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Luminescence dating: basics, methods and applications

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Abstract: Luminescence dating is a tool frequently used for age determination of Quaternary materials such as archaeological artefacts, volcanic deposits and a variety of sediments from different environmental settings. The present paper gives an overview of the physical basics of luminescence dating, the necessary procedures from sampling to age calculation, potential problems that may interfere with correct age calculation as well as procedures to identify and resolve those problems. Finally, a brief summary of the most common fields of application is given ranging from artefacts to the variety of different sediments suitable for luminescence dating.

[Lumineszenzdatierung: Grundlagen, Methoden und Anwendungen]

Kurzfassung: Lumineszenzdatierung ist eine häufig angewendete Methode zur Altersbestimmung quartärer Materialien, wie z.B. archäologischer Artefakte, vulkanischer Ablagerungen oder von Sedimenten unterschiedlicher Ablagerungsräumen. Das vorliegende Manuskript gibt einen Überblick über die physikalischen Grundlagen der Lumineszenzdatierung, erläutert die notwendigen Prozeduren von der Probenahme bis hin zur Altersberechnung, diskutiert potenzielle Probleme die eine korrekte Altersberechnung beeinträchtigen können und stellt Verfahren vor, mit denen diese Probleme erkannt und beseitigt werden können. Abschließend wird ein kurzer Überblick über die gängigsten Anwendungsgebiete gegeben, von Artefakten bis hin zu verschiedenen Sedimenten, die für Lumineszenzdatierung geeignet sind.

Keywords: Luminescence, physical dating methods, archaeology, geosciences, Quaternary

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1 Introduction

Luminescence techniques enable evaluation of the time that has elapsed since mineral grains crystallised, were last exposed to daylight or heated to a few hundred degrees Celsius. The method uses an optically and thermally sensitive light or *luminescence* signal in minerals such as quartz and feldspar. During exposure to light or heat the luminescence signal within the grains is erased (optically bleached or thermally annealed) until it is completely removed (zeroed) (Fig. 1). Once the grains are sealed from daylight and remain at normal environmental temperatures, the luminescence signal accumulates again, being induced by naturally occurring radioactivity. For dating, the amount of absorbed energy per mass of mineral ($1 \text{ J kg}^{-1} = 1 \text{ Gy}$ (Gray)) due to natural radiation exposure since zeroing - known as the palaeodose - is determined by comparing the natural luminescence signal of a sample with that induced by artificial irradiation. Several laboratory techniques have been developed to accomplish the necessary stimulation and recording of the weak but measurable luminescence emitted from minerals. The time elapsed since the last daylight exposure or heating is calculated by dividing the palaeodose by the dose rate, the latter representing the amount of energy deposited per mass of mineral due to radiation exposure acting on the sample over a certain time (Gy a^{-1}). This relation is represented by the following simple equation:

$$\text{Luminescence age (a)} = \frac{\text{Palaeodose (Gy)}}{\text{Dose rate (Gy a}^{-1}\text{)}} \quad (1)$$

Luminescence dating has been applied to a wide range of topics within Quaternary research such as landscape evolution, palaeoclimate, geohazards and (geo)-archaeology, and has undergone several important methodological refinements since its early days. One of the first publications suggesting the use of thermoluminescence (TL) as a research tool was by DANIELS et al. (1953) and a few years later TL was used to date ceramics (GRÖGLER et al. 1958; AITKEN et

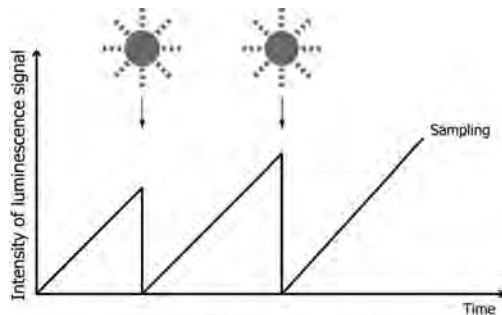


Fig. 1: The basic principle of luminescence dating: zeroing of the signal during daylight exposure (or by heating) and subsequent accumulation of the signal with time, when the material is sealed from daylight and not exposed to heat.

Abb. 1: Die Grundlage der Lumineszenzdatierung: Nullstellung des Signals während Tageslichtexposition (oder durch Erhitzen) und anschließende Akkumulierung des Signals während der Zeit, in der das Material vor Sonnenlicht geschützt bzw. keiner Erhitzung ausgesetzt ist.

al. 1964, 1968). An important benchmark was the application of TL to the dating sediments (WINTLE & HUNTLEY 1979, 1980; WINTLE 1980). Another breakthrough came with the introduction of optical stimulation by both visible (HUNTLEY et al. 1985) and infrared light (HÜTT et al. 1988). More recent advances concern the development of measurement procedures. Of these, notable developments are the use of a single aliquot (DULLER 1991; MURRAY & WINTLE 2000), as well as single grain dating techniques (MURRAY & ROBERTS 1997; DULLER et al. 2000), new analytical tools such as linearly modulated luminescence (BULUR 1996), radiofluorescence (KRBETSCHKE et al. 2000; ERFURT et al. 2003) and spatially-resolved luminescence (GREILICH et al. 2002; GREILICH & WAGNER 2006).

The aim of the present paper is to give an overview of the method as a whole with particular emphasis towards non-luminescence specialist researchers interested in the application of luminescence dating in Quaternary research. It is neither intended to give a full literature review nor to explain every technical aspect in too much detail but rather to give the reader

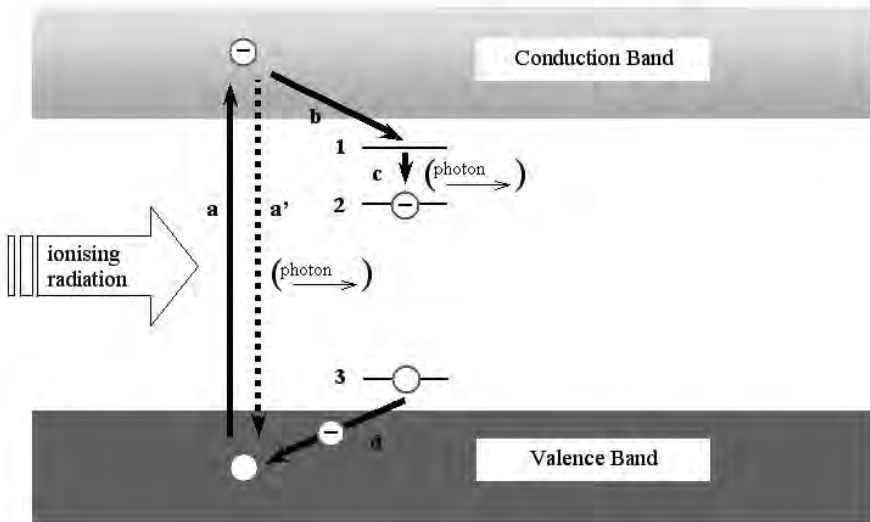


Fig.2.1: Basic processes leading to a latent luminescence signal (schematic):

- a – A valence electron is excited by ionising radiation, and has sufficient energy to reach the conduction band, leaving a hole in the valence band.
- a' – Most of the excited electrons dissipate their energy by recombining immediately with a hole in the valence band; the transition is sometimes accompanied by photon emission.
- b, c – Prompt transition of a few excited electrons into localised energy states below the band edge resulting in electron trapping; light is potentially emitted during these processes..
- d – A hole in the valence band may be filled by electrons from localised levels above the valence band edge; hence the hole transfers from the valence band to the localised level.

Abb. 2.1: Grundlegende Prozesse, die zu einem latenten Lumineszenzsignal führen (schematisch):

- a – Ein Elektron aus dem Valenzband wird durch ionisierende Strahlung angeregt, es besitzt ausreichend Energie um das Leitungsband zu erreichen und hinterlässt ein Loch im Valenzband.
- a' – Die Mehrheit der angeregten Elektronen verliert ihre Energie durch unverzügliche Rekombination mit einem Loch im Valenzband; dieser Übergang ist mitunter mit der Emission von Photonen verbunden.
- b, c – Spontane Übergänge einiger angeregter Elektronen in lokalisierte Energiezustände unterhalb der Bandkante („Fallen“) führen zu stabilem Elektroneneinfang; zum Teil wird bei diesen Prozessen Licht emittiert.
- d – Ein Loch im Valenzband kann durch Elektronen aus lokalisierten Niveaus oberhalb der Valenzbandkante aufgefüllt werden; damit geht das Loch vom Valenzband in den lokalisierten Zustand über.

a firm grounding for the potential, limitations and modern approaches of quality control in luminescence dating of Quaternary materials.

2 Physical background

2.1 Origin of the luminescence signal

The process behind the phenomenon of luminescence is best described by the energy-level

representation of insulating solids (Figs. 2.1 and 2.2). The basic mechanism is that ionising radiation causes the excitation of atoms within the crystal lattice, leading to activated electrons at higher energy states. The vast majority of the activated electrons leave the activated states instantaneously, but some charge is captured at electronic levels below the edge of the conduction band (Fig. 2.1 a-c). These levels are called *electron traps*. The charge deficit creates a hole

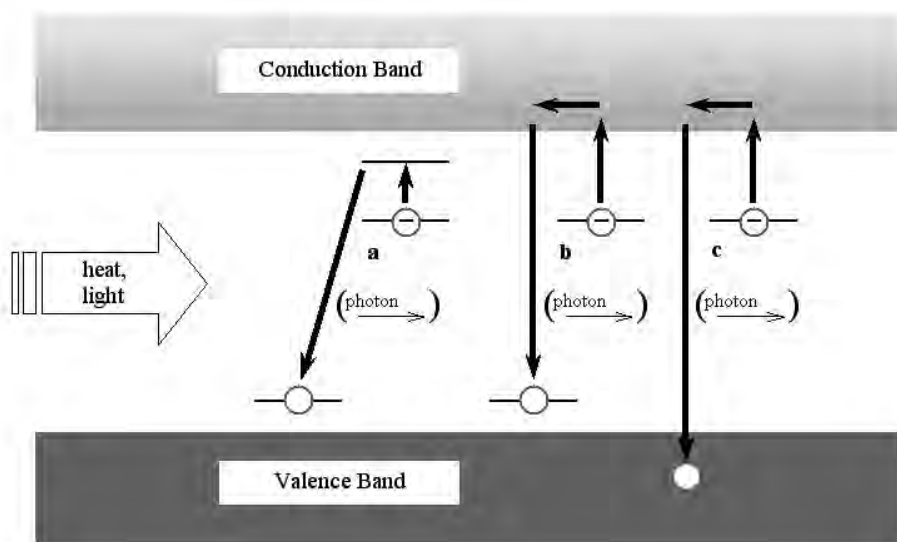


Fig. 2.2: Stimulation of luminescence by heat and light. Possible processes leading to the emptying of trap levels after excitation by light or heat (schematic):

- a, b – Recombination with localised holes above the valence band edge; a) the electron is first transferred to an unstable level below the conduction band; b) the recombines via the conduction band.
- c – Recombination with a hole in the valence band.
The energy loss may be released in the form of light (= luminescence).

Abb. 2.2: Lumineszenzanregung durch Wärme oder Licht. Mögliche Prozesse, die zum Entleeren der Fallen-niveaus nach Anregung durch Licht oder Hitze führen (schematisch):

- a, b – Rekombination mit lokalisierten Löchern oberhalb der Valenzbandkante ; im Fall a wird das Elektron zuerst in einen instabilen Zustand unterhalb des Leitungsbandes transferiert, in Fall b rekombiniert es über das Leitungsband.
- c – Rekombination mit einem Loch im Valenzband.
Der Energieverlust kann als Licht freigesetzt werden (= Lumineszenz).

in the valence band, which may move to form *recombination centres* within the band gap (Fig. 2.1 d). Both electron traps and recombination centres are linked to lattice defects such as oxygen vacancies or foreign atoms (e.g., Al^{3+} instead of Si^{4+} in the quartz lattice). It is important to note that various types of defects exist in the different minerals used in luminescence dating and that the energy representation shown in Fig. 2 very much simplifies the complex situation found in natural minerals. In reality, several kinds of traps and recombination centres exist (cf., CHEN & MCKEEVER 1997; KRBETSCHKE et al. 1997; MCKEEVER & CHEN 1997; MCKEEVER 2001). A basic characterisa-

tion of an electron trap is its energetic depth below the conduction band. The trap depth represents the energy necessary to lift the electron from the trap back to the conduction band. Only electrons captured at traps of a certain depth (in energy terms ~ 1.6 eV or more) remain captured for several million years, which is the prerequisite for dating Quaternary materials since the possibility of charge leakage is less likely for longer storage times (AITKEN 1998). With time, more and more electrons will be captured at the traps within a crystal and so the latent luminescence signal within the mineral will increase. Since the number of traps is limited the latent luminescence cannot increase indefinitely but

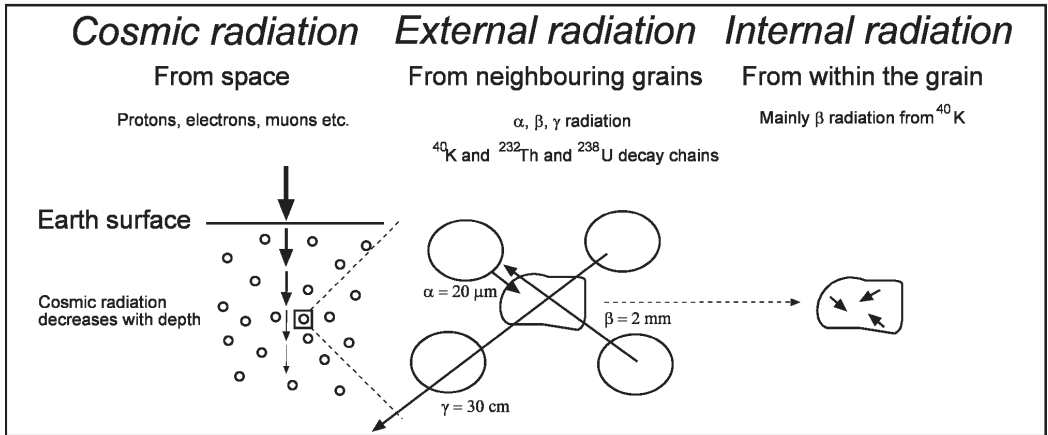


Fig. 3: Different components relevant for the calculation of dose rate for luminescence dating.

Abb. 3: Verschiedene Komponenten, die für die Berechnung der Dosisleistung relevant sind.

will reach a saturation value (saturation dose). Electrons are released from the traps once the mineral is exposed to a particular stimulation energy (Fig. 2.2). In this context it is necessary to consider that the depth of a trap defines the kind of stimulation the captured electron is sensitive to. The deeper the trap, the higher is the required stimulation energy. Once the electron has been lifted to the appropriate recombination level within or below the conduction band, there is a certain probability that it will recombine at an electron hole (recombination centre) and in doing so, release some of the stored energy in the form of light. It is this light released during the recombination process that is referred to as luminescence, and is dependent in various ways on whether TL, optically stimulated luminescence (OSL), etc., is used as the stimulation method. The intensity of light emitted by a sample during stimulation is proportional to the number of recombining electrons and, hence, proportional to the amount of electrons trapped prior to stimulation. The latter is a function of both time and the number of electrons trapped per time interval, which in turn is a function of the dose rate. As a consequence, the intensity of emitted luminescence is linked to the total energy imparted to the mineral due to radioactive exposure since the signal was last zeroed via sufficient stimulation energy.

2.2 Environmental dose rate

Natural ionising radiation occurs in the form of alpha, beta and gamma radiation and cosmic rays (Fig. 3). Alpha radiation consists of Helium nuclei (He^{2+}) that have a penetration depth in sediment of about $20\ \mu\text{m}$ and hence penetrate only the outer part of sand-sized mineral grains. Beta particles (electrons) and gamma rays (photons) have a much higher penetration depth in sediments, of the order of a few mm or dm, respectively, and thus pervade the whole mineral. Because of their small size compared to the penetration depth of alphas, silt and clay-sized particles are completely penetrated by all three types of radiation. In addition to their low penetrating power, a further important factor in the context of dosimetry is that alpha particles induce less luminescence signal, with respect to the absorbed energy, being usually only about 5–20 % compared to beta and gamma radiation (AITKEN 1998).

With regard to dating, the environmental dose rate comprises internal, external and cosmic dose rate components. Internal dose rate originates from radioactive elements that may be present within the lattice of the luminescent mineral. Most important is the contribution from ^{40}K when using alkali feldspars as natural dosimeters. In quartz, the contribution of inter-

nal dose rate to the total dose rate is usually considered to be negligible. External dose rate comprises all radiation acting on the luminescent grain from the surrounding sediment, and is derived from ^{40}K , abundant in some feldspars, micas and clay minerals, and the isotopes of the $^{238}\text{U}/^{235}\text{U}$ and ^{232}Th decay chains. Uranium and Thorium are found at relatively high concentrations in zircon and some exotic minerals, but are ubiquitously present in low concentrations in a variety of common minerals such as carbonates (uranium) and clay minerals. A minor contribution to the total dose rate comes from the presence of ^{87}Rb (typically < 1 %). The contribution from other radioactive elements is so small that it is usually neglected. For calculating external dose rate the moisture content of the sediments is of great importance, as the attenuation of ionising radiation is much greater if the pores in the sediment are filled with water rather than air. Cosmic radiation consists of a variety of different particles such as neutrons, muons, electrons and photons that constantly penetrate the Earth's surface. Due to the nature of the Earth's magnetic field and absorption by the atmosphere, the intensity of cosmic radiation increases pole-wards and with altitude; overburden by both sediment and water (e.g., in lake or marine environments) will shield the sample and reduce the strength of cosmic radiation.

