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## Luminescence dating of Netherlands' sediments

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### Abstract

Over the last decades luminescence dating techniques have been developed that allow earth scientists to determine the time of deposition of sediments. In this contribution we review: 1) the development of the methodology; 2) tests of the reliability of luminescence dating on Netherlands' sediments; and 3) geological applications of the method in the Netherlands. Our review shows that optically stimulated luminescence dating of quartz grains using the single aliquot regenerative dose method yields results in agreement with independent age control for deposits ranging in age from a few years up to 125 ka. Optical dating of quartz has successfully been applied to sediments from a wide range of depositional environments such as coastal dunes, cover sands, fluvial channel deposits, colluvial deposits and fimic soils. These results demonstrate that optical dating is a powerful tool to explore the natural archive of the Netherlands' subsurface.

**Keywords:** luminescence dating, OSL, quartz, feldspar, Quaternary

### Introduction

Owing to the location of the Netherlands in a subsiding basin the subsurface of the country largely consists of unconsolidated materials deposited during the past two million years (the Quaternary). Benefiting from this geological archive, Dutch researchers have played an important role in Quaternary geology. In the absence of dating techniques for clastic material, investigations of Quaternary deposits were largely based on palynological and sediment-petrological evidence (e.g. Zagwijn, 1974; 1985; 1989; De Jong, 1988; Gibbard et al., 1991). Determining the age of the deposits was only possible using radiocarbon dating which is restricted to the last 30,000 to 40,000 years. Moreover, suitable organic material is needed to apply radiocarbon dating. As a consequence, the chronology information on sediments can only be gained indirectly through dating intercalated organic horizons.

With the development of luminescence-dating techniques it has become feasible to determine the time of sediment

deposition directly. Luminescence dating allows the establishment of chronologies for aeolian, fluvial and colluvial deposits formed during the last glacial cycle, and sometimes beyond that. Thereby this technique aids us to improve understanding of the development of the Netherlands' subsurface during the later part of the Quaternary, and to test hypotheses about the landscape evolution that were earlier suggested.

This paper is aimed at geological users of luminescence dating in the Netherlands. We outline the principles of luminescence dating and summarise the advances in dating technology that led to improvements in accuracy and precision, and broadened the range of applications. We limit the technical intricacies; for those we refer to the abundant literature of specialist books and papers (e.g. Daniels et al., 1953; Aitken, 1985, 1998; Bøtter-Jensen et al., 2003; Wintle & Murray, 2006 and references therein). We review the performance of luminescence dating methods in the Netherlands as tested by comparison of luminescence ages with independent age control. We also present an overview of geological applications of

luminescence dating in the Netherlands and we discuss the methodological developments and geological applications that are to be expected in the near future.

## Luminescence dating

### Basic principles

Sediments are slightly radioactive due to the natural occurrence of radionuclides from the uranium (U) and thorium (Th) decay chains and from potassium ( $^{40}\text{K}$ ). As a consequence, mineral grains of quartz and feldspar, which are the main constituents of Netherlands' sediments, are exposed to a constant fluence of ionizing radiation. Some of the energy from this radiation is stored in the crystal structure of the minerals. When the mineral grains are eroded and transported the energy stored in the crystal is erased by exposure to daylight. Thereby the energy stored in the crystal is a measure for amount of ionizing radiation received by the mineral grain since its last exposure to daylight, i.e. since burial (Fig. 1).

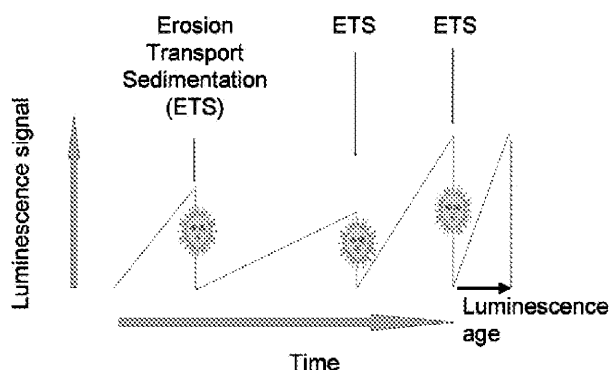


Fig. 1. The luminescence signal of grains of quartz or feldspar is reset by daylight exposure during erosion, transport and sedimentation, and builds up after burial due to exposure to natural ionizing radiation from the surrounding. Thereby the luminescence signal provides a measure of the amount of ionizing radiation received since burial (equivalent dose). This information combined with the ionizing radiation flux provides the age of the sample (equation 1).

