using feldspar than are currently obtainable [117, 118]. Some studies have demonstrated that IRSL signals that are acquired by stimulating feldspar at a low temperature (for instance, 50˚C) after which another measurement is immediately conducted at a higher temperature (for instance, 290˚C), have a lower fading rate than those obtained using standard protocols [118]. This experimental procedure, termed post-IR IRSL [118] has yielded encouraging results that could potentially raise the upper limit of ages that can be obtained using current methods [118].

Improvements in instrumentation are also continuously being realized and advances include the development of luminescence readers equipped with broader spectral ranges for emission detection [54]. The recent development of portable OSL measurement systems that can be used to conduct rapid on-site measurements is also worth mentioning [119]. Compared to regular OSL readers, the portable systems have limited functions. However, if they were to be equipped with add-ons such as external X-ray sources for sample irradiation, practitioners would be faced with expanded options for signal collection.

Today, luminescence dating is a dynamic field of research that is set to continue witnessing ground-breaking developments in years to come. As both laboratory procedures and analytical equipment are improved, ages should be reported with higher precision and accuracy.

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