3 Luminescent minerals and their properties

Although a variety of minerals show the phenomenon of luminescence, the application of luminescence for dating purposes is, so far, mainly limited to quartz and feldspar. This is due to their abundance in sediments at most geological settings, as well as their ability to fulfil the requirements of sensitivity to radiation dose and behavioural characteristics. They are also sufficiently resistant to weathering compared to, for example, carbonates, which are also known to carry a luminescence signal (e.g., WIESER et al. 1993; CARMICHAEL et al. 1994). Furthermore, the luminescence signal in both feldspar and quartz is suffi-

ciently bleachable by daylight, is stable over long periods of time, and signal growth can be readily described using mathematical functions for the physical behavior. The dating potential of other minerals such as zircon (TEMPLER & SMITH 1988; SMITH et al. 1991; VAN ES et al. 2000) and halite (BAILEY et al. 2000) was initially tested but little systematic work has been carried out so far to investigate the potential of these minerals in more detail. The following briefly describes the key properties of quartz and feldspar and discusses their advantages and disadvantages for dating.

3.1 Quartz

Quartz is presently the mineral favoured for dating by many luminescence specialists. Beside its common occurrence in sediments, resistance to weathering and relatively well investigated luminescence properties, this is in particular due to the fact that it is not effected by the phenomenon of anomalous fading, as is at least some feldspar (see section 6.3). Due to these advantages, most research during the past decade has concentrated on developing quartz luminescence dating with special attention paid to the development of procedures to determine the palaeodose (e.g., WINTLE & MURRAY 2006). However, there are a few circumstances when quartz may not be the mineral of choice and it appears that many problems related to quartz have only more recently been identified. First of all, there are some geological settings, although relatively rare, where quartz does not occur due to the alkaline composition of the host rock. In other areas, luminescence from feldspar inclusions within the quartz may complicate the detection of the luminescence originating from the quartz itself. Although procedures have been suggested to deal with feldspar inclusions (WALLINGA et al. 2002) these are not straightforward. It has also been shown that quartz of volcanic origin may display anomalous fading (BONDE et al. 2001; TSUKAMOTO et al. 2007) and this problem needs to be addressed in future research. A further problem, possibly related to a young sedimentary history of the grain, is

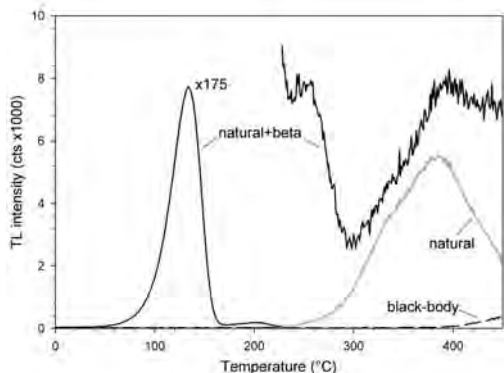


Fig. 4: Natural and artificial TL glow curve and black body radiation of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007).

Abb. 4: Natürliche und künstlich induzierte TL-Glühkurve und Schwarzkörperstrahlung einer Quarz-Probe aus Nigeria (GUMNIOR & PREUSSER 2007).

that quartz from some areas, shows rather low, if any, luminescence (e.g., LUKAS et al. 2007) and changes in sensitivity that are not able to be corrected for (PREUSSER et al. 2006). Thermal transfer of charge from light-insensitive to light-sensitive traps, that may cause palaeodose overestimation, has also been reported in this context (RHODES & BAILEY 1997; RHODES 2000). A considerable disadvantage of quartz is that the saturation dose, when all traps are captured by electrons, and, as a consequence, its potential dating range, is usually up to one order of magnitude lower than that of feldspar.

3.2 Feldspars

Potassium-rich feldspars are commonly used in luminescence dating studies. The disadvantages of using Na-rich feldspars for dating are discussed in KRBETSCHKEK et al. (1997) and only a few studies have focussed on this mineral (e.g., KRAUSE et al. 1997). The major advantages of feldspar are that it typically has bright luminescence signals and a higher saturation dose, which allows the potential to date much older deposits than with quartz. Another advantage is that a substantial proportion of the dose rate comes from ^{40}K within the feldspar,

which lowers the uncertainty from external dose rate, in particular due to variations in past sediment moisture. A disadvantage that has often been attributed to feldspar is that its optical signal is less light sensitive compared to quartz (GODFREY-SMITH et al. 1988; WALLINGA 2002a), although this does not appear to be supported by other experimental evidence which shows a similar resetting behaviour for both minerals (PREUSSER 1999a; KLASSEN et al. 2006).

The major problem in feldspar dating is the loss of part of the signal with time, a phenomenon referred to as anomalous fading (WINTLE 1973), which affects feldspars of at least some geological origins and causes an underestimation of the luminescence age if this is not corrected for. This will be discussed in more detail below (section 6.3).

4 Luminescence methods

Luminescence dating comprises a range of different but related phenomena. The fact that a strict nomenclature is not always adhered to and that some techniques are not often used may cause some confusion to the non-specialist. The following nomenclature refers to the most-widely used terminology and the techniques used in luminescence dating are described (Table 1).

4.1 Thermoluminescence (TL)

The term thermoluminescence (TL) describes the light emitted by a mineral, other than incandescence or black body radiation, when heated. The light originates from captured electrons being freed due to heat stimulation and subsequent recombination. Thus, it is also (and more correctly) referred to as thermally-stimulated luminescence (TSL) although this term is not often used. In addition to sensitivity to heat, a number of the electrons are also sensitive to optical activation, and this has also been utilised in the dating of sediments by TL. In practice, the sample is typically heated from ambient temperature to 450°C while the emitted photons are detected by a photomulti-

Table 1: Summary of the different methods used in luminescence dating.

Tab. 1: Zusammenfassung der verschiedenen Methoden der Lumineszenzdatierung.

Abbrev.	Method	Main application	Primary Reference
TL	Thermoluminescence	Dating heated materials	AITKEN et al. (1964)
ITL	Isothermal TL	Experimental (quartz)	JAIN et al. (2005)
OSL	Optically stimulated lum.	Dating sediments (quartz)	HUNTLEY et al. (1985)
IRSL	Infrared stimulated lum.	Dating sediments (feldspar)	HÜTT et al. (1988)
IR-RF	Infrared radiofluorescence	Dating sediments (feldspar)	TRAUTMANN et al. (1999a)
LM-OSL	Linearly modulated OSL	Analytical tool (quartz)	BULUR (1996)
HR-OSL	Spatially resolved lum.	Dating rock surfaces	GREILICH et al. (2002)
TT-OSL	Thermally transferred OSL	Experimental (quartz)	WANG et al. (2006 a)

plier tube and subsequently counted. The first measurement of TL empties the electron traps after which a second heated measurement is undertaken to record the unwanted light signal due to black body radiation or incandescence. This starts to grow around 400°C when using blue-violet detection filters, and the sum of this is subtracted from the first signal (Fig. 4). The different TL peaks of the glow curve represent different trap populations, with electrons from “shallow” traps recombining at lower stimulation temperatures than those from “deep” traps at higher temperatures, and a comparison of naturally and artificially induced TL reveals information about the stability of different signal components. This characteristic is utilised to identify the appropriate thermal pre-treatment for reliable measurement, and to investigate the extent to which a sample was zeroed by the natural heating/bleaching process.

TL was the only method used in retrospective dosimetry and luminescence dating until the mid-1980s, and was originally developed for the dating of ceramics (AITKEN et al. 1964), although it was also used later to date volcanic rocks (e.g., HWANG 1970; MAY 1979; RAYNAL et al. 1982), heated artefacts and sediments (HUXTABLE et al. 1978) as well as aeolian deposits (e.g., WINTLE & HUNTLEY 1979, 1980; WINTLE 1981; SINGHVI et al. 1982). However, of the applications mentioned above it is only the TL dating of ceramics and heated artefacts that remains in regular use today, while the dating

of sediments using TL was largely abandoned in the mid-1990s as it presents a significant disadvantage compared to optical dating. This arises because part of the thermally stimulated luminescence signal is not sensitive to natural daylight, on which sediments rely on for zeroing or bleaching. Thermal stimulation releases trapped charges associated with both light-sensitive and light-insensitive components and so can lead to potentially severe age overestimates. Under optimum experimental settings, i.e. direct sunlight exposure of min-

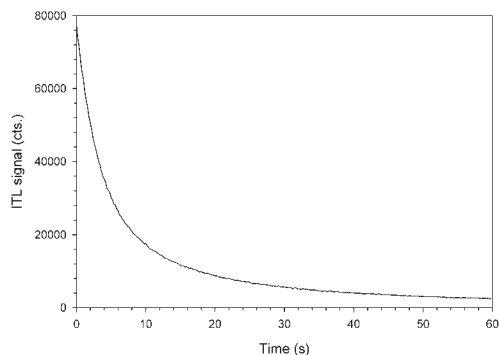


Fig. 5: Natural isothermal decay curve of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007), preheated at 230°C for 10 s and measured at 325°C.

Abb. 5: Natürliches isothermales Zerfallssignal einer Quarz Probe aus Nigeria (GUMNIOR & PREUSSER 2007), die bei 230°C für 10 s vorgeheizt und anschliessend bei 325°C gemessen wurde.

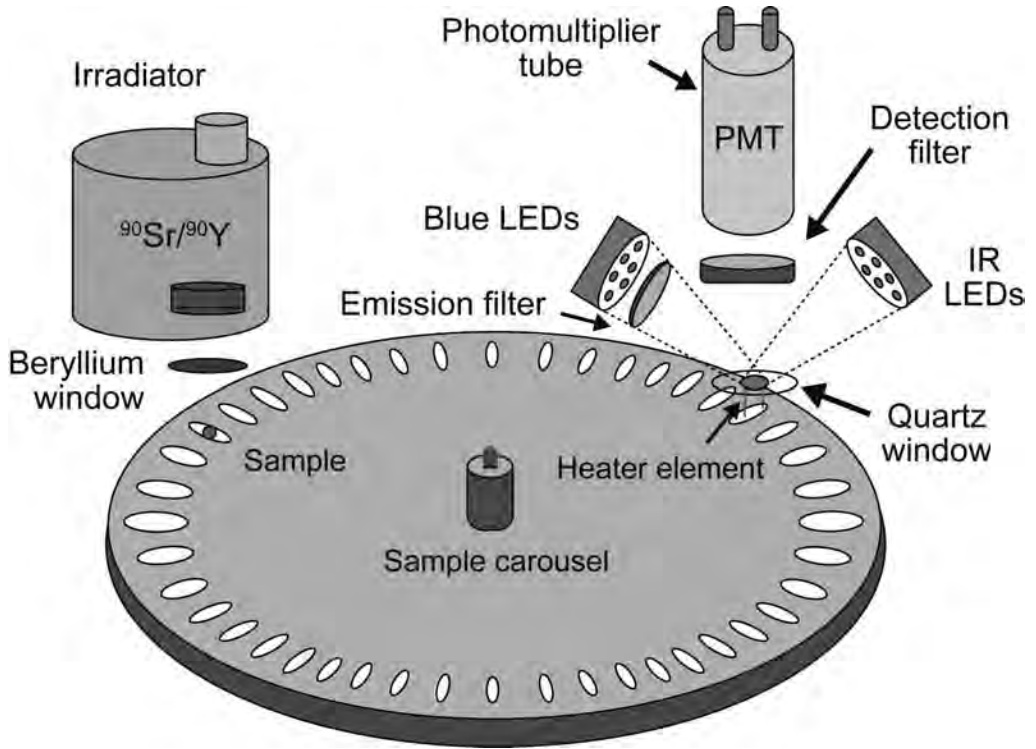


Fig. 6: Sketch showing the basic features of a TL/OSL reader (redrawn after Risø National Laboratory).

Abb. 6: Die Skizze zeigt den grundlegenden Aufbau eines TL/OSL Gerätes (umgezeichnet nach Risø National Laboratory).

eral grains, it will take several hours for the TL signal to be reduced to a small residual signal while the optical signal is almost fully reset within a few minutes (GODFREY-SMITH et al. 1988).

A modification of the classical TL approach is the isothermal TL (ITL) method, in which TL is recorded while the sample is held at a constant or isothermal temperature (JAIN et al. 2005; HUOT et al. 2006) (Fig. 5). The advantage of this method is that a specific trap population is emptied while deeper traps are not stimulated. In contrast to TL it is thus possible to focus on a trap population with certain physical properties. A first test study indicated that the potential of the method lies in the prospect of increasing the age range towards older samples (JAIN et al. 2005; CHOI et al. 2006a), although, while some tests on quartz

showed quite promising results, other test studies identified several problems and age overestimates in comparison to optical dating (BUYLAERT et al. 2006). Nevertheless, ITL appears to be an interesting approach with some potential, especially for older sedimentary samples and heated objects.

4.2 Optical stimulation

The most important breakthrough in recent years in luminescence dating has been the use of optical stimulation (optical dating). This area of luminescence dating mainly comprises stimulation by visible light as introduced by HUNTLEY et al. (1985), and by infrared (IR) as first described by HÜTT et al. (1988), may be collectively referred to as photoluminescence or photon-stimulated luminescence (PSL). The

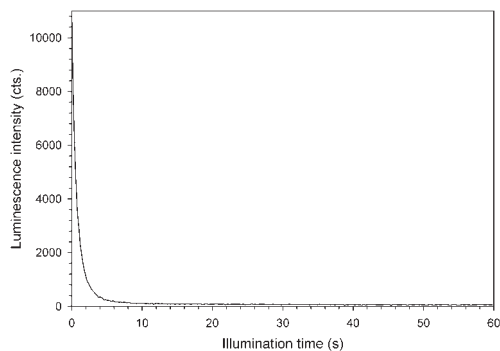


Fig. 7: OSL decay curve of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007) resulting from blue diode stimulation. Sample preheated at 230°C for 10 s and measured at 125°C.

Abb. 7: OSL-Zerfallskurve einer Quarz-Probe aus Nigeria (GUMNIOR & PREUSSER 2007), stimuliert mit blauen Dioden. Die Probe wurde bei 230° vorgeheizt und bei 125°C gemessen.

advantage of any optical method is that only the light-sensitive part of the luminescence signal is stimulated, which for sedimentary deposits considerably minimises the problem of incomplete bleaching of the luminescence signal prior to deposition. Furthermore, more reliable and precise methods for determining the palaeodose have been developed since the mid-1990s and have revolutionised luminescence dating. These methods will be described below. Regardless of what kind of photon-stimulation is used, optical filters are placed between the sample and the light-sensitive surface of the photomultiplier tube to minimise crosstalk between light from the stimulation source and light emitted from the sample (Fig. 6). When optical excitation begins the latent luminescence in the mineral will start to decay, resulting in a so-called decay curve recorded by the photomultiplier (Fig. 7).

4.2.1 Optically stimulated luminescence (OSL)

The term optically stimulated luminescence (OSL) is used here only with reference to stimulation by visible light, although in some

literature OSL is used as an umbrella term for all light stimulation including IR wavelengths. Stimulation by visible light is conducted by exposing the sample to a laser beam, the light of a filtered halogen lamp or to high-power light emitting diodes (LEDs), the latter being the latest and most versatile development, and hence used in the majority of currently manufactured luminescence readers (Fig. 6). The width of the excitation waveband differs for different stimulation sources but all lie in the green to blue part of the spectrum, i.e. between 420-550 nm. Both quartz and feldspar respond to stimulation within this waveband although for dating applications it is principally used for measurements of quartz. Light of longer wavelengths becomes increasingly inefficient at stimulating OSL in quartz, whereas wavelengths in the near infrared excite luminescence in feldspars due to one or more excitation resonances. Light emissions for OSL from quartz are detected in the ultraviolet range.

4.2.2 Infrared stimulated luminescence (IRSL)

In contrast to visible light, low-energy photons from the IR band will stimulate luminescence in feldspars but not in quartz (HÜTT & JAEK 1989). The most common term for this phenomenon is infrared stimulated luminescence (IRSL) although some researchers refer to it as IR-OSL or IR-PSL. Stimulation uses either an IR laser or IR diodes, the latter now being the most commonly used. The fact that quartz does not respond to IR stimulation is regularly used to check quartz-separates for feldspar contamination. One advantage of feldspar stimulation with IR is that it allows the detection of different recombination processes in feldspars with different physical properties that are reflected by distinct light emission in the UV, blue, yellow and red emission bands (KRBETSCHKEK et al. 1997). The potential of comparing different emission bands, in particular in the context of detecting and overcoming fading of feldspar IRSL (see below), is not yet fully utilised as only a few studies have so far compared the

various sample emissions (e.g., KRAUSE et al. 1997; PREUSSER 1999b; PREUSSER et al. 2003). Recent research has focused on IRSL red emissions and it is expected that the apparent stability of the IRSL signal in this part of the spectrum may overcome problems related to signal stability, i.e. regarding fading and resulting age underestimation (FATTAHI & STOKES 2003 a, b; STOKES & FATTAHI 2003). However, the detection of red IRSL from feldspars using conventional luminescence readers presents some problems, and the use of more sophisticated instrumentation has shed doubt on its stability (BARIL & HUNTLEY, 2003). Spectral measurements in this study identified a very low stability of the red (about 700 nm) IRSL emission of feldspars.

4.2.3 Infrared radiofluorescence (IR-RF)

Infrared radiofluorescence (IR-RF) dating was first introduced by TRAUTMANN et al. (1999a) and was referred to as radioluminescence (RL) dating. Because the underlying physical process of this type of luminescence is quite different from that associated with TL, OSL and IRSL dating, the change in nomenclature was useful to physically distinguish this more recently developed dating method (ERFURT & KRBETSCHKEK 2003b). In IR-RF dating the palaeodose is determined by a (IR emitting) fluorescence process, i.e. a prompt radiative charge transition. An emission at 865 nm during excitation by ionising radiation is typical for potassium feldspars (orthoclase, microcline) only. The physical basis of this phenomenon and methodological aspects of its application in dating were investigated to develop a reliable new technique for age determination of sediments (SCHILLES et al. 1999; TRAUTMANN et al. 1999a, b; KRBETSCHKEK et al. 2000; SCHILLES & HABERMANN 2000; TRAUTMANN et al. 2000; TRAUTMANN 2000; ERFURT 2003, ERFURT & KRBETSCHKEK 2003a). The IR emission can be interpreted as the transition of electrons from the conduction band into a particular kind of electron trap. During ionising irradiation a number of electrons reach the conduction

band from which they promptly recombine with luminescence centres and emit visible (VIS) fluorescence or transfer to that electron trap by emission of near-IR fluorescence (cf., process c in Fig. 2.1). The trap is probably the same as stimulated by IRSL (process a in Fig. 2.2). Compared to OSL and IRSL, the IR-RF signal bleaches slightly more slowly under light exposure although much faster than TL. As the RF-dose characteristics in quartz are far more complex and variable than in K-rich feldspars (KRBETSCHKEK & TRAUTMANN 2000), the majority of studies have so far concentrated on feldspars.