In luminescence dating both the ionizing radiation flux and the total amount of radiation received by the sample since burial are determined. The ionizing radiation flux is called the dose rate or annual dose; it is derived from measurements of the concentration of radionuclides in the sediment. The total amount of ionizing radiation received by the sample since burial is called the burial dose or equivalent dose; it is determined through measurement of the luminescence signal. Luminescence is a minute light signal that is emitted when the energy stored in the crystal is liberated by heating the crystals or exposing them to light of a specific wavelength. The phenomenon is called thermoluminescence (TL) or optically stimulated luminescence (OSL) depending on the mode of stimulation.

The age of a sample is obtained by combining equivalent dose and dose rate following equation 1. The unit of absorbed dose is the Gray (symbol Gy), and is defined as 1 Joule per kg.

$$\text{Age (yr)} = \text{equivalent dose (Gy)} / \text{dose rate (Gy/yr)} \quad (1)$$

### A brief history of luminescence dating

Thermoluminescence (TL) dating techniques were first developed to determine the age of pottery (reviewed by Aitken, 1985). With pottery the luminescence signal of incorporated quartz and feldspar crystals is reset upon heating in the baking process. Geological application of TL dating started with attempts to date volcanic eruptions through TL measurements on ash, glass and lava (e.g. Aitken et al., 1968; Miallier et al., 1983). Application to unheated sediments arose from the finding that the TL signal of mineral grains was reset prior to burial by exposure to sunlight (Wintle and Huntley, 1979; 1980). Because a relatively long exposure to sunlight was needed to reset the TL signal, successful application of TL dating to sediments was largely restricted to aeolian deposits such as loess. Nevertheless, the technique provided the first possibility to directly date clastic sediments and has been widely applied. For more information on TL dating techniques and applications see e.g. Prescott and Robertson (1997).

In the mid eighties Huntley et al. (1985) discovered the possibility of using the optically stimulated luminescence (OSL) signal for dating sediments. The OSL signal is more suitable for sediment dating because it is far more sensitive to light than the TL signal; a few tens of seconds exposure to sunlight is enough to reduce the OSL signal down to 1% (e.g. Godfrey-Smith et al., 1988). OSL or optical dating is now the method of choice for determining the burial age of sediments.

### Determining the dose rate

Sedimentary minerals are exposed to a low level of ionizing radiation that is omnipresent in nature. The radiation originates from the radioactive decay of radionuclides that are present in the sediment, either within the minerals used for dating and/or in adjacent material. For dating we are concerned with alpha and beta particles, with gamma rays and with cosmic radiation.

The dose rate is derived from a determination of the concentration of the naturally occurring radioisotopes. These concentrations can be determined by a wide range of methods, including ICP-MS, XRF, NAA and high-resolution gamma-ray spectrometry (see e.g. Hossain et al., 2002). The latter is the preferred technique as it allows determining the concentration of individual radionuclides from the U and Th decay chains. In this way, it can be tested whether or not some of these radionuclides have been washed out or accumulated during burial (Krbetschek et al., 1994; Olley et al., 1996), i.e.



whether or not the dose rate has remained constant from the time of deposition to the time of sampling.

The radiation received by minerals in the sediment is dependent on water and organic contents of the sediment. Both substances absorb radiation from their surrounding and thereby shield the minerals from radiation and reduce the effective dose rate (Aitken, 1985). As a consequence of this dependency, assumptions have to be made on the water and organic contents since burial to assess the dose rate. As a rule of thumb, a change of 1% in water or organic contents (expressed as weight of water or organic matter divided by the weight of the clastic material) will have an effect of ~1% on the dose rate and thus on the luminescence age of the sample. For sandy sediments water content cannot exceed about 20% by weight; variations during burial have limited consequences for the dose rate and thus luminescence age obtained. However, for muddy and/or organic sediments the water and organic contents can greatly vary through time and may cause significant uncertainties in the dose-rate estimation.

Besides the radiation flux from radionuclides in the sediment, grains receive an additional dose from cosmic radiation. The cosmic radiation flux decreases with depth below the surface because it is attenuated within the sediment (Prescott & Hutton, 1994). To correctly assess the cosmic dose during burial, assumptions have to be made on the overburden depth during the burial history. The effect of these assumptions on the age depends on the relative contribution of cosmic radiation to the total dose rate, but is usually minor.

For quartz grains the dose rate due to decay of radionuclides incorporated in the crystal is very small and often neglected (but see Vandenberghe et al., 2003, De Corte et al., 2006). This is different for feldspar and zircon grains which receive a significant additional component of radiation flux from internal sources. For potassium-rich feldspars the internal beta dose originates from  $^{40}\text{K}$  incorporated in the crystal; the dose rate due to this internal component is roughly of the same order of magnitude as the external dose rate. Zircon grains contain large amounts of U and Th which results in very large internal alpha and beta doses of roughly two orders of magnitude greater than the external radiation dose (Van Es et al., 2002).