Palaeodose determination based on the phenomenon of IR-RF of potassium feldspars has different advantages compared to TL, OSL and IRSL. Firstly it is a direct measure of the electron density of a (well defined) trap, which does not rely on the conventional luminescence centres (which, as shown, can fade). This also explains why IR-RF dose response curves decrease with increasing dose (Fig. 8) – if the electron trap is empty (zero or low dose) the number of transitions into the traps are high (large signal), if the trap is filling up, the signal decreases due to the decreasing number of free traps. Another advantage of this method is that luminescence stimulation and dose accumulation are applied at the same time. This allows continuous measurement and the recording of a very high number of dose points (Fig. 8) and follows a strict stretched single-exponential decay curve for physically defined reasons. Furthermore, single aliquot regeneration procedures (see Section 5.3) are used for dose determination (ERFURT & KRBETSCHKEK 2003b) and also contribute to high precision in palaeodose estimates using the IR-RF method.

The measurement of an IR luminescence emission itself however, together with its low dynamic range and the special procedure of dose application and measurement is not a simple task and requires special instrumentation (ERFURT et al. 2003), which is not commercially available, and explains why this method is broadly accepted (BØTTER-JENSEN et al. 2003a; GEYH 2005) but only carried out at

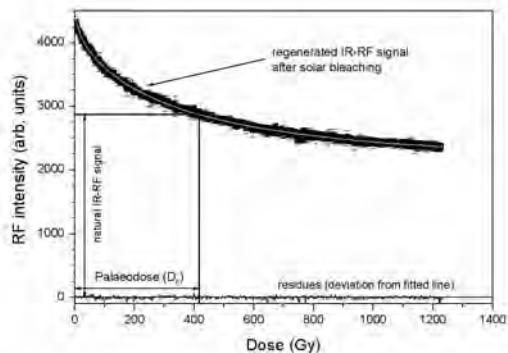


Fig. 8: IR-RF single aliquot regeneration dose response curve of a fluvial sample from central Germany (open cast mine Delitzsch) used to determine the palaeodose (IRSAR-protocol by ERFURT & KRBECSHEK 2003b). Note the high number of dose points and how the bottom curve (residual: deviations from the fitted line) shows good agreement to the fitted stretched-single-exponential decay curve. The low dynamic range of the IR-RF signal can also be seen.

Fig. 8: IR-RF – Dosisfunktion eines Einzelpräparates einer fluviatilen Probe aus Mitteldeutschland (Tagebau Delitzsch), verwendet zur Bestimmung der Paläodosis (IRSAR-Protokoll nach ERFURT & KRBECSHEK 2003b). Man beachte die große Zahl an Dosispunkten und die durch die untere Linie (Residuen: Abweichung von der angepassten Kurve) gezeigte gute Übereinstimmung mit der Anpassung an eine gestreckt einfach-exponentiell absinkende Kurve. Der geringe dynamische Umfang des IR-RF-Signals ist ebenfalls sichtbar.

one luminescence dating laboratory (Freiberg, Germany). Its dating-range spans 20 ka to about 350 ka in most cases and as far back as 500 ka with low dose rates. The IR-RF signal stability has been proven through different physical experiments, and also shows a good agreement with independent age control up to 250-300 ka (ERFURT et al. 2003; DEGERING & KRBECSHEK 2007). It has so far been applied to sediment dating where the signal is optically reset but can also be used to date thermally reset events. DEGERING & KRBECSHEK (2007) and KRBECSHEK et al. (2008) have published data of limnic, fluvial and other water-lain sediments back to about 350 ka. Developments

in instrumentation including the application of single grain dating, for which the first successful experiments have been made, will improve this method with regard to its precision and the potential to extend the age range.

4.2.4 Linearly modulated OSL (LM-OSL)

In contrast to “conventional” optical stimulation where the energy of the stimulation source is kept constant (continuous wave, CW-OSL), in linearly modulated OSL (LM-OSL) the stimulation power from the LEDs is ramped slowly (typically over 10^3 to 10^4 s) in a linear fashion from zero to some preset value (BULUR 1996; BULUR et al. 2000). The result of this slow increase of stimulation intensity is that in quartz the trapped charge is released first from shallow, and subsequently to deeper traps. This procedure results in overlapping peaks of luminescence emission related to different traps (in some sense similar to TL peaks). Due to the overlap of different components it is necessary to apply mathematical deconvolution to the LM-OSL data to discriminate between different components (cf., CHOI et al. 2006b). The result of this fitting gives information on the presence of different OSL components in a particular sample (Fig. 9), which are grouped according to when they occur during LM-OSL. Fast, medium and slow components have been identified, with the latter consisting of up to five individual sub-components (JAIN et al. 2003; SINGARAYER & BAILEY 2003), and all of which have different light sensitivities (BAILEY et al. 1997). The first of these is the fastest emission and it follows that this is the easiest to optically bleach in the natural sedimentary environment. Whether or not these different components originate from the same or different optical charge traps is still not fully understood. It is important to note that not all components are present in quartz of different geological origin (Fig. 9) and that the relation between sample properties and different trap populations remains vague. Furthermore, due to uncertainties related to mathematical deconvolution, LM-OSL has rarely been used for direct dating purposes. However, LM-OSL

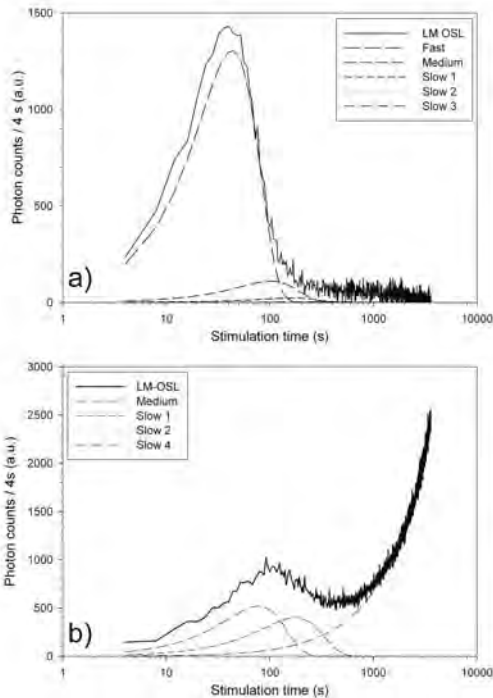


Fig. 9: Deconvoluted LM-OSL curve for (a) well-behaved Australian quartz and (b) quartz sample from Bavaria. The fast component that dominates the signal in sample (a) is not present in sample (b), which is dominated by an unstable medium component.

Abb. 9: Entfaltete LM-OSL Kurve für (a) einen australischen Quarz mit guten und (b) einen Quarz aus Bayern mit schlechten Lumineszenzeigenschaften. Die schnelle Komponente die das Signal in Probe (a) dominiert ist in Probe (b) nicht vorhanden; diese wird durch eine instabile mittlere Komponente dominiert.

is an extremely useful tool for characterising the properties of individual samples and can be used to detect the potential for poor behaviour in quartz, in an early phase of the dating process, although most important is the use of the fast component in circumventing problems of partial bleaching.

4.2.5 Spatially-resolved luminescence (HR-OSL)

A novel approach in luminescence is high resolution spatially-resolved luminescence (HR-

OSL) dating of stone surfaces (GREILICH 2004; GREILICH et al. 2002, 2005; GREILICH & WAGNER 2006). Similar to sediment dating, the aim is to determine the time when a stone surface was last exposed to daylight. The upper and lower surface of a granitic block, for example, will be shielded from daylight after erecting the wall of a historic monument. Dating the last daylight exposure of mineral grains within construction material hence reflects the age of a building. Besides archaeology, the approach also has great potential for geological applications, as sediments often contain pebbles and boulders. Optical dating of stone surfaces requires new approaches to sample preparation, palaeodose measurement and dose rate determination. In contrast to conventional luminescence dating, stone surfaces are kept intact to ensure only the grains exposed to light during the construction process are analysed. Because samples remain intact, this method has the great advantage that all information on the micro-dosimetric field is preserved; a detail which is destroyed during the traditional sample preparation. Aliquots are prepared by retrieving small drill cores (diameter ~10 mm) from a stone surface, cutting a thin slice (~2 mm thick) from the surface end of the core and mounting this slice in a specially designed metal holder after which, the luminescence measurements basically follow the single-aliquot regenerative-dose (SAR) protocol (see Section 5.2). However, the conventional way of measuring bulk luminescence signals using a photomultiplier tube is not appropriate for a stone surface with varying minerals and grain-size. In contrast, detection of the luminescence signal is achieved by means of a charged-coupled device (CCD) chip, which permits spatial resolution down to 25 μm x 25 μm . This produces a large amount of data (several thousand data points per individual measurement) and data management and analysis require a special software program (GREILICH et al. 2006). This software allows production of palaeodose values for each of the many picture elements, filtering of data and statistical analyses that can be spatially resolved for each pixel and then displayed in histograms.

The same spatial resolution for palaeodose calculation is needed for dose rate determination. This is achieved by using an energy dispersive X-ray analysis detector mounted on a scanning electron microscope (SEM-EDX) for the determination of the potassium content of feldspar grains. A special variant of fission-track analysis using tracks induced by fast neutrons is used to determine dose rates from uranium and thorium (WAGNER et al. 2005). From the spatially-resolved palaeodose and dose-rate data, luminescence ages are calculated for each of the analysed grains from a drill-core surface-slice. The last daylight exposure of a stone surface is calculated by combining data of several individually drilled samples.

4.2.6 Thermally transferred OSL

A new approach in the optical dating of quartz is the thermally transferred OSL (TT-OSL) technique (WANG et al. 2006 a). This method makes use of the two components of the thermally transferred OSL signal, which can be divided into the recuperated OSL signal and the basic transferred signal (AITKEN 1998). The recuperated OSL signal is generated by electrons being transferred via optical stimulation from the OSL traps into thermally unstable traps (refuge traps) (WANG et al. 2006a). After the OSL traps are emptied (through light exposure), the transfer from the refuge traps into the OSL traps is generated by thermal treatment. The subsequent light exposure results in a recuperated OSL signal, which is less sensitive compared to the OSL signal (WANG et al. 2006b), and is believed to have the potential to date samples beyond the level of saturation of the fast component of the OSL signal used in a common SAR-procedure (WANG et al. 2007). In contrast, the basic transfer signal derives from electrons trapped in light-insensitive traps and is released into light-sensitive traps by preheating and therefore inapplicable for dating. WANG et al. (2007) presented a SAR procedure using the recuperated OSL signal for palaeodose determination and showed agreement with OSL results.

5 Luminescence procedures

Several values are required to calculate a luminescence age and are either taken from empirical observations or are derived from mathematical expressions. These values are either associated with the determination of palaeodose and the reliability of this measurement or are used for the calculation of a dose rate. The following section gives a brief overview of how these values are determined, how they are taken into account in the age model and which values give information on the reliability of the applied dating procedures.

5.1 Sampling

A first and important point in any dating process is correct sampling. It is mandatory to collect detailed information on the geological context (including photographs, detailed field notes and so forth), particularly concerning the environment of sedimentary deposition and sediment overburden (sample depth below surface, indication for post-sedimentary disturbance, hiatuses, sedimentation cycles). Present and past hydrological conditions (sediment moisture) of the sample need to be assessed as far as possible. To ensure a uniform radiation field, the sample should be taken from a homogenous layer with a thickness of at least 50 cm. If no such layer is available, on-site measurement of gamma radiation (using a portable gamma spectrometer or a gamma counter) should be utilised to minimise the impact of inhomogeneity on dose rate calculation. Samples used for the determination of palaeodose have to be taken without exposing the material to daylight, and is usually achieved by forcing opaque metal or plastic tubes into a freshly cleaned exposure. Material from both ends of the tubes, which is likely to have had some daylight exposure, is discarded for palaeodose measurements but can be used for dose rate determination. Some laboratories require additional material for dose rate determination, particularly when high-resolution gamma spectrometry measurements are performed, but this

material need not be protected from daylight exposure. It is strongly recommended that researchers having little experience in sampling for luminescence dating should contact an expert prior to sample collection to get information on specific requirements.

5.2 Sample preparation

Prior to any luminescence measurements, a preparation procedure is carried out to retrieve quartz and K-feldspar separates. All preparation work for the determination of the palaeodose is carried out under subdued laboratory illumination to avoid any loss of luminescence. Most luminescence facilities have either red or orange low intensity lights installed. The first steps include treatment by HCl and H₂O₂ to remove carbonates and organic material, respectively, always followed by washing with deionised water. A particular grain size is then isolated by

either sieving (sand-size grains) or settling (silt). For sand sized grains, quartz and K-feldspar separates are isolated using heavy liquids with densities of 2.58 g cm⁻³, 2.62 g cm⁻³ and 2.70 g cm⁻³ (MEJDAHL 1985). Treating the quartz fraction with concentrated HF acid etches the surface of the quartz by about 10 µm to minimise the alpha-luminescence and also to minimise feldspar minerals by dissolution (plagioclase has a similar density as quartz), followed by treatment with HCl to remove any fluorides that may have formed during etching and dissolution. Etching with 10 % HF is often applied to remove the outer part of K-feldspar grains and etching with either HF (at various concentrations) or H₂SiF₆ is used to isolate fine-grain quartz separates (cf., MAUZ & LANG 2004). Alternatively, one can also use polymineral fine-grain samples for palaeodose determination. In such samples the IRSL signal will usually be dominated by feldspar emissions as quartz is not sensitive to

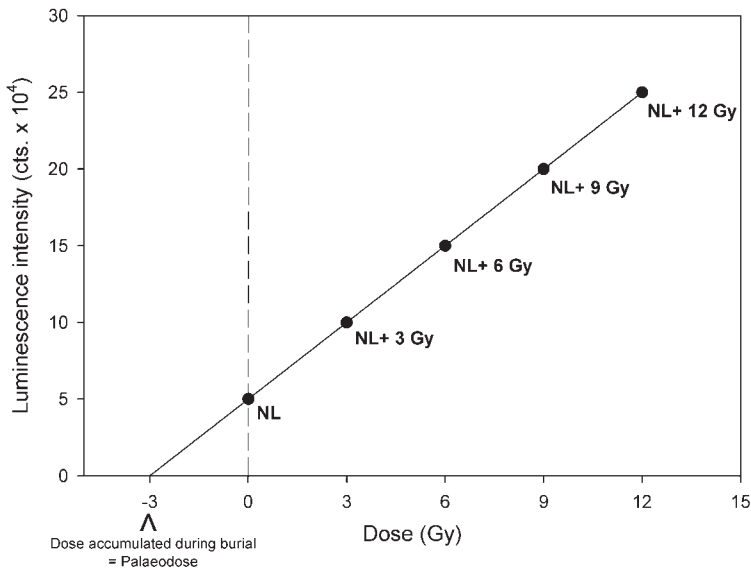


Fig. 10: Additive-dose response curve. Different laboratory doses are added to the natural signal (NL) to characterise the increase of luminescence intensity with dose. The best fitting function is then extrapolated to calculate the dose absorbed during burial (palaeodose).

Abb. 10: Dosisfunktion nach der additiven Methode. Verschiedene Dosiswerte werden im Labor zum natürlichen Signal (NL) hinzugefügt um das Anwachsen der Lumineszenz mit zunehmender Dosis zu charakterisieren. Die beste Kurvenanpassung an die Messwerte wird dann extrapoliert, um die Dosis zu bestimmen, die während der Lagerungszeit absorbiert wurde (Paläodosis).

IR stimulation. In all approaches the grains are mounted on ~10 mm diameter stainless steel or aluminium discs. Fine-grained silts are left to slowly settle (usually through a small vial of acetone) onto the discs; a silicon spray is used to mount sand-sized grains. With sand-sized grains, circular templates are commonly used to determine the quantity of mounted grains.

5.3 Determination of the palaeodose

Measurements are carried out on specialised luminescence equipment and further information regarding technical details about the most commonly used device manufactured by the Risø National Laboratory is found in BØTTER-JENSEN et al. (2003b). The machine consists of a measurement chamber equipped with a sample changer, a sample heating device, stimulation sources (laser or LEDs), a detection unit (photomultiplier tube and optical filters) and, usually in all laboratories, an integrated irradiation source ($^{90}\text{Sr}/^{90}\text{Y}$ beta source) (Fig. 6). In multiple aliquot dating (see below) irradiation is often carried out using external radioactive sources.

In principle there are two different approaches to determine the palaeodose: additive dose and regenerative dose. For both approaches, a so-called dose response curve is calculated reflecting the increase of latent luminescence in the mineral grains with increasing dose. In the additive dose approach an artificial laboratory-induced signal is added to the natural signal (Fig. 10), while in the regenerative dose method the latent luminescence signal is first removed and the sample is then irradiated and a laboratory signal regenerated (Fig. 11). The advantage of the regenerative method is that no extrapolation of the dose response curve is necessary. This fitting of the curve can cause significant uncertainties in the additive dose approach, especially where data are scattered or for older samples where the growth curve reaches saturation. Small fitting errors can produce large errors in palaeodose determination. The disadvantage of the regenerative method is that the process of zeroing the signal in the grains has been proven to often change the luminescence

properties of a sample. If such changes in luminescence sensitivity are not corrected for, the palaeodose will be systematically under- or overestimated (WINTLE 1997).