### Determining the equivalent dose

Most developments in luminescence dating techniques have been in the improvement of the assessment of the equivalent dose. In the section 'a brief history of luminescence dating' we discussed the shift of stimulation mode from thermal to optical. In the earliest optical dating methods many subsamples (aliquots) were needed to obtain an equivalent dose estimate. These 'multiple aliquot' methods assume that the equivalent dose in each subsample is identical, an assumption that is not always valid.

Duller (1991) proposed methods in which all measurements needed for equivalent dose assessment are made on a single subsample. Such single-aliquot methods allow researchers to investigate the spread in equivalent doses between subsamples. Single-aliquot methods were revolutionized with the development of Single Aliquot Regenerative dose (SAR) procedures (Murray and Wintle, 2000). In the SAR procedure luminescence sensitivity changes are monitored and corrected for (Fig. 2). This method is now widely applied to quartz, and similar procedures have been developed for polymineral fine grains (Banerjee et al., 2001) and feldspar samples (Wallinga et al., 2000a).

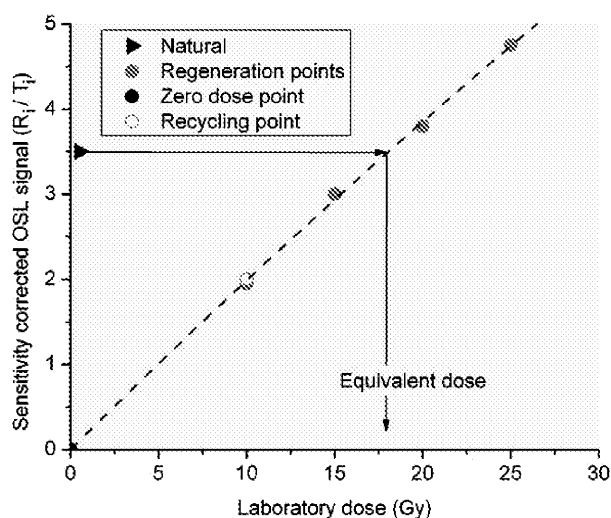


Fig. 2. The Single-Aliquot Regenerative dose (SAR) protocol (Murray and Wintle, 2000; 2003) is the most robust and reliable method for quartz equivalent dose determination. On a single aliquot (1 - 10 mg of quartz grains) the natural OSL signal and the OSL response to a number of laboratory irradiations is measured. Each OSL measurement is followed by measurement of the response to a fixed (test) dose to monitor OSL sensitivity changes during the measurement procedure. The equivalent dose is obtained by projection of the sensitivity corrected natural OSL signal (black square) on the sensitivity corrected dose response curve (obtained through fitting the sensitivity corrected OSL responses to laboratory irradiation, red triangles). To test the performance of the SAR method the completeness of resetting of the OSL signal after a SAR cycle is checked (recuperation point, green triangle) and one of the laboratory irradiations is repeated (recycling point, blue triangle). The latter should yield the same sensitivity corrected OSL signal as the first measurement of the same dose.

Single aliquot methods are essential to determine the dose received by the sample since burial in the case that light exposure prior to burial was limited, such as may be expected for e.g. fluvial and colluvial sediments. Limited light exposure may result in incomplete resetting of the luminescence signal; a remaining luminescence signal at the time of deposition results in an apparent remnant dose. The burial dose builds up

on top of this remnant dose and as a consequence the equivalent dose determined on the sample will overestimate the burial dose. This will lead to overestimation of the burial age of the sample, and should be avoided (Fig. 3).

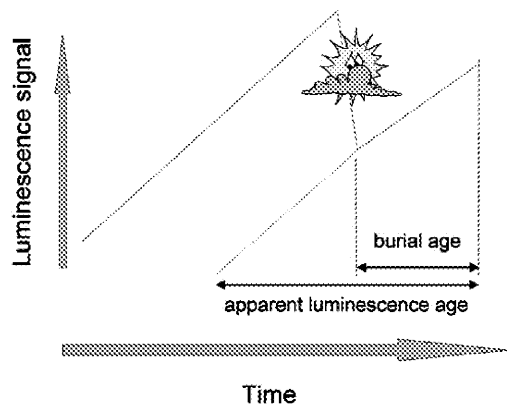


Fig. 3. Limited light exposure of mineral grains before burial (e.g. in a fluvial environment) may result in incomplete resetting of the luminescence signal. If such is the case, the apparent luminescence age will overestimate the true burial age unless special measures are taken.