Regardless of which approach is being used, the measurement of palaeodose comprises three different steps: irradiation, preheating and measurement of luminescence. Irradiation is carried out to induce a latent luminescence signal in the grains and is performed by using either beta sources (external or integrated in the luminescence reader) or external gamma sources. During artificial irradiation all traps, including those unstable over longer time periods, capture electrons. To allow comparison with the palaeodose absorbed over the burial time, the unstable component of the luminescence signal has to be removed, and is achieved by heating the sample (between 200°C and 300°C for usually 10 s, or several hours at temperatures of about 150°C) causing eviction of the electrons caught at shallow traps. Once this preheating of the sample has been performed, the luminescence is measured by stimulating the sample with either heat or light.

5.3.1 Multiple aliquot techniques

Up to the late 1990s, most luminescence dating studies used multiple aliquot approaches. With this method, determination of the palaeodose is carried out using several (usually five) subsamples (aliquots) for the different irradiation doses. The average of these individual dose groups is then used for the construction of a dose response curve. The major disadvantage of a multiple aliquot approach is that each aliquot often behaves differently (due to e.g., variable sensitivity changes, incomplete and variable bleaching, etc.) giving rise to scatter in the data. For this reason, a relatively large uncertainty is often associated with mathematical fitting of dose response curves constructed by multiple aliquot approaches. Furthermore, it has been shown that samples are often affected by changes in luminescence sensitivity when using regenerated doses and this approach should not be used if this change cannot be

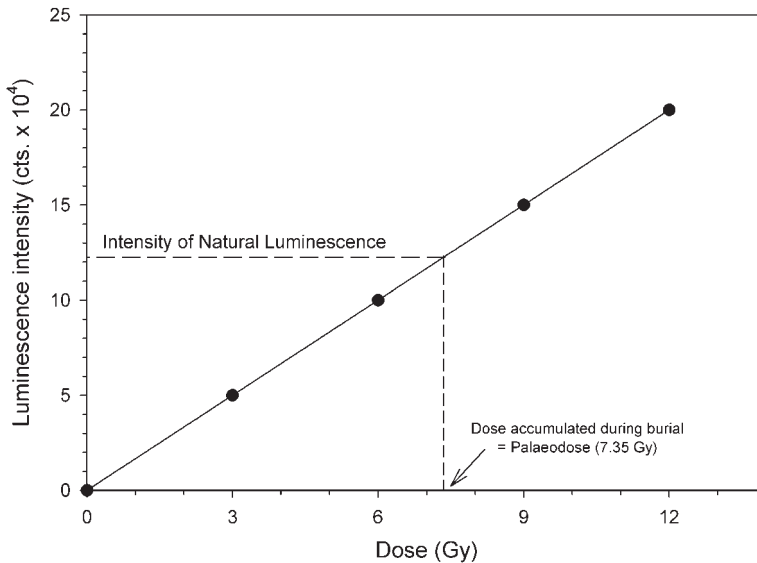


Fig. 11: Regenerative-dose response curve describing the increase of luminescence with dose. Once the natural luminescence signal has been erased by either light or heat exposure, different laboratory doses are administered to the aliquots, and the luminescence response to these used to construct a curve of best fit. The intensity of the natural luminescence signal (dotted line) is then projected onto this to determine the dose absorbed during burial (palaeodose) using interpolation.

Abb. 11: Dosisfunktion nach der regenerativen Methode. Mit verschiedenen Dosiswerten werden im Labor Einzelpräparate bestrahlt, deren Lumineszenzsignal zuvor durch Licht oder Wärme gelöscht wurde. Damit soll das Anwachsen des Lumineszenzsignals mit der Dosis bestimmt werden. Die Intensität des natürlichen Lumineszenzsignals (gestrichelte Linie) wird dann auf die beste Kurvenanpassung projiziert, um die Dosis durch Interpolation zu bestimmen, die während der Lagerungszeit akkumuliert wurde (Paläodosis).

corrected (DULLER 1994; WINTLE 1997). The major disadvantage with the additive dose approach as mentioned above is the uncertainty of extrapolation with particular regard to older samples.

5.3.2 Single aliquot techniques

When HUNTLEY et al. (1985) published their benchmark paper on optical dating, they already mentioned the possibility of using single aliquot approaches. The main aspect of any such approach is that all measurements are carried out on a single portion or aliquot of the same grains, which circumvents the problem of individual luminescence properties of grains from different aliquots. Several approaches were tested in the 1990s, most of which con-

tributed to the improvement of methodology but included methodological problems that threw doubt on the use of these approaches in routine dating applications (e.g., DULLER 1991; MEJDAHL & BØTTER-JENSEN 1994; ZANDER et al. 2000). A major breakthrough was made with the development of the single-aliquot regenerative-dose (SAR) technique that was mainly developed and further refined by MURRAY & WINTLE (2000, 2003). As this method is now used in most dating applications by the majority of laboratories world-wide, the principles and procedures will be described in some detail below. A more detailed review focussing on technical details has been presented by WINTLE & MURRAY (2006).

The basic idea behind the SAR technique is to utilise a regenerative dose approach and correct

for any sensitivity change that may occur in the course of the measurement procedure. To carry out this sensitivity correction, a so-called test dose, which remains constant during the whole protocol, is administered after measuring each regenerative (including natural and zero) dose (Fig. 12). The luminescence intensity resulting from each regenerative dose (L_x) is normalised by the luminescence intensity of the following test dose (T_x) and the ratio L_x/T_x , which represents the sensitivity-corrected luminescence signal, is used to construct a dose response curve (Fig. 13). For suitable curve fitting, construction of the latter requires at least three different regenerative dose points (plus a zero dose point). Flexibility in the SAR protocol permits an increase in the number of dose points if required, and can be used to improve the mathematical fitting of the dose response curve when a sample is approaching saturation.

The performance of each individual SAR cycle is verified by two internal tests: recuperation and recycling. The first test considers the signal from an aliquot given zero regenerative dose and then the same test dose as every other SAR cycle, for which L_0/T_0 should be close to 0.00. Values above zero indicate that an unwanted signal was induced by preheating the sample prior to measurement (recuperation), and MURRAY & WINTLE (2000) suggest that recuperation should not exceed 5 % of the natural sensitivity corrected signal intensity (L_N/T_N). If recuperation exceeds this value, it is necessary to modify the protocol by either changing the preheat procedure or by adding a so-called “hot-bleach” (illuminating the sample at elevated temperature) to avoid any such effect (MURRAY & WINTLE 2003). The second test (recycling ratio) confirms whether correction for sensitivity change was successful. This test is carried out by measuring the luminescence response to the same regenerative dose at the beginning, and then again at the end of the SAR cycle (Fig. 13). If the sensitivity correction was successful, the ratio of the two measurements will be unity. MURRAY & WINTLE (2000) suggest discarding any measurements where the

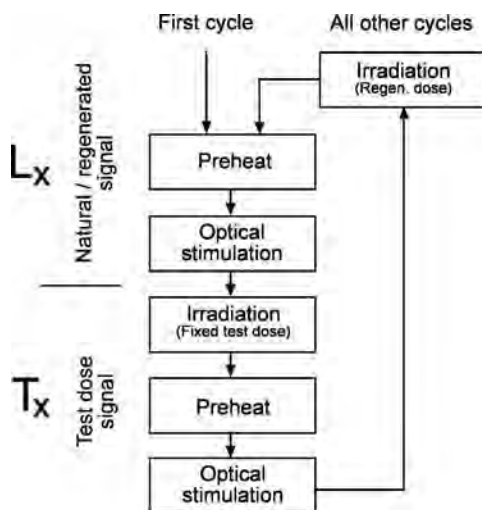


Fig. 12: Overview of the single-aliquot regenerative-dose (SAR) protocol (MURRAY & WINTLE 2000). L_x describes the luminescence signal of the natural sample (L_N) or different regenerative doses (e.g., $L_{10\text{ Gy}}$). T_x is the luminescence response to a constant test dose (e.g., 2 Gy) that is measured after the natural and each regenerative dose.

Abb. 12: Überblick über das Einzelproben-Protokoll mit regenerierter Dosis (SAR) (MURRAY & WINTLE 2000). L_x ist das Lumineszenzsignal der natürlichen Probe (L_N) bzw. verschiedener regenerierter Dosispunkte (z.B., $L_{10\text{ Gy}}$). T_x ist das Lumineszenzsignal, das aus der Bestrahlung mit einer konstanten Testdosis (z.B. 2 Gy) gewonnen wird. Dieses Signal wird nach der natürlichen Lumineszenz und nach jedem regenerierten Dosispunkt gemessen.

recycling ratio is $>10\%$ from unity (i.e., $0.90 < \text{recycling ratio} < 1.10$).

In addition to these quality controls performed during every SAR measurement, a series of further tests have been developed to ensure the reliability of the measurement procedure. If these tests do not provide acceptable results it is necessary to modify the measurement procedure. In this context it is important to recognise that, due to the nature of the signal and the variety of sediment sources, luminescence properties of minerals are different in different geological settings. Hence, it is mandatory to carry out a rigorous testing program for all dating projects (e.g., BLAIR et al. 2005; KLASSEN et al. 2006).

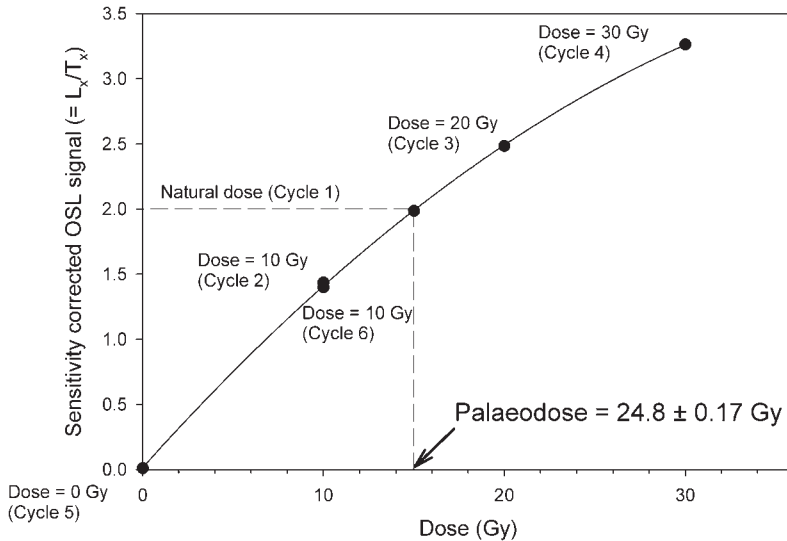


Fig. 13: A typical SAR-generated dose response curve. The ratio of L_x / T_x (e.g., L_{10} / T_{10}) is plotted versus applied regenerative dose. L_N / T_N is then projected onto this curve to determine the palaeodose through interpolation. The lowest regenerative dose is measured twice, at the beginning and end of each individual SAR cycle, to ensure that sensitivity changes have been sufficiently corrected for. The ratio of these two measurements is referred to as the *recycling ratio* and should be within 10 % of unity.

Abb. 13: Eine typische Dosisfunktion, die mit dem SAR-Protokoll gewonnen wurde. Der Quotient L_x / T_x (z. B., L_{10} / T_{10}) wird gegen die regenerierte Dosis aufgetragen. L_N / T_N wird dann auf diese Kurve projiziert um die Paläodosis durch Interpolation zu bestimmen. Das Signal der niedrigsten regenerierten Dosis wird zweimal, am Anfang und am Ende jedes einzelnen SAR-Zyklus, gemessen, um zu überprüfen, ob Sensitivitätsänderungen hinreichend korrigiert wurden. Das Verhältnis dieser wiederholten Messungen wird Wiederfindungsverhältnis (*recycling ratio*) genannt und sollte um weniger als 10% von Eins abweichen.

A basic requirement that proves the suitability of the SAR protocol is that a known dose that is administered in the laboratory can be retrieved by the measurement procedure (ROBERTS et al. 1999; WALLINGA et al. 2000). For testing this, a so-called dose recovery test is carried out during which the natural signal is first optically erased and then a known laboratory dose is given to the sample. This known dose is then treated as the natural dose in the SAR protocol and the ratio of measured/given dose should be unity if the procedure works correctly, and repeating this test for a sample gives information on the best achievable precision. This very much depends on the luminescence properties of the sample, especially luminescence intensity. Reported values range between relative standard deviations of a few percent, e.g. 4 % for Dutch

aeolian sands (VANDENBERGE et al. 2003), to between 10-15 % found for glacial deposits from Switzerland (PREUSSER et al. 2007).

A major problem that can, at least in some areas, interfere with correct determination of the palaeodose and is not directly detectable in standard SAR measurements is basic thermal transfer (RHODES & POWNALL 1994; RHODES & BAILEY 1997; RHODES 2000). The phenomenon is similar to the effect of recuperation (cf., AITKEN 1998) and has a major effect on the natural sample but not on subsequent regenerative dose measurements (RHODES 2000). A simple test to identify if thermal transfer affects a sample is carried out by erasing the latent luminescence and then measuring the apparent palaeodose (Fig. 14). Where the apparent palaeodose is significantly > 0 Gy this is due to a transfer of

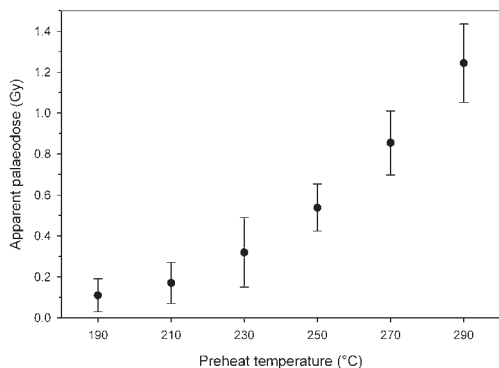


Fig. 14: Plot of palaeodose versus preheat temperature for a sample in which the OSL signal was zeroed by previous optical stimulation (thermal transfer test). The increase of apparent palaeodose results from an OSL signal that is induced by thermal transfer of electrons from light-insensitive to light-sensitive traps.

Abb. 14: Abhängigkeit der ermittelten Paläodosis von der Vorheiztemperatur für eine Probe, deren OSL-Signal durch vorherige optische Anregung auf Null gesetzt wurde (Test des thermischen Transfers). Das Anwachsen der scheinbaren Paläodosis resultiert aus einem OSL Signal, welches durch thermischen Transfer von Elektronen aus lichtinsensitiven in lichtsensitiven Fallen hervorgerufen wird.

charge from hard-to-bleach to easy-to-bleach traps. Most studies that have reported significant thermal transfer are related to quartz with a young sedimentary history, i.e. proglacial deposits (RHODES & POWNALL 1994; RHODES & BAILEY 1997; RHODES 2000; PREUSSER et al. 2006), although other studies have shown that thermal transfer may be negligible for some proglacial sediments (e.g., KLASSEN et al. 2006; PREUSSER et al. 2007). The test described above is a quick and effective way to exclude the presence of thermal transfer.

A further test exists to check whether the applied preheating procedure is sufficient to remove unstable signal components in the artificially induced signal (AITKEN 1985). For this, the palaeodose is determined for a series of different preheat temperatures (Fig. 15). If all the unstable charge is removed, the calculated palaeodose should not change with increasing

preheat temperature. However, it is possible that the phenomenon of thermal transfer is also present and in this case a compromise between a preheat procedure suitable to remove all unstable charge but without causing significant thermal transfer is necessary.

For feldspar samples, storage tests are carried out to monitor the short-term stability of the artificially-induced luminescence signal. This is carried out because feldspars can sometimes be affected by a phenomenon referred to as anomalous fading that describes a loss of signal with time (WINTLE 1973) (see section 6.3).

5.4 Determination of the dose rate

After obtaining the palaeodose, the next step in age determination for a sample requires assessment of the rate at which the radiation dose was delivered to the mineral grains. Similar to the palaeodose this assessment is made over the burial period, being the time since the sample last saw daylight during transport and deposition or since it was last exposed to heat. In most cases, the accumulated dose per unit time, the dose rate, is constant and the age t of a sample is simply calculated from the measured palaeodose D according to Equation 1. A minor component of the dose rate is produced by ionising cosmic radiation and can be estimated from geographic position and burial depth of the sampled material (PRESCOTT & HUTTON 1994) (Fig. 16). The larger part originates from the natural radionuclide ^{40}K and from the uranium and thorium decay series.

In principle, there are three ways to determine this contribution by either measuring the dose rate (a) directly in the sediment using dosimeters, (b) by measuring alpha, beta and gamma dose rates using radiation counting devices, or (c) by analysing the activity and/or concentration of the dose rate relevant nuclides followed by determination of the dose rate using well-established conversion factors. In practice, most laboratories use the last approach. Within the calculation some general assumptions are made for convenience (AITKEN 1985):

- The material is isotropic and homogenous

with respect to the distribution of the sources of ionising radiation.

- The investigated system is indefinitely expanded.
- All radionuclide concentrations are constant in time.

Infinite system dose rates are easily calculated from the specific activities of the radionuclides using conversion factors tabulated in the literature (cf., ADAMIEC & AITKEN 1998). Moisture corrections as described in AITKEN (1985) are necessary to consider the radiation attenuation by pore water. Inhomogeneity due to grain size, pore volume and moisture distributions, as well as of the mineral composition are removed in multi-grain samples by averaging over a large number of grains. An inhomogeneity remains between the radionuclide content of the investigated grain itself (causing the internal dose) and the averaged irradiation from all sources outside the grain (the external dose). This effect is dealt

with using a correction factor, which denotes the ratio of the actual absorbed dose to the theoretical one of an infinite system. Furthermore, the influence of HF etching of grain surfaces can be taken into account by such a factor. Calculated correction values were originally reported by MEJDAHL (1979) and BELL (1979, 1980) and more recently determined by Monte Carlo simulations (BRENNAN et al. 1991, BRENNAN 2003).