Several approaches have been taken to avoid or reduce age overestimation due to incomplete resetting of the luminescence signal (often referred to as poor bleaching or heterogeneous bleaching). First of all, one can make use of the luminescence signal that is most readily reset by daylight exposure; hence the OSL signal is preferred over the TL signal. Additionally, the OSL signal consists of multiple components with differing sensitivity to light (Bailey et al., 1997). The 'fast component' is most rapidly reset, and equivalent-dose assessment should make use of that signal (Wintle and Murray, 2006).

Secondly, one can try to select the right grains. When light exposure is limited, it is very likely that the OSL signal of different grains will be reset to different degrees (Duller, 1994; Murray and Olley, 1999). As long as the OSL signal of some grains is completely reset it is in principal possible to date the sediment if only those grains are selected for equivalent-dose determination. Single aliquot methods (Murray and Wintle, 2000, 2003) are essential for this purpose and recently developed equipment (Bøtter-Jensen et al., 2000) even allows measurement of aliquots containing a single grain of quartz or feldspar.

### Minerals for luminescence dating

Quartz and feldspar minerals are mostly used for luminescence dating because they are most abundant in Quaternary sediments. Comparison with independent age control has shown that sand-sized quartz provides the most accurate dating results (Wallinga et al., 2001; Murray and Olley, 2002). The quartz OSL signal is reset rapidly when exposed to daylight and is stable

during geological burial (e.g. Wintle and Murray, 2006). Drawback of the quartz OSL signal is that it saturates at relatively low doses (Fig. 4) which usually limits its applicability to the last glacial cycle (~125 ka), although in the Netherlands the age range is often longer owing to low environmental dose rates.

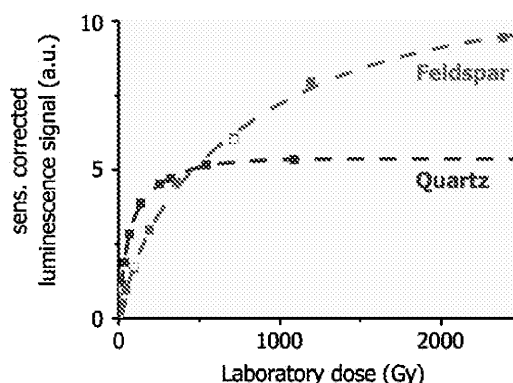


Fig. 4. Sensitivity-corrected quartz OSL and feldspar IRSL signals as a function of the laboratory dose received. Note that the quartz OSL signal saturates at far lower doses than the feldspar IRSL signal.

Optical dating of feldspar is usually referred to as infrared stimulated luminescence (IRSL) dating after the wavelength used for stimulation. Advantage of feldspar dating is that the IRSL signal saturates at far higher doses than the quartz OSL signal (Fig. 4). It can therefore in principle be used to date older deposits. However, it is widely known that feldspar ages may underestimate the burial age as a consequence of anomalous fading, the decay of the luminescence signal due to quantum-mechanical tunneling of trapped charge (Wintle, 1973; Spooner, 1994; Huntley, 2006). Recently, procedures to correct for anomalous fading have been suggested (Huntley & Lamothe, 2001; Lamothe et al., 2003). Using such correction procedures ages in agreement with independent age estimates were obtained for Holocene samples, but validity of these procedures for sediments deposited before the last glacial-interglacial cycle is questionable (Wallinga et al., 2007). Besides the dating of sand-sized feldspar fraction, the IRSL signal is also used for dating a mixture of undifferentiated fine-grained (4 - 11  $\mu\text{m}$ ) minerals. As quartz is insensitive to infrared stimulation, IRSL dating of fine grains is similar to using feldspar minerals and shares the advantages and drawbacks.

A third mineral that can be used for dating is zircon. Due to the high internal concentrations of U and Th, almost all ionizing radiation to zircon grains comes from inside the grain. Therefore the dose rate is independent of water content and burial history. Drawback of zircon dating is that the mineral occurs in low concentrations; hence very large samples are needed to obtain enough material for dating and sample preparation procedures are tedious. Methods for zircon dating are under development (Van Es et al., 2000, 2002) and, so far, have seen little application.



## Testing luminescence dating methods in the Netherlands

### Early attempts – feldspar TL

In the eighties of the last century Prof. E.A. Koster of Utrecht University initiated investigations of the applicability of luminescence dating to Netherlands aeolian deposits. This resulted in experimental work by Dijkmans and Wintle (1991) who tested feldspar TL dating methods by applying them to Weichselian coversand deposits and Holocene drift sands from the Lutterzand area in the eastern Netherlands. The site is indicated in Fig. 5, as are the locations of all other sampling sites discussed in this paper.

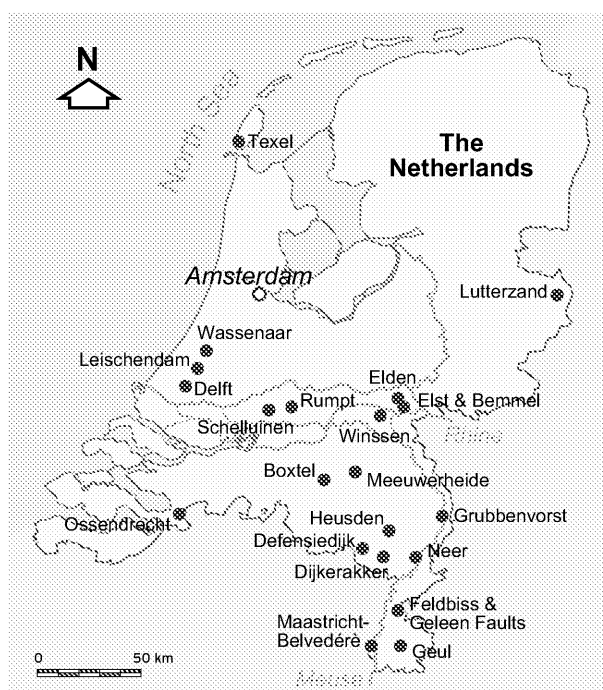


Fig. 5. Locations of all luminescence-dating sites discussed in this paper.

For the Young Holocene drift sands Dijkmans and Wintle (1991) obtained ages that agreed reasonably well with the expected ages, although incomplete resetting of the TL signal prior to deposition may have affected the youngest sample. For the Late Weichselian coversand samples an age underestimation of 20 - 40% was observed relative to radiocarbon chronologies. In similar settings in the southern Netherlands TL ages for Holocene drift sands (Defensiedijk) agreed satisfactory with independent age control whereas those on Late Weichselian coversand deposits (Meeuwerheide) underestimated compared to the radiocarbon chronology (Dijkmans et al., 1992). Reasons for the reported age underestimation could be anomalous fading of the feldspar TL signal, or the use of a UV transmitting filter for luminescence signal collection (Krbetschek et al., 1997).

Debenham (1993) reports on TL dating of fine-grained material from the Maastricht-Belvédère Palaeolithic site. Three loess samples of presumed Weichselian age returned an average TL age of  $\sim 15.7 \pm 1.9$  ka. For two samples of presumed Saalian age only minimum ages ( $>150$  ka and  $>80$  ka) could be determined due to instability of the TL signal used.

Frechen and Van den Berg (2002) applied TL dating to feldspar extracts from samples obtained from coversands along the Peel Boundary Fault in the southern Netherlands. The authors state that they found systematic TL age underestimation compared to the IRSL ages obtained for most of the samples, but they do not discuss the causes. A look at their data shows that only for the oldest two samples the age underestimation is clear, for the other eight samples TL and IRSL ages are in agreement. It is possible that for these younger samples a systematic TL age underestimation is compensated by an overestimation due to incomplete resetting of the TL signal prior to deposition, although this interpretation remains speculative.

As discussed in the methodological section, TL dating is not the method of choice for the dating of sediments because the TL signal is not reset as readily by daylight exposure as the OSL signal. The studies using feldspar TL dating in the Netherlands have shown an additional problem in that the burial age of the deposits is often underestimated. In the light of these problems, TL methods should not be used for the dating of Netherlands' sediments and published results should be regarded with caution.

### Quartz OSL

Quartz OSL dating has been applied to deposits of known age in a number of studies. Smith et al. (1990) were the first to test the accuracy of quartz OSL dating in the Netherlands. They applied multiple-aliquot OSL dating methods to coversands above and below the Usselo layer at the coversand type locality Lutterzand in the eastern Netherlands. The Usselo layer is an organic layer attributed to the Allerød Interstadial; it has been radiocarbon dated to  $\sim 13$  cal. ka BP (Smith et al., 1990; Schwan, 1991; Vandenberghe et al., 2004). Smith et al. (1990) obtained ages of  $7.2 \pm 1.8$  and  $8.2 \pm 1.7$  ka on Younger Cover sands above the Usselo layer using multiple aliquot additive-dose (MAAD) and regenerative-dose (MAR) methods, respectively. For sediments below the Usselo layer (Older Coversands) they obtained ages of  $11.0 \pm 2.5$  and  $10.6 \pm 2.4$  ka using the two methods. Stokes (1991) presented different quartz OSL ages obtained by a MAAD procedure for exactly the same samples. In this study, ages of  $11.4 \pm 1.4$  and  $13.2 \pm 2.4$  ka were obtained for samples above and below the Usselo layer, respectively. Reasons for the age discrepancy between the two studies remain unclear as the papers do not refer to each other.