There is a variety of methods that can be utilised for radionuclide analysis in this context (cf., SINGHVI & KRBETSCHKEK 1996). Most commonly used are inductively-coupled-plasma mass spectrometry (ICP-MS), high-resolution gamma spectrometry, neutron activation analysis (NAA) and flame photometry (for K only). To check the performance of the procedures certified reference materials are available for all methods. ICP-MS has three main advantages: (a) small sample size (< 1 g required), (b) low detection limit and (c) high precision. The disadvantages of ICP-MS are: (a) small sample size can be a problem if the sediment exposure is inhomogeneous, (b) total dissolution of the sample, especially if zircon is present, is not straightforward, (c) measuring K by ICP-MS is rather problematic (cf., PREUSSER & KASPER 2001) and (d) this method does not give any information concerning whether radioactive disequilibrium is present in the uranium and thorium decay chains. High-resolution gamma spectrometry is rather time-intensive (each measurement takes at least a day) and requires usually at least 100 g of sample material. Larger sample sizes do, however, reduce uncertainty due to inhomogeneity. The most important advantage of gamma spectrometry is related to the assessment of possible radioactive disequilibrium in the thorium and uranium decay chains (see section 6.4).

The internal dose rate contribution is usually considered to be negligible for quartz grains. In K-feldspars it is often estimated from the stoichiometry of the mineral. DÜTSCH & KRBETSCHKEK (1997) proposed a method for the direct determination of the internal potassium concentration of feldspar grains by studying the peak position of the red radiophosphorescence at about 720 nm. This method was successfully

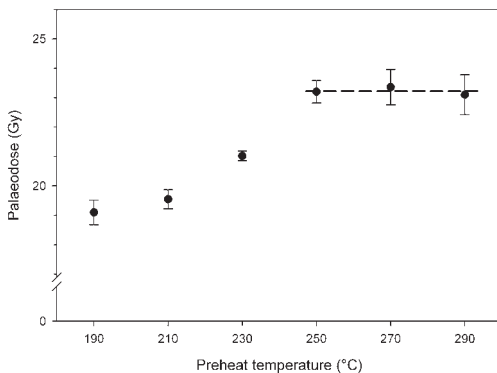


Fig. 15: Preheat tests are performed to determine the thermal pre-treatment that erases all thermally unstable luminescence components in a sample. In this example, palaeodose remains constant for preheat temperatures of 250°C and above (preheat plateau).

Abb. 15: Vorheiztests werden durchgeführt, um diejenige thermische Vorbehandlung zu ermitteln, mit der alle thermisch instabilen Lumineszenzkomponenten in der Probe entfernt werden. In diesem Beispiel bleibt die Paläodosis konstant für Vorheiztemperaturen ab 250°C (Vorheizplateau).

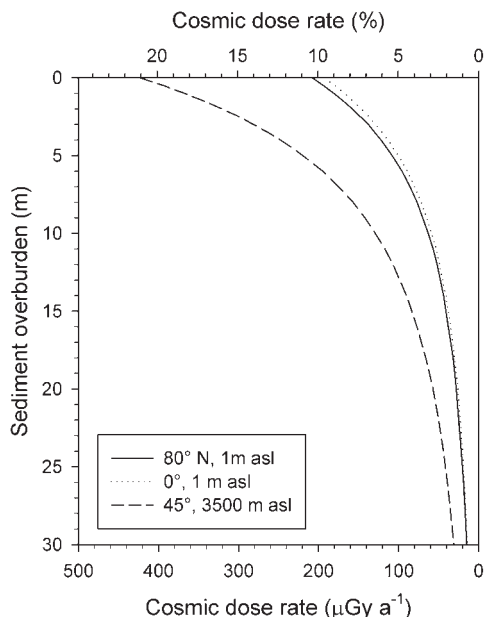


Fig. 16: Dependency of cosmic dose rate on geographic location and sediment overburden, with examples illustrated for sea-level at both high latitude and an equatorial position, as well as a mid latitude position at high altitude. Longitude has a negligible effect on cosmic dose rate. The lower x-axis gives absolute values while the upper x-axis indicates the relative contribution from cosmic rays for a generic sample reflecting a typical sediment with a mean dose rate of 2 mGy a^{-1} .

Abb. 16: Abhängigkeit der kosmischen Dosisleistung von der geographischen Position und der Sedimentüberdeckung. Dargestellt sind Beispiele für hohe Breiten bzw. am Äquator auf Meeressniveau sowie eine Position in den mittleren Breiten in großer Höhe. Der Längengrad hat einen vernachlässigbaren Effekt auf die kosmische Dosisleistung. Die untere x-Achse enthält Absolutwerte, während die obere x-Achse den relativen Beitrag der kosmischen Strahlung für eine Probe mit einer für viele Sedimente typischen mittleren Dosisleistung von 2 mGy a^{-1} anzeigt.

applied to sediments with non-stoichiometric K contents (DEGERING & KRIBETSCHKE 2007).

5.5 Age calculation

Once both the palaeodose and all dose-rate relevant information are available the lumi-

nescence age of a sample together with its uncertainty can be calculated (cf., Aitken 1985). In single aliquot dating, the technique now used by the majority of luminescence dating laboratories, the palaeodose is assessed from many replicated single aliquots. Several statistical methods have been developed to analyse luminescence data and determine the palaeodose. For those samples shown to be completely bleached prior to deposition, some form of central tendency approach is used such as (weighted) arithmetic mean, median or a central age model (cf., Galbraith et al. 1999; Bailey & Arnold 2006). For the palaeodose values determined using any of these approaches standard deviation and standard error are then determined (cf., Geyh 2008). For incompletely bleached samples determination of the palaeodose is more complex (see sections 6.1 and 6.2). For dose rate calculation, the results of measuring dose rate relevant elements (K, Th, U) are combined with the contribution from cosmic radiation and several sample specific factors such as grain size and moisture content have also to be considered (see sections 5.4 and 6.4). These quite complex calculations are usually carried out using special computer programs such as AGE (by R. Grün, Canberra) or ADELE (G. Kuhlrig, Freiberg).

6 Problems hindering accurate age determination

6.1 Incomplete bleaching

One of the major problems in luminescence dating is incomplete resetting or bleaching of the latent signal. For sediments, this can occur when the grains are exposed to adequate daylight conditions for too little time and/or if the grains have been transported and deposited by water. Incomplete bleaching is a particular problem for sediments from glacial environments as well as for some fluvial and hill-slope deposits. For all such deposits it is mandatory to investigate if the sample has been completely bleached prior to deposition or not and several approaches exist for doing so.

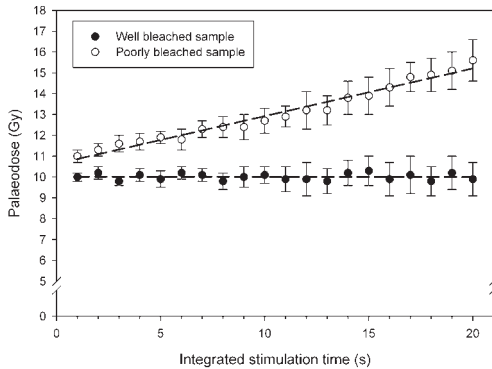


Fig. 17: Shine plateau tests for two samples indicating completely (plateau) and incompletely (increasing palaeodose values) bleached samples. The presence of a shine plateau does not guarantee that a sample has been completely bleached prior to deposition, where they may be potential interference from an unstable signal component.

Abb. 17: Ausleuchtplateautests für zwei Proben, die auf eine vollständig (flaches Plateau) bzw. unvollständig (ansteigende Paläodosisiswerte) gebleichte Probe hinweisen. Das Vorhandensein eines Ausleuchtplateaus belegt jedoch nicht zweifelsfrei, dass eine Probe vor der Ablagerung vollständig gebleicht wurde, da mögliche Störungen durch instabile Signalkomponenten vorliegen können.

One approach is to plot palaeodose as a function of optical stimulation time (in the case of OSL and IRSL) or temperature (in the case of TL). The basic idea behind this approach is that the signal during initial stimulation and at lower temperature, respectively, is more light-sensitive than the subsequent following signal (LI 1991, SINGHVI & LANG 1998, BAILEY 2003a, b). As a consequence, one would expect an increase of palaeodose with stimulation time (temperature) for incompletely bleached samples and a flat shine-plateau-plot for samples that were completely reset at deposition (Fig. 17). Unfortunately, this approach has not been proven to ubiquitously identify incomplete bleaching (e.g., FIEBIG & PREUSSER 2007). Hence, a flat shine-plateau may only be interpreted as evidence for, but not proof of complete bleaching.

The alternative and recently more widely used approach is based on the assumption that sediment grains are not only incompletely, but differentially bleached prior to deposition. This means that the luminescence signal may be completely bleached in some of the grains while the remaining may carry varying amounts of residual signal. When measuring several individual grains from a sample, the lower values in a distribution of doses will most likely represent the grains that were zeroed while the upper values in the distribution will reflect grains that had residual luminescence at deposition (MURRAY et al. 1995). Typically, a completely bleached sample is expected to reveal a Gaussian distribution while incompletely bleached samples should show a positively skewed distribution of palaeodose estimates (Fig. 18). Although many studies have demonstrated Gaussian distributions for samples with the same radiation dose, recent work indicates that in some cases a Gaussian distribution may not be a sufficient criterion for identifying completely bleached samples (FUCHS et al. 2007).

If a sample has been identified as being incompletely bleached the age estimate based on the mean palaeodose for all aliquots has to be considered as the maximum deposition age. To determine the true burial palaeodose the dose distribution must be investigated further, and for this, it is necessary to measure at least a few dozen individual palaeodose estimates to ensure a substantial statistical basis. The different statistical approaches to extract the true burial palaeodose from the distribution of differentially bleached samples will not be discussed in detail here, we would refer the reader to the overview by BAILEY & ARNOLD (2006). For the time being we have to consider that none of the approaches has proven to ubiquitously provide the correct result when compared to independent age control. Dating results for samples taken from certain environments, in particular proglacial settings, should be considered carefully until more sophisticated methods are available. As each sample has its own sedimentary history, it is advisable to date several

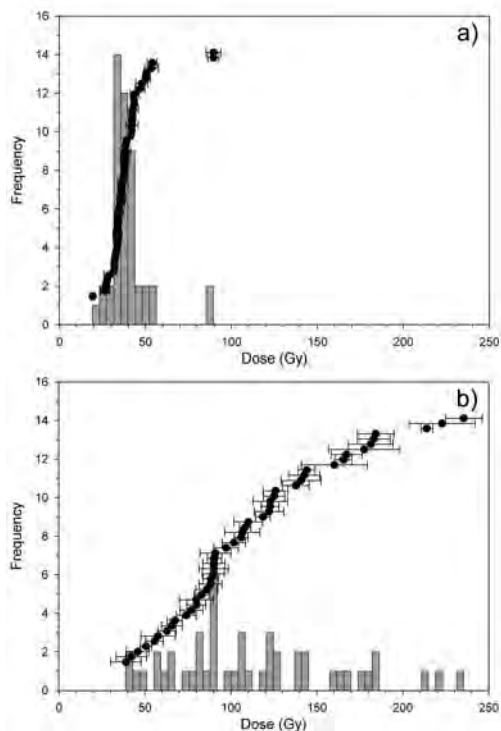


Fig. 18: Scatter of repeated palaeodose measurements for (a) a relatively well-bleached and (b) very poorly bleached sample. While sample (a) displays a symmetric distribution with only two aliquots having significantly higher doses, aliquots of sample (b) show a large scatter in palaeodose values. Both samples are from last glacial (Würmian, ca. 24 ka old) proglacial sediments from Switzerland (from PREUSSER et al. 2007).

Abb. 18: Streuung wiederholter Paläodosismessungen für (a) eine relative gut gebleichte und (b) eine sehr schlecht gebleichte Probe. Während Probe (a) eine symmetrische Verteilung mit nur zwei Aliquots bei signifikant höheren Dosiswerten zeigt, weisen die Aliquots der Probe (b) ein deutlich höheres Streuen der Paläodosismessungen auf. Beide Proben sind aus proglazialen Sedimenten der letzten Vergletscherung (Würmeiszeit, ca. 24 ka alt) aus der Schweiz entnommen worden (aus PREUSSER et al. 2007).

samples from the same sedimentary layer and cross-check the results, and will in most cases help to prove if the age can be considered reliable or needs to be interpreted with caution. Earlier studies also explored the lower light sensitivity of TL compared to optical signals.

For example, WINTLE et al. (1993) compared TL and IRSL of colluvial samples and found that TL ages are up to 10 ka higher than IRSL ages, although IRSL ages also overestimated the expected age and demonstrated that the approach can only be used for sediments where incomplete bleaching is of minor importance such as loess.

Different grain size fractions may also show different degrees of bleaching, with generally two different fractions used in luminescence dating: fine grains (4–11 μm) and coarse grains (90–200 μm). For alluvial sediments, several case studies have shown that the coarser grains appear to be better bleached than finer fractions (e.g., OLLEY et al. 1998; TRUELSEN & WALLINGA 2003) although relatively little systematic work has been carried out on this issue.

6.2 Scatter of single aliquot and single grain palaeodose values

Multiple replicates of palaeodose measurements on sub-samples from the same sediment sample always show a certain variation among the individual estimates, the causes of which are not all fully understood or quantified at present. One major demand in luminescence studies is to assess the extent of the scatter in palaeodose distributions and determine whether the extent of such scatter represents the natural variation of the dose absorbed during burial. If such a baseline variation is known, additional sources of scatter between palaeodose estimates, in particular caused by incomplete resetting of the luminescence signal prior to burial or pedogenesis (see below), can be identified and discriminated outside of this natural baseline variation (GALBRAITH et al. 2005). Only if these requirements are fulfilled can the true palaeodose accumulated during burial be extracted from complex dose-distributions. One important issue in this context is that the spread in palaeodose is very much dependent on the number of grains on the measured aliquot (WALLINGA 2002b). With regard to aliquot size, it is also important to recognise that only a small proportion of all grains show

a measurable optical signal (cf., DULLER 2004), hence, the luminescence signal from small aliquots most likely originates from a few or even single grains only. For large aliquots, containing several hundred or thousand of grains, the variation of natural palaeodose will be relatively small, whereas for small aliquots the variation will increase, and for single grains will increase still further. For example, the variation in palaeodose values observed for some fine-grain aliquots, which contain hundreds of thousands of grains, can be less than 1 %. On the other hand, a scatter of 20-30 % for small aliquots (less than 50 grains) of bright Australian aeolian quartz has been reported (LOMAX et al. 2003, 2007), although the OSL characteristics of this material are considered to be very suitable. Poorly behaved dim quartz grains, for example from the foreland of the Swiss Alps, show an even higher variation (PREUSSER et al. 2007).

Four different major sources may cause broad and complex palaeodose distributions and these are: (a) laboratory reproducibility of the measurement; (b) partial bleaching; (c) post-depositional sediment mixing caused by bioturbation or similar phenomena; and (d) variation in the distribution of radioactivity within the sediment (microdosimetry).

Reproducibility of the laboratory measurements can easily be determined for each individual sample with a dose recovery test (section 5.3.2). Depending on the particular properties of the minerals investigated and on aliquot size, this typically varies from about 5 % to 15 % (cf., PREUSSER et al. 2007). This is much less than the spread of palaeodose values observed in many natural samples. Incomplete bleaching of the luminescence signal, as discussed above, can result in a substantial scatter of palaeodose values resulting in overestimation of OSL ages. Additionally, pedo-/bioturbation can have an important effect on dose distributions, in particular for sandy deposits such as aeolian dunes (BATEMAN et al. 2003). Pedoturbation is common in former drylands that have later experienced increased humidity resulting in high floral and faunal occupation (BATEMAN et

al. 2007). The most common effect of pedoturbation will be contamination of older sediment by younger surficial material. This will cause a negatively skewed dose distribution where the lower palaeodose values are not representative of the variation in true burial dose. In contrast, partial bleaching will cause a positively skewed dose distribution where the true burial dose lies in the lower dose region, while the upper part of the distribution represents grains that were incompletely zeroed at deposition.

A source that may cause variation of palaeodose is inhomogeneity of exposure to beta irradiation from grain-to-grain within sediments (microdosimetry). This occurs due to the fact that radioactive elements are sufficiently abundant in only a few minerals such as K-feldspar (^{40}K) and zircon (^{238}U and ^{232}Th), so-called “hotspots” of radioactivity. As beta particles penetrate to only a few mm within typical sediment, grains proximal to hotspots will be exposed to a much higher dose rate than other grains and hence absorb more dose during burial. Although the majority of causes of natural variation of palaeodose are difficult to constrain, it is likely that the effect of microdosimetry may be inversely related to the number of hotspots within the sediment matrix. MAYYA et al. (2006) calculated that inhomogeneous microdosimetry will cause positively skewed palaeodose distributions and that the effect on the standard deviation for repeated single grain measurements may be between 18 % and 46 %, being inversely dependent on the K-content of the sediment.

6.3 Anomalous fading

The phenomenon of anomalous fading was first observed by WINTLE (1973) in a study of TL dating of sanidine. Fading is a loss of luminescence signal with time; this implies that some of the electrons captured at traps are unstable and do not possess the trap lifetime exhibited by the majority of electrons in the trap. The effect in a dating application will be that the laboratory induced signal from a particular trap will originate from both stable and unstable charge,

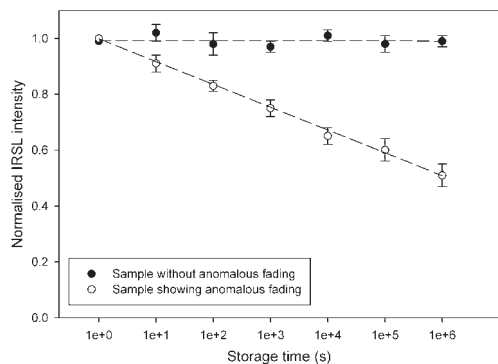


Fig. 19: In the presence of anomalous fading laboratory irradiation will produce a signal that decays (fades) over time. This same unstable signal is not present in the natural sample and, as a consequence, the absorbed palaeodose and hence the age of the sample will be underestimated.

Abb. 19: Bei vorhandenem anormalem Ausheilen (Fading) produziert die Laborbestrahlung ein Signal, welches mit der Zeit zerfällt. Dieses instabile Signal ist in der natürlichen Probe nicht vorhanden. Daraus resultiert, dass die absorbierte Paläodosis und somit das Alter der Probe unterschätzt werden.

whereas the natural signal from the same trap will derive only from the stable charge. The same artificial radiation dose will produce a much higher signal than that resulting from natural irradiation and, as a consequence, the palaeodose will be underestimated as will the age of the sample (Fig. 19).

Preheating the samples prior to measurement (section 5.3) will remove most of the unstable charge, but several studies have shown that this unwanted charge may remain even after applying rigorous thermal pre-treatments. One possible fading mechanism that has been suggested is attributed to the tunnelling of electrons (e.g., VISOCEKAS 1985), whereby charge tunnels between traps and luminescence centres rather than recombining via the conduction band. While the phenomenon is normally only attributed to feldspars, recent publications imply that it may also exist in volcanic quartz (BONDE et al. 2001; TSUKAMOTO et al. 2007). With regard to feldspar, there is much debate over whether fading affects this mineral ubiqui-

tously (e.g., HUNTLEY & LAMOTHE 2001) or, if it is related to feldspar of specific geological origin and, at least partly, inappropriate laboratory procedures. With regard to the latter, two issues are of major relevance. Firstly, the thermal pre-treatment must be long enough, and the temperature high enough to remove thermally unstable components. Secondly, KRBETSCHKEK et al. (1997) have shown that unstable components in feldspars are mainly related to emissions in the UV band, and recommend that these emissions should be avoided, suggesting either the use of short-wave blocking optical filters (e.g., a Schott GG400) or interference filters that only allow transmission of emissions in a very narrow band. While studies implementing these recommendations often produce ages consistent with independent age control (e.g., CLARKE & RENDELL 2003; PREUSSER 2003), those studies incorporating the UV emissions have reported significant underestimation of luminescence ages (e.g., HUNTLEY & LAMOTHE 2001). Nevertheless, further studies utilising a restricted detection window have still observed fading of feldspars in their samples (e.g., WALLINGA et al. 2007), and this may be related to the measurement of feldspars of volcanic origin, which have been shown to be predominantly affected by fading, and may be due to the disordered crystal lattice in volcanic minerals resulting from rapid cooling and crystallisation (SPOONER 1994). This may also explain the fading observed in volcanic quartz.

Storage tests performed at either room or elevated temperature indicate whether a sample is affected by fading (AITKEN 1985; SPOONER 1992), and are a suitable approach as it is expected that fading components decay on a logarithmic scale (VISOCEKAS 1985, 2002; SPOONER 1994). Where fading is observed in samples it is possible to calculate the rate of fading per decade and use it to correct the age (HUNTLEY & LAMOTHE 2001; AUCLAIR et al. 2003; LAMOTHE et al. 2003). LAMOTHE et al. (2003) undertook fading correction for samples both on the linear part of the dose response curve and for geologically old sediments in luminescence field saturation. However, a recent study by WALLINGA et al. (2007) dem-

onstrated that the applied fading correction was not sufficient to account for the offset between feldspar and quartz ages. More research in this area is necessary to fully utilise the potential of feldspars in dating; in particular the higher saturation dose and hence increased dating range.

6.4 Accurate dose rate determination

Besides the problems associated with the determination of the dose absorbed during burial (palaeodose) there are also potential errors related to the correct calculation of dose rate, some of the most important of which are discussed in the following.

6.4.1 Alpha efficiency

Alpha particles produce much less luminescence signal compared to beta and gamma rays, and so the effective dose from alpha irradiation is therefore multiplied by a factor that reflects this lower ionisation efficiency; this factor, referred to as the a -value, is usually < 0.1 (AITKEN & BOWMAN 1975).

For sand-size minerals, for which most research has concentrated during the last decade, a -values are of minor importance as alpha particles penetrate only the outer ca. $10 \mu\text{m}$ of the grain, and the outer rim of such grains is usually removed by HF etching during sample preparation. When measuring fine-silt grains, for example when dating loess, lacustrine sediments or fine silicate inclusions from ceramics, the a -value is of major importance. For such materials it is recommended that the a -value is determined for each individual sample, which involves comparison of laboratory-induced luminescence due to both alpha and beta radiation (e.g. AITKEN 1985).

6.4.2 Internal K-content

With regard to the internal dose rate of feldspar, there is the potential to underestimate the K-concentration where chemical analyses (e.g. AAS) or beta counting are used, and is due to the fact that potassium content lower than the

stoichiometric maximum can be caused by contamination of the sample by, for example, quartz that is often found in the feldspar fraction, but is not relevant for palaeodose determination. An extended chemical analysis (e.g. K, Na, Ca, Al, Si concentration), the use of micro-probe equipment and the calculation of a theoretical feldspar composition may overcome this problem to some extent (DÜTSCH & KRBETSCHKE 1997).

6.4.3 Sediment moisture

Another major uncertainty is associated with the moisture content of a sample during burial. Water in the sediment pores absorbs much more radiation than air and so the effective dose acting on a grain is higher in sediments with low moisture content compared to a similar saturated sediment (Fig. 20). In age calculation, an average value for moisture over the whole burial time is usually assumed, but the lack of information concerning the extent of moisture variation throughout the burial period means that this is often the greatest source of uncertainty in a luminescence age. The present moisture content of the sample is often used as a guideline for age calculation but it is crucial that potential changes in the hydrological conditions are carefully considered during sampling. It is often also

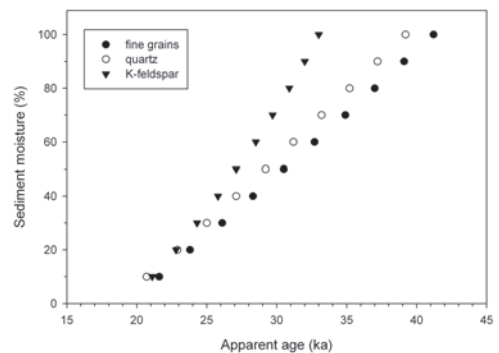


Fig. 20: Impact of sediment moisture on calculated luminescence age.

Abb. 20: Einfluss der Sedimentfeuchte auf das berechnete Lumineszenzalter.

appropriate to measure water up-take capability of samples to give an estimate of maximum moisture content (cf., AITKEN 1998). For example, fluvial sediments are usually water saturated directly after burial but the height of the water table may change when rivers incise or terraces are tectonically up-lifted. In such cases the sediment may be above the water table for a significant part of the burial period and pores may be only partially filled with water. In lacustrine settings, pore space will decrease with on-going consolidation of the sediment as discussed in the context of luminescence dating by JUSCHUS et al. (2007). Uncertainties related to sediment moisture are most important for water-lain sediments but can in principle also be significant in certain other sedimentary settings such as coastal deposits or sediments that have been affected by permafrost in the past.

6.4.4 Cosmic dose rate

The contribution of cosmic radiation to the total dose rate is usually calculated according to the sampling depth and geographical position of the sampling site following PRESCOTT & HUTTON (1994). The dynamic character of sedimentary systems with repeated events of erosion and accumulation results in variation of the thickness of the overburden above a buried sediment sample. Therefore, the contribution of cosmic rays to the total dose rate of the sample is not constant over the entire period of burial time, but may vary significantly. Such fluctuation in the overburden thickness is less significant in sedimentary environments where the contribution of cosmic rays to the total dose is generally low. Frequently the proportion measures less than 10 %. However in quartz-rich aeolian dune sand system the dose rate tends to be low and the cosmic ray contribution to the annual dose can be a major component of the total dose rate. In such cases a substantial error in total dose rate calculation and, hence, luminescence age can be introduced if the cosmic dose contribution is calculated using present sampling depth. To provide a more ac-

curate dose rate assessment the burial history of a sample should be taken into account, as far as this can be reconstructed (MUNYIKWA 2000).

6.4.5 Radioactive disequilibria

Special attention must be directed to sediments exhibiting radioactive disequilibria since the prerequisite of constant radionuclide concentrations does not hold (cf., KRBETSCHKEK et al. 1994). The radioactive elements of uranium and thorium are the beginning members or 'parents' of three decay series starting with the isotopes ^{238}U , ^{235}U and ^{232}Th . Their half-lives are of the order of 10^9 to 10^{10} years and thus they are still present in nature. These parent radionuclides and their stable end 'daughter' products of ^{206}Pb , ^{207}Pb and ^{208}Pb , respectively, are linked together by decay chains including several daughter isotopes of various elements but with half-life times much shorter than 10^9 years. Table 2 gives the physical and chemical properties of the most significant members of ^{238}U and ^{232}Th decay series in dose-rate determination.

In an undisturbed geological situation the activities within each decay series are in 'radioactive equilibrium', characterised by equal activities of all involved nuclides. Physical and chemical differentiation processes lead to a loss of this balance and to radioactive disequilibria. In closed systems the balance is restored on a time scale of the order of ca. five half-lives of the radionuclide concerned. Open systems can reach stationary states if geochemical properties do not change with time. As a consequence of the time dependent change in activities in the case of radioactive disequilibria, the external dose rate also becomes a function that varies with time. Equation (1) must then be replaced by an iteration technique which searches for the point in time t , at which equation (2) holds:

$$\int_{t_0}^{t_0+t} \dot{D}(\tau) d\tau = D \quad (2)$$

where t_0 is the moment of sample bleaching. In the case of radioactive disequilibria one needs: (a) preferably an analysis of all long-lived ra-

Table 2: Physical and chemical properties of the dosimetrically most significant isotopes of ^{238}U and ^{232}Th decay series.Tab. 2: Physikalische und chemische Eigenschaften der dosimetrisch wichtigsten Isotope der ^{238}U und ^{232}Th Zerfallsreihen.

Decay series	Nuclide	Half life	Significant Properties
^{238}U series	^{238}U	$4.5 \cdot 10^9$ a	Actinide, heavy metal, forms organic complexes, valence states U(IV) (low solubility) and U(VI) (soluble as uranyl ion (UO_2) $^{2+}$)
	^{234}U	$2.5 \cdot 10^5$ a	
	^{230}Th	$7.5 \cdot 10^4$ a	Actinide, tetravalent, low solubility
	^{226}Ra	$1.6 \cdot 10^3$ a	Earth alkali metal, soluble
	^{222}Rn	3.8 d	Inert gas
	^{210}Pb	22 a	Heavy metal, forms organic complexes
^{232}Th series	^{232}Th	$1.4 \cdot 10^{10}$ a	cf. ^{230}Th
	^{228}Ra	5.8 a	cf. ^{226}Ra
	^{228}Th	1.9 a	cf. ^{230}Th

dionuclides; and (b) a model of the possible activity evolutions in the past in agreement with both the geochemical parameters of the system and with the modern radionuclide contents.

Low-level high-resolution gamma spectrometry is suitable for multi-nuclide analyses, mostly fulfilling criterium (a). Generally, this method is suitable for determination of the following long-lived nuclides (in some cases with the use of short-lived daughter nuclides): ^{238}U , (^{230}Th), ^{226}Ra , ^{210}Pb from ^{238}U series, ^{228}Ra , ^{228}Th from ^{232}Th series and ^{40}K .

The time evolution of radioactive disequilibria in closed systems and in special types of open systems can be analytically solved. Mathematically, the solution for each activity term is a superposition of exponential decays with coefficients containing the initial activities, the decay constants and exchange parameters in open systems (DEGERING & KRBETSCHKE 2007).

Modelling the disequilibrium in the past is based upon assumptions concerning the value of the prevailing parameters and their variability with time. In most cases this results in a more or less broad range of possible time evolutions, leading to the current radionuclide concentrations. In comparison to ages ignoring

disequilibria, the uncertainty in age increases but so too does reliability.

In recent years several samples exhibiting radioactive disequilibria have been accurately dated when compared to independent age control from radiocarbon dating. From these investigations some indications of “suspicious” systems and their treatment can be derived:

- Organic substances tend to accumulate complex-forming elements like uranium or lead. Since the solubility of U and Th are very different, organic sediments like peat often show strong U/Th imbalance. The most important is $^{234}\text{U}/^{230}\text{Th}$ disequilibrium and is definitely predictable for closed systems (DEGERING & KRBETSCHKE 2007).
- Changes in the moisture content or in the groundwater level may lead to variable redox conditions and hence changes between open and closed system characteristics. Especially for fine grain dating, the dosimetric consequence of variable moisture is a variable attenuation of the external irradiation with time. The resulting enhanced uncertainty in the dating result can only be reduced by applying a model considering this variation (PREUSSER & DEGERING 2007).

– Calcareous material deposits containing, for example, mollusc shells or corals are able to exchange radionuclides with their environment. Reasonable explanations of the modern radionuclide contents partly require the assumption of an open system, mainly connected with uranium uptake after deposition. The clear reconstruction of the evolution of disequilibrium in the past is hardly practical here. A careful investigation should include the calculation of borderline cases to give an impression of the likely span of reliable ages (ZANDER et al. 2007).

7 Suitable materials

Up until the 1980s most research in luminescence dating had focused on fundamental problems and the application of TL for dating heated objects and volcanic deposits as well as some initial work on TL dating of sediments (cf., AITKEN 1985). Since the introduction of optical dating (using IR and visible light) most research has focused on the dating of sediments, in particular in the context of palaeoclimate change as well as archaeology (Table 3, 4). General overviews on early work are provided by PRESCOTT & ROBERTSON (1997), AITKEN (1998) or STOKES (1999) and the following sections will concentrate on benchmark publications and the most recent developments.

7.1 Archaeological materials

A wealth of archaeological materials can be dated with luminescence methods. Apart from unheated sediments (see section 7.3) containing archaeological remains, two main categories can be distinguished: (1) heated sediment e.g. ceramics, pottery, bricks, fire places, fire pits, hearths, kilns; and (2) heated rocks e.g. flint, chert, quartzite, quartz, silcrete, sandstone and other rock materials. An additional category includes man made materials such as slag and walls constructed from rock materials.

The association of dated material and human occupation is always a key question when applying chronometric dating methods in es-

tablishing the age of archaeological sites or, in other words, the relationship between the event dated and the question asked has to be clearly shown. However, it is often only through luminescence on heated materials that an age of an archaeological event can directly be provided (cf., RICHTER 2007; ALPERSON-AFIL et al. 2007 for discussion of natural fires).

Table 3 lists the commonly used materials/samples in luminescence dating, together with the appropriate method and the event dated, which is sometimes not evident at first sight. In some cases it is important to take the life time of an artefact or feature into consideration, e.g. a kiln could have been used for an extended period of time, but in general only its last use can be dated and not the time of construction. Or in the case of dating rock surfaces, the time since the last exposure to light is dated, which can be the time of construction or destruction of a monument. Ceramics could be much younger than an associated construction or much older, e.g. when they are discarded in a pit or trench from older deposits and are therefore out of context. The depositional age of sediment infill provides age information concerning abandonment rather than construction, e.g. of a trench or pit. Anthropogenic sediments or sediments altered by human occupation are difficult to date with luminescence methods. It is therefore often advisable to select more suitable sedimentological units bracketing the archaeological layer of interest and thus OSL dating can provide *termini post quem* and *ante quem* age estimates.

Heated rocks are usually dated by TL but if the material is sufficiently sensitive OSL methods can be applied as well. In such cases, bleaching during excavation might be a major concern (e.g., TRIBOLO 2003), although flint, for example, has been shown to be insensitive to optical stimulation (POOLTON et al. 1995). Conversely, TL of flint is very hard to bleach by light for most samples from a highly diverse material group (RICHTER et al. 1999).

The first luminescence ages on archaeological material were obtained using TL on ceramics by GRÖGLER et al. (1958). TL dating of ceram-

Table 3: Overview of different dating applications for archaeological materials by luminescence methods.

Tab. 3: Überblick über die verschiedenen Anwendungsbereiche der Lumineszenzmethoden zur Datierung archäologischer Materialien.

Material	Material details	Method	Dated event	Interpreted as time of	Archaeological example
Brick	Quartz, feldspar	TL/OSL	Last firing	Manufacture	BAILIFF & HOLLAND (2000)
Brick surface	Quartz, feldspar	OSL	Last exposure	Construction; repair; destruction	BAILIFF & HOLLAND (2000)
Ceramic, pottery, tile	Quartz, feldspar	TL/OSL	Last firing	Manufacture; authenticity	BARNETT (2000)
Daub (burnt)	Quartz, feldspar	TL/OSL	Last heating	Destruction	QUICKERT et al. (2003)
fFigurine	Quartz, feldspar	TL/OSL	Last heating	Manufacture; authenticity	ZINK & PORTO (2005)
Hearth stone	Sandstone, limestone, granite (quartz, feldspar)	TL/OSL	Last heating	Last use	ICHIKAWA & NAGATOMO (1978)
Kiln	Quartz, feldspar	TL	Last firing	Last use	HONG et al. (2001)
Lithic artefact (heated)	Flint, chert, quartzite, quartz, silerete	TL	Last heating	Discard	RICHTER et al. (2007)
Limestone (heated)	Calcite, quartz	TL	Last heating	Last use	ROOUE et al. (2001)
Mortar	Quartz, feldspar	TL/OSL	Last light exposure	Building	GOEDICKE (2003)
Oven	Quartz, feldspar	TL	Last firing	Last use	ROOUE et al. (2002)
Pit/trench infill	Quartz, feldspar	OSL	Last light exposure	Abandonment; infilling	LANG & WAGNER (1996)
Rock surface	Granite, marble, limestone (quartz, feldspar, calcite)	OSL/TL	Last light exposure	Construction; destruction	GREILICH et al. (2005, 2006); LIRITZIS & VAFIADOU (2005)
Sediment (burnt)	Quartz, feldspar	TL/OSL	Last heating	Last use	GODFREY-SMITH & SHALEV (2002)
Sediment, aeolian	Quartz, feldspar	OSL	Last light exposure	Deposition	JACOBS et al. (2003 a,b)
Sediment, colluvial	Quartz, feldspar	OSL	Last light exposure	Deposition	LANG & HÖNSCHIEDT (1999)
Sediment, fluvial	Quartz, feldspar	OSL	Last light exposure	Deposition	FOLZ et al. (2001)
Slag	Quartz	TL	Last firing	Last use	HAUSTEIN et al. (2003)
Wasp nest	Quartz, feldspar	OSL	Last light exposure	Building	YOSHIDA et al. (2003)

ics and pottery soon became the backbone for establishing many archaeological chronologies in the world, but with the increased precision of archaeological dating (including seriation etc.) and the possibility of providing calendar ages by calibrating radiocarbon data, the application of TL dating of ceramics and pottery became less important from the 1980s onward. However, BARNETT (2000) used TL to verify the typo-chronologies for the British Late Bronze and Iron Age, using both shards that were diagnostic by their shape and/or decoration, as well as those that were undiagnostic (fabric based chronology), and it was shown that the established chronology for diagnostic pottery was supported by the TL results. As the conditions for luminescence dating were not different for the diagnostic samples, it was concluded on the other hand that the accuracy of chronologies based on fabric only required refinement (BARNETT 2000).