Bateman & Van Huissteden (1999) revisited the Lutterzand site and used MAAD and single-aliquot additive dose (SAAD)

OSL dating of sand-sized quartz grains to date Holocene drift sands and Weichselian coversands, the ages reported are those obtained using the SAAD method. The authors compare their results with a radiocarbon chronology. However, the comparison should be regarded with caution as some of the radiocarbon ages may be affected by reworking or hardwater effects and the correlations with other sites are unsure. For the Older Coversand I deposits the authors obtained a single OSL age of  $21.9 \pm 1.9$  ka, which agrees with the limited radiocarbon age control (22.5 - 30.5 cal. ka BP; calibration following the original publication). For the Older Coversand II they obtained an average OSL age of  $15.8 \pm 1.8$  ka, in agreement with the calibrated radiocarbon age range of 15.3 - 16.8 cal ka BP for these deposits. For Younger Coversands they obtained an average OSL age of  $12.5 \pm 1.1$  ka which is in good agreement with the inferred age of 11.8 - 15.3 cal. ka BP. The authors obtained an age of  $0.6 \pm 0.1$  ka for younger drift sands known to have been deposited during the past millennium.

In a similar setting near Ossendrecht (southern Netherlands) Vandenberghe et al. (2004) used several quartz OSL dating methodologies to date known age coversands. Optical ages obtained on coversands above and below the Usselo soil are compared to radiocarbon ages obtained on that soil layer. The authors conclude that the SAR technique is most suitable for OSL dating these deposits; it performs better than the MAAD methods and SAAD methods that were also investigated. Optical dating using SAR provided average ages of  $14.7 \pm 0.6$  ka below the Usselo soil (Older Coversand II and Younger Coversand I) and  $12.3 \pm 0.8$  above the Usselo soil. Both optical ages are averages of three samples and are in good agreement with the radiocarbon age control. In a separate study, Vandenberghe et al. (2003) investigated the cause for the relatively large spread in quartz SAR equivalent doses observed for these samples. Based on a very detailed study on a single sample they concluded that the spread was most likely a consequence of small-scale differences in the dose rate experienced by different grains. The authors conclude that the accuracy of the optical ages in their study is not affected because the scale of analysis (for sampling, equivalent-dose and dose-rate determination) is large enough to average out effects. Although not published in the international literature, we also mention the work of Fink (2000) who obtained similar but less precise results on the Ossendrecht coversands using quartz OSL SAR methods; the results of this study are summarized by Vandenberghe et al. (2004).

Wallinga et al. (2001) applied OSL dating to sand-sized quartz from fluvial channel deposits of three Holocene channel belts. These authors also used the SAR method of Murray and Wintle (2000). The age of the older two channel belts (Rumpt and Schelluinen sites) is restricted through radiocarbon dates of the period of activity of the streams, while the time of formation of the youngest channel deposits is reconstructed from historical maps. Results showed that quartz OSL ages

agree well with independent age control for the older two, whereas the age of the youngest sample (Winssen site) is overestimated by about 600 years, likely due to insufficient resetting of the OSL signal prior to deposition. Truelsen and Wallinga (2003) carried out additional experiments on this sample to investigate the dependency of bleaching on the grain size used for analysis and found that the coarser grain sizes were better bleached. In addition to the Holocene samples, Wallinga et al. (2001) dated late Weichselian fluvial deposits containing pumice from the Laacher see eruption (Elden site). The quartz OSL age obtained ( $13.3 \pm 0.8$  ka) agreed with the age of the Laacher See eruption (13.0 - 13.3 cal ka BP; Friedrich et al., 1999).

To test applicability of quartz OSL dating to deposits formed during the past centuries to decades, Ballarini et al. (2003) carried out quartz OSL dating using the SAR method to approximately 30 samples from a range of dune ridges on the southwest coast of Wadden island Texel (Fig. 6). The age of the dune ridges formed during the past 300 years is accurately known from historic documents and maps. OSL ages obtained agreed very well for these samples; it proved even possible to use the method to date dune deposits formed during the last decades. A single outlier was found for a very recent dune blown up from nourishment sand; these sands had probably not been through as many bleaching cycles as the truly natural deposits and as a consequence the OSL signal was less completely reset. For another sample the OSL age was ~40 years younger than expected for that dune ridge; the difference may be caused by sampling sand that was disturbed by digging animals (e.g. a rabbit).