Establishing the timing of construction, repair and destruction of parts of the medieval citadel of Termez in Usbekistan was attempted by VIELLEVIGNE et al. (2006), who showed the feasibility of a combined OSL and TL approach on different bricks, that could be related to the reuse of older bricks, as was also done by BAILIFF & HOLLAND (2000).

The correlation of features considered to be fire places, hearths or fire pits, which lie close to an archaeological settlement site but lack remains like hearth stones, is often difficult to establish. The heat-reddened rim and infill of a pit from an Early Bronze Age context at Ashkelon Marina in Israel was dated with TL additive methods and OSL by GODFREY-SMITH & SHALEV (2002). While the TL age for the heated sediment agrees well with radiocarbon data, the TL result for the pit infill is greatly overestimated and clearly shows that this method is inappropriate for unheated sediment. On the other hand an OSL age for the pit infill agrees well with the TL age of the heat-reddened rim, suggesting the pit was rapidly filled after disuse and confirming the likely function of the fire pits for early copper smelting technology.

TL-dating of domestic ovens for the Neolithic

site of Dikili Tash in Greece (ROQUE et al. 2002) provided confidence in the often assumed association of radiocarbon dating samples with occupational levels and strengthens the Neolithic chronological framework for that region. It is possible to establish the authenticity of figurines and ceramics using TL and OSL, although an accurate date cannot usually be given due to the lack of knowledge of the precise radiation field of each individual object. ZINK & PORTO (2005) investigated a large collection of Tanagra figurines and used probability statistics to distinguish fake from authentic ones.

Quartz pebbles from the site of Pedra Furada in Brazil were dated with TL techniques by VALLADAS et al. (2003). These pebbles are assumed to have been used as hearthstones and/or for heating liquids. The site of Pedra Furada is controversial because of the claim that it has a much greater antiquity than the time generally recognised for the arrival of humans in the Americas. While the TL age estimates prove the antiquity of the heating, the question of the anthropogenic use of the pebbles cannot be solved solely by establishing the time when fire occurred (VALLADAS et al. 2003). Sandstones are the most common material used to establish the age of distinct hearths but limestone was more frequently used to construct fireplaces in the Palaeolithic. The feasibility of using TL for dating heated limestone is reported by ROQUE et al. (2001) from the Upper Palaeolithic site of Combe Saunière in France.

TL dating of heated flint plays a major role in establishing the relationships in time between tool assemblages, technocomplexes and hominid species, under the assumption that the time difference between archaeological material surrounding the intrusive burial and skeletal material is negligible. Heated flints from layers containing Middle Palaeolithic artefacts in several caves in Israel which are associated with Neanderthal remains in some and with modern humans in others were dated (VALLADAS et al. 1987; 1988, 1999; MERCIER et al. 1993). These dates provide evidence for an extended overlap of several thousand years of the two species within a relatively small region. In general, TL

Table 4: Overview of different applications of luminescence methods for dating sediments (n.a. = not available).

Tab. 4: Überblick über verschiedene Anwendungsgebiete der Lumineszenz zur Datierung von Sedimenten.

Sediment type	Severity	Most common problem	Pioneering study	Review
Loess	Minor		TL: WINTLE (1981)	SINGHVI et al. (2001)
Aeolian sand	Minor		TL: SINGHVI et al. (1982)	SINGHVI et al. (2001)
Fluvial deposits	Average	Partial bleaching	TL: FORMAN et al. (1988) OSL: PERKINS & RHODES (1994)	WALLINGA (2002 a)
Hillslope sediments	Average	Partial bleaching	IRSL: WINTLE et al. (1993)	FUCHS & LANG (2008)
Lacustrine sediments	Average	Sediment moisture	TL: BERGER (1990)	n.a.
Proglacial deposits	Challenging	Partial bleaching	TL: GEMMELL (1985) IRSL: HÜTT & JUNGNER (1992)	n.a.
Coastal deposits	Average	Radioactive disequilibria	TL: BAILESCU et al. (1991)	n.a.
Deep sea sediments	Average	Radioactive disequilibria	TL: WINTLE & HUNTLEY (1979)	n.a.

methods are used to establish the age of individual archaeological layers and thus help to establish the chronologies of technocomplexes beyond the range of radiocarbon dating, e.g. for the middle Palaeolithic chronology of Western Europe (RICHTER et al. 2007). Recently, a SAR protocol employing red TL was developed, which allows the dating of small samples of heated flint, thus increasing the number of Palaeolithic sites, which could be dated (RICHTER & KRBETSCHKE 2006). Mastering fire is regarded as one of the hallmarks in human evolution and certainly played an important role in dramatic changes in human behaviour connected with diet, defence and social interaction. Analysis of lithics suspected of having been heated using TL helped provide evidence for the controlled use of fire at the Acheulian site of Gesher Benot Ya'aqov in Israel, where distinct clustering of heated and non-heated materials reveal the positions of former fire places (ALPERSON-AFIL et al. 2007). TL dating of heated flint can also be used to verify the integrity of an archaeological site or layer. Results of heated flint from an assemblage dated by DEBENHAM (1994) provided

evidence of an otherwise unobserved severe mixing of a Palaeolithic site.

Slags are often the only remains from archaeometallurgical activities and the association of dates obtained from materials in the vicinity is often questioned. The application of a combination of the red TL with a SAR protocol on extracted quartz by HAUSTEIN et al. (2003) showed a fair agreement in comparison with independent age control.

Determining the age when a wall was built, a megalith was erected, repair work or destruction took place, or even when rocks were moved and discarded or reused is of great importance in archaeology. The recent development by GREILICH et al. (2005, 2006) on the OSL dating of surface exposure is a significant development. These authors were able to establish surface exposure ages of granitic surfaces from rocks used to construct some of the Nasca geoglyphs. These results were contrasted by the surface exposure age of rocks which were unmoved for several 10^3 years. But even standard TL and OSL methods are capable of producing reasonable age estimates of surface exposure of

Neolithic as well as Greek monuments (LIRITZIS & VAFIADOU 2005).

7.2 Volcanic deposits

The dating of volcanic deposits was developed in the early 1970s, soon after the successful application of TL to date archaeological materials. Despite the fact that anomalous fading appears to affect most volcanic feldspar (WINTLE 1973), and possibly volcanic quartz as well, (TSUKAMOTO et al. 2007) more than 50 studies have been published on the dating volcanic deposits. A comprehensive review on studies carried out in the 1970-90s is provided by FATTAHI & STOKES (2003b). TL dates have been used to establish chronological frameworks for volcanic activity in areas such as the Massif Central, France (VERNET et al. 1998) and China (LI & YIN 2001). While FATTAHI & STOKES (2003b) summarise good consistency of TL ages with independent age control for several dating studies, problems with anomalous fading occurred in many other studies. An important aspect is the use of the red emission from feldspar, which has proven to be more stable than other TL emissions (e.g. FATTAHI & STOKES 2000; MIALLIER et al. 2004; GANZAWA et al. 2005; BASSINET et al. 2006). However, relatively little research has been carried out and to date only a few research groups have focused on this topic.

7.3 Sediments

7.3.1 Loess

Loess was among the first sediments that were systematically dated by luminescence methods, in particular by TL (e.g., WINTLE 1981; WINTLE 1990). In most early dating studies, the optically insensitive TL signal was determined by laboratory bleaching using UV lamps (or by using daylight) and used to define the zero point of the dose response curve (WINTLE & HUNTLEY 1979). This total bleach approach assumes that all light-sensitive TL is removed from the grains during transportation and this

has been investigated by plotting palaeodose versus TL-temperature for the samples investigated (the so-called plateau test). It has been demonstrated that partial bleaching is of minor importance for loess that has been deposited by aeolian processes but can be relevant for re-worked loess.

A frequent problem has been a systematic underestimation of TL (e.g., ZHOU et al. 1995), the reason for which is not fully understood. This may be related in part to the use of UV filters for luminescence detection, which allow an unstable UV emission from feldspathic minerals to be transmitted. However, as many other studies have provided dates consistent with independent age control and geological time models it also became evident that the application of luminescence (both TL and optical stimulation) is apparently restricted to the last glacial cycle (i.e. the last 130 ka). A comprehensive review of the work carried out in the 1980-90s is provided by SINGHVI et al. (2001). More recently, single-aliquot methodology, in particular for quartz, has been applied to loess to improve both the precision and reliability of the chronology as well as to extend the datable age range. ZANDER et al. (2000) compared single-aliquot sand-size quartz OSL and multiple-aliquot silt-size polymineral IRSL and TL on samples from a site in Czech Republic. They found good consistency for the different methods but higher precision of the quartz ages. WATANUKI et al. (2005) dated a loess sequence in Japan applying the SAR methodology on quartz and polymineral fine-grain extracts. They produced reliable quartz ages as old as 500 ka with independent age control from tephra chronology. Polymineral fine-grain ages, which have been measured using the UV emission band, are underestimated by 10-20 % in this study. A similar approach has been tested by WATANUKI et al. (2003) and WANG et al. (2006a) on Chinese loess and these authors also identified a systematic underestimation of polymineral ages using UV emissions. A comprehensive summary of previous work on luminescence dating of Chinese loess is provided by STEVENS et al. (2007). These authors

also conclude, using high resolution sampling for OSL dating, that loess accumulation has been episodic and not continuous during the last glacial cycle, in contrast to many previous assumptions. BUYLAERT *et al.* (2007) concluded that the SAR procedures used in their studies do not allow dating beyond the Last Interglacial, and yielded a systematic underestimation of quartz OSL ages, for example, an OSL age of 112 ± 7 ka and fading corrected IRSL age of 301 ± 30 ka for loess from below the Brunhes/Matuyama (B/M) boundary.

Present work is focusing on extending the dating range in Chinese loess by developing new measuring techniques such as red TL from quartz (LAI & MURRAY 2006; LAI *et al.* 2006) and thermally-transferred OSL (WANG *et al.* 2007). The latter in particular looks rather promising, with WANG *et al.* (2006b) reporting a mean TT-OSL age of 771 ± 15 ka for loess from the B/M boundary.

7.3.2 Aeolian sand

Due to the aeolian transport processes of saltation, raptation (creep) and suspension the sediment grains are most likely exposed to sufficient light for optical re-setting of the luminescence signal prior to burial, and so poor bleaching is rarely a problem in aeolian materials (DULLER 2004). As aeolian environments provide ideal conditions for the application of luminescence dating, a vast number of dating studies on dune sands has been carried out during the past decades since SINGHVI *et al.* (1982) published the first study on aeolian sand dunes from Rajasthan, India. Since then, luminescence dating has been used to constrain the chronology of aeolian sands in a variety of drylands such as the Kalahari (STOKES *et al.* 1997a; TELFER & THOMAS 2007), Namib (BRISTOW *et al.* 2005, 2007), Sahara (LANCASTER *et al.* 2002; BUBENZER *et al.* 2007), Sahel (STOKES *et al.* 2004), Arabian (PREUSSER *et al.* 2002; STOKES & BRAY 2005), Taklamakan (YANG *et al.* 2006) and Australian (GARDNER *et al.* 1987; NANSON *et al.* 1992; TWIDALE *et al.* 2001; LOMAX *et al.* 2003; FITZSIMMONS *et al.* 2007;) deserts.

Furthermore, several studies have focussed on aeolian sand deposits in former periglacial regions such as those in the European sand belt (DIJKMANS *et al.* 1992; BATEMAN 1998; BATEMAN & VAN HUISSTEDEN 1999; HILGERS *et al.* 2001; VANDENBERGHE *et al.* 2004; HILGERS 2007; KASSE *et al.* 2007), the Pampa of Argentina (TRIPALDI & FORMAN 2007) and in North America (STOKES *et al.* 1997b; FEATHERS *et al.* 2006; FORMAN *et al.* 2006).

Compared to water-lain deposits luminescence dating of aeolian sand, especially considering the recent advances in methodology (e.g. SAR protocol), is relatively straightforward as partial bleaching is not usually a major concern for such sediments. However, aeolian deposits play a major role in understanding the complexity of dose distributions and hence in improving the accuracy and precision of luminescence dating (BATEMAN *et al.* 2003; VANDENBERGHE *et al.* 2003; LOMAX *et al.* 2007; see Section 6.4 for details). Aeolian deposits are also important in studies that have explored the possibilities of extending the minimum and maximum age limits. For example, BALLARINI *et al.* (2007) successfully explored the potential to date dune sand as young as 10 a. Some of the oldest luminescence ages (in this case TL) ever published have been determined for stranded beach dunes from SE Australia that yielded ages as old as 800 ka (HUNTLEY *et al.* 1993; HUNTLEY & PRESCOTT 2001). More recently, RHODES *et al.* (2006) provided OSL ages of about 500 ka for aeolian deposits from Morocco and a single age of 1 Ma, the latter being from inversely magnetised sediments attributed to the Matuyama chron. These latter two examples are, however, exceptional due to the low dose rate (< 1 mGy a⁻¹) of the investigated sediments, and saturation of the luminescence signal will be reached earlier at higher dose rates.

Dunes are complex and composite features formed during several events of deposition which may have been interrupted by episodes of erosion, and as such represent inherently discontinuous archives, which complicate the interpretation of OSL records in terms of their significance for palaeoclimate or pal-

aeo-environmental reconstructions. Thus, any interpretation of OSL data has to be done in the context of the complete environmental system, including the effective geomorphological processes (e.g., BUBENZER et al. 2007). Various approaches are taken to minimise this problem and to optimise the value of luminescence dating results. The necessity of an increase of samples analysed per dune as well as the investigation of several dunes within a dune field to obtain not only detailed but informative records of aeolian processes in a certain area have been demonstrated for example by TELFER & THOMAS (2007) and HILGERS (2007). The combination of ground-penetrating radar (GPR) and OSL dating provides a powerful tool to reveal the dynamic history of dunes. Based on the GPR profiles of the internal structure of the dunes sampling locations for OSL dating can be determined (e.g., BRISTOW et al. 2007).

7.3.3 Fluvial deposits

Most of the early studies using TL for dating fluvial and other water-lain sediments revealed age overestimations due to incomplete bleaching of the signal prior to deposition (e.g., FORMAN et al. 1988). With the advent of OSL methodology the dating of waterlain sediments was reconsidered, but took nearly a decade from the breakthrough publication of HUNTLEY et al. (1985) to the first systematic studies on dating waterlain deposits. For example, PERKINS & RHODES (1994) used multiple-aliquot methodology to date sandy River Thames sediments and achieved a good consistency with independent age control. Nevertheless, up to the late 1990s fluvial and in particular glaciofluvial sediments (see section 7.3.6) have been considered rather challenging and the reliability of optical dating of such deposits has been considered questionable at least.

However, it has been the dating of fluvial deposits that led to a major improvement in luminescence methodology with the development of single aliquot techniques (see section 5.3.2). One of the pioneering studies was that of MURRAY et al. (1995) who investigated sub-modern fluvial sediments from Australia using

a modified version of the so-called SARA protocol (MEJDAHL & BØTTER-JENSEN 1994), a technique that is only infrequently used nowadays. The study by MURRAY et al. (1995) was the first in which a large number (>100) of individual palaeodose values were calculated for individual samples. As already indicated by DULLER (1994), it was demonstrated that samples not fully zeroed consist of a mixture of grains having a different degree of bleaching, including grains that have been completely bleached at deposition. Hence, the scatter of several individual palaeodose values will be broad and positively skewed for incompletely bleached samples, and tight and normally distributed for well-bleached samples (cf., OLLEY et al. 1998, 1999). A comprehensive review on the problems and potential of optical dating of fluvial deposits has been provided by WALLINGA (2002a) and the following discussion will concentrate on a few aspects only.

As a major concern has always been the degree of resetting of the optical signal prior to deposition, several studies have investigated the resetting of modern analogues. For example, STOKES et al. (2001) observed a monotonic decrease of apparent palaeodose in modern deposits of the River Loire downstream from the source, with values close to zero for transport distances of more than 300 km. Consequently, STOKES et al. (2001) concluded that the resetting of the OSL signal in a large drainage basin is much more likely than considered from many earlier studies. This assertion was later confirmed by SINGARAYER et al. (2005) who identified different resetting for individual components of the OSL signal. FUCHS et al. (2005) investigated the degree of zeroing in deposits of the 2002 millennium flood in Saxony and compared quartz and feldspar signals. These authors observed residual ages of 0.14–0.45 ka for silt-size quartz and 1.12–4.00 ka for polymineral fine-grains for samples from the River Elbe. For the River Rote Weißeritz, where sand-size grains have been investigated, residual ages of 0.36–0.83 ka for quartz and 2.12–4.67 ka for feldspar have been calculated. Similar low residual quartz OSL ages are reported for mod-

ern deposits of River Ahr in western Germany (CHOI et al. 2007). On the other hand, FIEBIG & PREUSSER (2007) observed rather high residual ages in some of the young deposits of the River Danube, parts of which were deposited by the floods in 2002. For some of these samples residual ages are as high as several 10 ka. However, these exceptional values are probably the result of an artificial input of sediment to prevent riverbed erosion.

During the last decade OSL dating has been applied to the reconstruction of the fluvial history in several regions, both in large drainage systems such as River Rhine (e.g., WALLINGA et al. 2004, 2007; BUSSCHERS et al. 2005; SCHOKKER et al. 2005), Loire (Colls et al. 2001) and Mississippi (RITTENOUR et al. 2005) as well as small scale catchments, for example, in Lincolnshire, England (BRIANT et al. 2004) or the Klip River, South Africa (RODNIGHT et al. 2006). Besides work in the middle latitudes, luminescence dating has also frequently been applied to date fluvial dynamics in the semi-arid subtropics in regions such as the Chad Basin (GUMNIOR & PREUSSER 2007), Death Valley (SOHN et al. 2007) and, in particular, on the Indian Subcontinent (e.g., JUYAL et al. 2000; JAIN & TANDON 2003).

7.3.4 Hillslope deposits

The term hillslope deposits describes sediments that are eroded from and transported along hillslopes by running water or gravity and that usually form wedge-shaped deposits on the foot-slope. Such deposits are often referred to as colluvium although the term is inconsistently used by different researchers. First successful studies on dating colluvial samples were carried out by FORMAN et al. (1988) on colluvium from the US using TL. Later, WINTLE et al. (1993) and BOTHA et al. (1994) applied IRSL dating to sediments from South Africa. Detailed reviews on dating hillslope deposits are provided by LANG et al. (1999) and FUCHS & LANG (2008).

The major limitation in dating hillslope deposits is incomplete bleaching of the luminescence signal, due to the fact that several processes are

involved in the formation of hillslope deposits such as soil erosion (ploughing, sheet and rill erosion), mass movements (sliding, falling) and soil creep (e.g. solifluction). Many of these processes cause downslope movement of sediment as a rather compact block such as in the case of sliding. Hence, with respect to daylight bleaching, only a small fraction of the sediment in the colluviation process (i.e. only the outer layer of sediment grain on a rigid block) will be exposed to daylight. If material is transported by water (e.g., sheet erosion), the intensity and spectral composition of the daylight is subdued by the usually high suspended load (DITLEFSEN 1992). Another problem is related to coagulation of grains that hampers sufficient zeroing of the luminescence signal, because the inner grains of an aggregate are shielded from daylight exposure (LANG & WAGNER 1996). Furthermore, due to the usually rather small catchments of colluvial systems sediment transport distance tends to be relatively small, decreasing the probability of efficient bleaching as the duration of light exposure will be relatively short.

Despite these shortcomings, there are many examples of successful luminescence dating of colluvium, which might be explained by two controlling factors. Firstly, hillslope deposits are usually repeatedly reworked, which increases the probability of daylight exposure. Secondly, bioturbation and mechanical processes in the soil as well as cultivation ensure that mineral grains are frequently exposed to daylight before the sediment grains are eroded and transported. Nevertheless, investigating the degree of bleaching prior to deposition, as described in section 6.1, is a fundamental requirement in dating colluvial deposits.

Although luminescence dating of hillslope deposits is rather challenging, it has been applied to a variety of case studies worldwide. The first systematic study by WINTLE et al. (1993) and BOTHA et al. (1994) correlated sediments and soils with dryer or wetter climatic periods of the Late Quaternary in South Africa and describe in detail the complexity of slope evolution processes. Similar studies have been car-

ried out by, for example, CLARKE et al. (2003) in the UK and ERIKSSON et al. (2000) in Tanzania. Another application of dating hillslope deposits was in the context of palaeoseismicity research as vertical displacement along faults scarp leads to its erosional degradation. This potential was first explored by PORAT et al. (1996) on colluvial sediments associated with fault scarp activities for estimating seismic hazards. These authors established an earthquake chronology with four large earthquakes for the period 37 – 14 ka and five smaller earthquakes more recently. PORAT et al. (1997) confirmed the suitability of luminescence dating in palaeoseismic research but PORAT et al. (2001) found a slope-face dependency for the bleaching degree of colluvial sediments, with colluvial sediments from south-facing scarps being better bleached than colluvium derived from north-facing scarps. Studies in a similar context have been provided by LU et al. (2002) for China and FATTAHI et al. (2006) for Iran.

Increased sediment transport down-slope can also be induced by human activity, in particular during the Holocene. Widespread clearance of woodlands and associated soil erosion typically causes increased erosion along hillslopes, for example, when humans established permanent settlements and agriculture. Among the first studies was that by LANG (1994) who investigated colluvial sediments derived from a loess-covered landscape in southwest Germany, and this was followed by similar studies (e.g., KADEREIT et al. 2002). A frequency distribution of IRSL ages from the area revealed that increased colluviation coincides with phases of higher population density and show that the intensity of farming activities is the main trigger for soil erosion (LANG 2003). In a study in Greece, FUCHS et al. (2004) and FUCHS & WAGNER (2005) applied the SAR protocol to the quartz coarse grain fraction for reconstructing Holocene soil erosion and to elucidate the interaction of humans and environment since Neolithic times. Colluviation strongly fluctuated in the course of the Holocene, with a sharp increase during the Early Neolithic and the onset of agricultural activities. Further periods

of increased deposition of hillslope sediments are the Middle and Late Bronze Age, the Roman period and the period since the sixteenth century AD (FUCHS 2007). Similar studies have been carried out, for example, in Romania (KADEREIT et al. 2006) and in Belgium (ROMMENS et al. 2007)

7.3.5 Lacustrine sediments

Relatively few studies have been reported on attempts to date lacustrine sediments, although such deposits are important archives of past environmental change and hence relevant for reconstructing Quaternary climate history. Early work by KRONBORG (1983) and BERGER (1990) studied the amount of resetting of TL in modern sediments and on dating deposits of known age. Both studies have highlighted the potential as well as the limitations of TL. With the introduction of OSL new dimensions were added to the dating of water-lain sediment due to the higher bleachability of the optical signal (HUNTLEY et al. 1985). DITLEFSEN (1992) experimentally investigated the bleaching of sediment in suspension demonstrating that the optical signal is almost completely bleached in dilute suspensions within a few hours, little bleaching has been observed in dense suspensions. KRAUSE et al. (1997) used IRSL to date lake deposits from Antarctica and compared four different emission bands (280, 330, 410, 560 nm), with the inconsistent results of palaeodose (D_e) measurements being attributed to different bleaching characteristics of the different emission bands.

Late Glacial to Holocene sediments from Lake Holzmaar, Germany, gave IRSL ages consistent with varve chronology for samples with low concentrations of biogenic material. Inaccurate ages obtained for organic rich samples are explained by problems with dose rate determination (LANG & ZOLITSCHKA 2001). THOMAS et al. (2003) applied different approaches to date lacustrine deposits from Greece revealing an underestimation of polymineral fine grain IRSL and post-IR OSL UV emissions compared to quartz OSL ages and radiocarbon chronology.

It was demonstrated that the underestimation of IRSL ages was due to anomalous fading that was observed in storage tests. However, the presence of fading is probably explained by using IRSL UV emissions or by a specific geological setting at that particular site since other studies on dating lacustrine sediments using blue emissions have provided correct age estimates (DORAN et al. 1999; Wolfe et al. 2000; BERGER & DORAN 2001; BERGER et al. 2004). VANDERGOES et al. (2005) dated a 150 000 year old lake record from Okarito Pakihi, New Zealand and got IRSL and post-IR OSL ages in agreement with the results of radiocarbon dating, tephra chronology and the chronological time frame deduced from comparison with pollen records from ocean sediment. JUSCHUS et al. (2007) found SAR IRSL ages consistent with the age model proposed from several proxies tuned to regional insolation.

While all the examples given above are from humid regions in the middle to high latitudes, a few studies have used luminescence methodology to date ancient lake deposits from arid zones. The particular advantages of luminescence are that it circumvents contamination problems that often affect radiocarbon in arid environments and extends the dating range beyond the few 10 ka possible with radiocarbon. However, only very few studies have utilised this potential so far, in particular RADIES et al. (2005) who dated lake deposits of the Early Holocene humid period in Oman and ARMITAGE et al. (2007) who demonstrated that a lake occupied the Fazzan Basin, Libyan Sahara, during the Early Holocene, the Last Interglacial and probably during Marine Isotope Stage (MIS) 11.

7.3.6 Proglacial deposits

Some of the first attempts to date glaciofluvial deposits were carried out by GEMMELL (1985, 1994) who investigated the resetting of the luminescence signal in modern samples. MEJDAHL (1991) compared TL and radiocarbon ages of Late Glacial sediments applying different bleaching techniques. BERGER & EAST-

ERBROOK (1993) and BERGER & EYLES (1994) dated glaciogenic and water-lain sediments from the Western Washington, British Columbia and Toronto area. HÜTT & JUNGNER (1992) compared TL and IRSL dating of Late Glacial sediments but their results were overestimated due to partial bleaching. DULLER (1994) investigated the bleaching characteristics of glaciofluvial feldspars and identified two different types of partial bleaching: one type showing grains that were incompletely bleached to the same amount (type A) and the other type showing a mixture of grains containing a majority of completely bleached grains, together with some insufficiently bleached grains (type B). He concluded that successful IRSL dating of glaciogenic sediments that were deposited only a few kilometres in front of the ice sheet is unlikely (DULLER et al. 1995). Further investigations compared the resetting of TL, IRSL and OSL signals in modern and ancient fluvial and glaciofluvial sediments from Switzerland and Northern Germany (PREUSSER 1999c). Dating results reveal that the sediments from Switzerland were completely bleached while those from Northern Germany were not, for most samples investigated. These results were additionally supported by further investigations of PREUSSER (2003) and PREUSSER et al. (2003, 2005a). Dating glaciofluvial deposits from New Zealand showed problems due to low luminescence sensitivity of the quartz grains (PREUSSER et al. 2005b, 2006). Beside the problem of insufficient resetting prior to deposition, which leads to age overestimation, the influence of 'thermal transfer' as reported by RHODES & BAILEY (1997) causes an overestimation of the expected age on the investigated sediments. OSL dating of glaciogenic deposits from Northern Eurasia were presented by LARSEN et al. (1999) and SVENDSEN et al. (2004) as well as FORMAN (1999), FORMAN et al. (1999, 2002) and MANGERUD et al. (2001) implicating glacier advances during the Late Saalian, the Early Weichselian, the Middle Weichselian and the Late Weichselian. Optical dating of proglacial and glaciofluvial sediments from inneralpine valleys and the Alpine Foreland provided dif-

ferent problems with low quartz luminescence sensitivity and partial bleaching (KLASEN et al. 2006, 2007). While samples from the inneralpine valleys can be assigned to bleaching type B (DULLER 1994), incompletely bleached samples from the Alpine foreland consist of grains being reset to the same amount and are therefore assigned to type A (DULLER 1994). For these samples, calculating a mean palaeodose is challenging, and the methods of OLLEY et al. (1998) and GALBRAITH et al. (1999) which extract individual palaeodose values out of a positively skewed distribution, failed due to the fact that distributions are Gaussian. This is supported by investigations of FUCHS et al. (2007) who reported that partially bleached samples can also be represented by normal distributions. Therefore, frequency distributions should be regarded very carefully when used for the interpretation of bleaching characteristics. Additionally, quartz samples from the northern Alpine Foreland appear not to contain any 'fast component' within the OSL-signal (cf., BULUR et al. 2000). Applying SAR- methodology to these samples resulted in an underestimation of the age of the sediment as this protocol is designed for samples dominated by a 'fast component' (KLASEN 2007). DULLER (2006) investigated glaciogenic deposits from Chile and Scotland using single grain optical dating, and showed that grains were commonly affected by low luminescence sensitivity. Nevertheless, DULLER (2006) concluded that the single grain methodology is the method of choice to detect partial bleaching. Despite this, PREUSSER et al. (2007) determined OSL ages consistent with radiocarbon ages for proglacial sediments from Switzerland using small aliquots.

7.3.7 Coastal (water-lain) deposits

The pioneering study on dating coastal deposits was carried out by BALESCU et al. (1991) on raised interglacial beaches from the Channel Region (France, Belgium). These authors used TL from feldspars detected in the UV band and found residual ages of no more than a few ka in modern deposits. However, TL ages of older

deposits were underestimated by up to 40 %. BALESCU & LAMOTHE (1992) later demonstrated that ages consistent with stratigraphy are obtained when blue TL emissions are used for dating. The same authors calculated a minimum age of 271 ± 36 ka for the marine Herzelee Formation in northern France that is interpreted as being of Holsteinian age (BALESCU & LAMOTHE 1993). The first applications of IRSL to coastal sediments reveal a good consistency with TL and independent age control (BALESCU & LAMOTHE 1994; BALESCU et al. 1997). In another early study, MAUZ et al. (1997) used TL from quartz separates to date littoral deposits from southern Italy revealing that eustatic oscillations deduced from a deep ocean sediment proxy are not completely recorded within the inner shelf deposits. MAUZ & HASLER (2000) found evidence from both feldspar IRSL and quartz TL dating for relatively high sea-level (-15 m) during early MIS 3 (ca. 50 ka ago) in the Mediterranean although this is inconsistent with that from the Huon Peninsular of New Guinea.

While the studies summarised above used multiple-aliquot approaches, more recent studies have used single-aliquot methodology. RICHARDSON (2001) investigated the degree of resetting in modern intertidal deposits from England and Wales revealing substantial residual ages in many of the samples, especially for those deposited by turbid water suspension. The author also highlights the potential problem associated with estimating the correct average water content during burial for samples from such environments. HONG et al. (2003), however, found clear evidence for complete bleaching of sub-modern tidal-flat deposits from Korea and report ages between 40-120 a. Excellent consistency of OSL and ^{210}Pb as well as ^{137}Cs dating of sub-modern estuarine sediments from Denmark has been shown by MADSEN et al. (2005, 2007) with OSL ages ranging from a few years to about 1000 a. The age of near-surface samples in one core is 9 ± 3 a and 7 ± 4 a in another, indicating that partial bleaching does not apparently have any major effect in such environments. The applicability

of OSL for dating young tidal flat deposits has also been demonstrated for the Frisian Coast, Germany, where age control is available from radiocarbon dating of peat (MAUZ & BUNGENSTOCK 2007). ZANDER et al. (2007) showed that Holocene coastal sediments from the Persian Gulf that are affected by radioactive disequilibrium in the Uranium decay chain due to high amounts of calcareous material (shell debris etc.) can still be correctly dated using OSL. MURRAY & FUNDER (2003) dated several samples from an Eemian site in Denmark to test the accuracy of OSL dating. Although the mean age of the individual samples is 119 ± 6 ka including systematic uncertainties and, hence, in acceptable agreement with the expected age of the sediment (128–132 ka), there is a suggestion that OSL may systematically underestimate the true age by about 10 % percent. This question has been addressed by MURRAY et al. (2007) who dated an Eemian site in northern Russia consisting of foreshore marine deposits with an expected age of about 130 ka. The mean of 16 OSL ages determined for this deposit was 112 ± 7 ka (including systematic uncertainties) showing a similar underestimation to that of the site in Denmark. The authors discuss several possible explanations for the apparent underestimation of OSL ages, among which are changes in sensitivity of the natural signal at the beginning of the SAR procedure, which is not corrected for, contamination of the stable fast component of the OSL signal by less-stable components as well as open system radionuclide behaviour and inaccurate estimates of long-term water content.

7.3.8 Deep sea sediments

One of the first applications of TL dating on sediments was on deep ocean deposits (WINTLE & HUNTLEY 1979, 1980; BERGER et al. 1984). More recently, STOKES et al. (2003) used SAR methodology on silt-size quartz gathered from two cores from the Arabian Sea where age control is provided by radiocarbon, tephrochronology and a correlative marine-proxy age model. The investigated silt is interpreted as dust input

from the Arabian Peninsula transferred by aeolian processes into the ocean. In this study, particular attention was paid to radioactive disequilibria that are a major potential error source in dating deep ocean sediments. The nine OSL ages obtained by STOKES et al. (2003) are, with one exception, in good agreement with independent age control and demonstrate the potential of OSL to date deep ocean sediments. OLLEY et al. (2004) applied sand-size single-grain quartz for dating a core off the NW Australian coast, and these authors also found good consistency of OSL with radiocarbon ages but highlight that partial bleaching may be important, even for grains that underwent long distance aeolian transport. BERGER (2006) carried out a test study on a variety of cores from the Arctic Ocean and found varying levels of signal resetting in different regions. Using a new technique enabled correct dates for the Late Holocene deposits to be established. This study demonstrated the potential but also highlighted the problems associated with dating samples from the Arctic Ocean, which are probably related to sediment input through glacial transport (e.g., ice raft). KORTEKAAS et al. (2007) dated a core covering the last 15 ka from the Arkona Basin, Baltic Sea, using fine-sand quartz (63–100 μm). These authors observed a good agreement between OSL and radiocarbon age from bivalve mollusc shells, although radiocarbon ages on bulk sediment samples were between 1000 and 3000 a higher than OSL and bivalve ages. The incorporation of reworked material in the bulk samples is suggested as the cause for overestimation.

8 Summary and outlook

Luminescence dating allows the dating of mineral crystallisation, heating to a few 100°C or the last daylight exposure of sediment grains. It covers the time range from a few years or decades up to several hundred-thousand years. Luminescence dating represents a suite of related techniques, all of which provide an estimate of radiation history due to accumulation of trapped charges in mineral lattices. During the last de-

cade, several methods have been developed that have considerably improved the reliability and precision of luminescence dating. It is highly recommended that information with regard to tests of quality assurance is included when publishing the results of luminescence dating.

Recent research is focusing on improved and innovative procedures of both palaeodose and dose rate determination. For palaeodose determination, it is necessary to better understand the sources of variation observed for individual palaeodose estimates determined for the same sample, especially to quantify the effect of microdosimetry. Furthermore, it is expected that it may be possible to extend the upper dating limit using approaches such as thermally-transferred OSL in quartz or by using a more stable luminescence emission, such as the red emission band of feldspars. With regard to dose rate determination, improvements in the handling of radioactive disequilibria and variation in sediment moisture may help to further improve the precision and accuracy of luminescence dating.

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