A single test of the validity of quartz OSL dating for sediments formed before the last glacial in the Netherlands is provided by Schokker et al. (2004). They used the SAR method to date a sand layer incorporated in peats that were ascribed to the Eemian (OIS 5e) based on their pollen contents. The age obtained ( $114 \pm 12$  ka) agrees reasonably well with the expected age (~125 ka).

The SAR method is now widely accepted as the most reliable and robust method for quartz OSL equivalent dose determination (e.g. Murray and Olley, 2002; Wintle and Murray, 2006). In Fig. 7 we summarize all quartz SAR OSL dating results on known age sediments from the Netherlands. The overall agreement is good over the entire range from a few years to the last interglacial with the exception of some young samples where the OSL age overestimates compared to the independent age as a consequence of incomplete resetting of the OSL signal at the time of deposition.

### **Feldspar IRSL**

Wallinga et al. (2001) applied feldspar IRSL dating, using the feldspar SAR procedure (Wallinga et al., 2000a), to the same channel sands which they used for testing quartz OSL methods (discussed above). The feldspar ages consistently underestimated

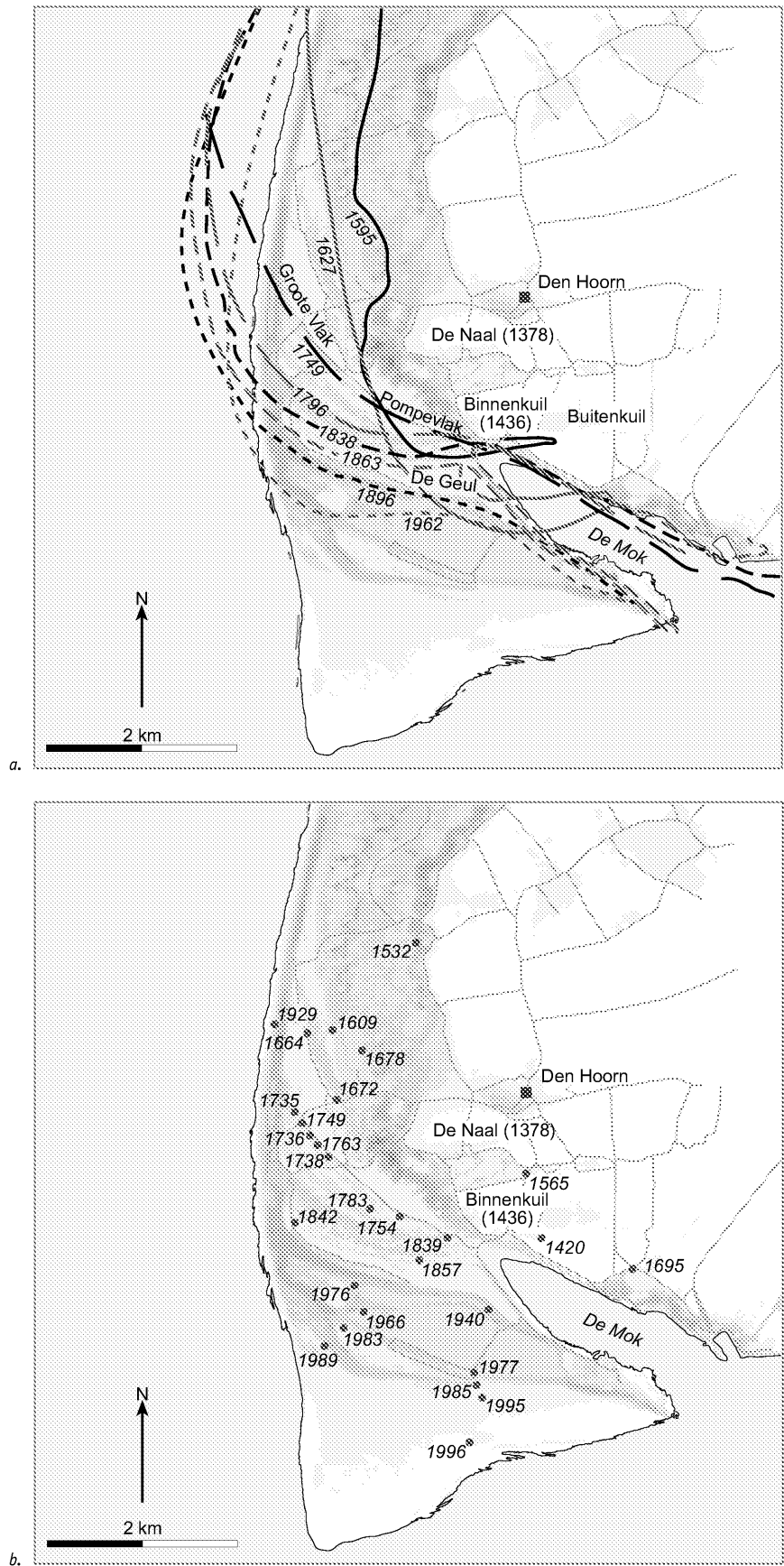


Fig. 6. The southern part of Wadden Island Texel indicating the age of dune ridges determined from historical maps (left) and quartz OSL ages (right). Data from Ballarini et al. (2003) and Van Heteren et al. (2006); figures modified from Van Heteren et al. (2006).



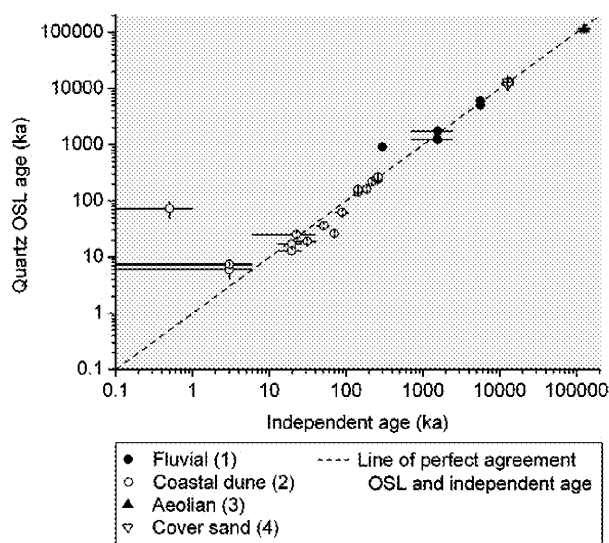


Fig. 7. All quartz OSL ages obtained with the SAR method for sediment samples with independent age control from the Netherlands. Sources: 1) Wallinga et al. (2001); 2) Ballarini et al. (2003); 3) Vandenberghe et al. (2004); 4) Schokker et al. (2004). The vast majority of datapoints shows good agreement between OSL and independent age estimates. Deviations for a few points are discussed in the main text.

the burial age of the deposits. Only for the youngest deposit an age overestimation was found, which was attributed to insufficient resetting of the IRSL signal prior to deposition. The authors found that the age underestimation was partly caused by uncorrected sensitivity changes (Wallinga et al., 2000b) and optical absorption within the grains (Wallinga and Duller, 2000) but the observed underestimation of age could not entirely be accounted for. Anomalous fading of the IRSL signal was not observed by the authors. However, more detailed investigation that were carried out later on, showed that the samples may be affected by anomalous fading (Lamothe, priv. comm.).

Frechen and Van den Berg (2002) applied MAAD IRSL dating to K-feldspar extracts from coversands along the Peel Boundary Fault near Neer (southern Netherlands). The authors obtained an internally consistent IRSL chronology for the coversand deposits in the foot wall. According to the authors, their IRSL ages agreed with geological age estimates for most of the samples, indicating that their methods were more successful than those used by Wallinga et al. (2001). However, comparison of the ages obtained by Frechen and Van den Berg (2002) with independent age information and optical dating results from other studies is problematic because figures and tables in the paper contradict with respect to the level of the Beuningen gravel bed.

Wallinga et al. (2007) applied feldspar IRSL dating using the SAR method to samples from a core penetrating a relatively continuous sedimentary record in the Roer-Valley Graben (Boxtel core; Fig. 8). The samples were previously dated by quartz OSL methods (Schokker et al., 2004; 2005), and include

the Eemian age sample discussed in the previous section. Feldspar IRSL ages consistently and substantially underestimated the burial age compared to the quartz OSL ages obtained on the same samples, and compared to the known age for the Eemian sample. Anomalous fading was observed for these samples, but correcting the age using the measured laboratory fading rate could not satisfactory resolve the age underestimation. Wallinga et al. (2007) concluded that the fading rate measured in the laboratory likely underestimates the natural fading rate during geological burial.

From these studies we conclude that for Netherlands sediments single-aliquot feldspar IRSL methods consistently underestimate the burial age of the deposits. So far, no satisfactory solution has been found for this age underestimation problems. Multiple aliquot methods (such as used by Frechen and Van den Berg, 2002) may provide a better alternative but assume complete resetting of the IRSL signal prior to deposition. We conclude that presently no reliable method for feldspar IRSL dating of Netherlands' sediments is available.

## Applications of luminescence dating in the Netherlands

In the previous section we concluded that quartz OSL dating using the SAR method is best suited for sediment dating. In the following overview of applications of luminescence dating in the Netherlands we will therefore focus on studies using this method although we will also briefly mention other studies. For the sake of clarity, six application types are distinguished.

### Weichselian coversands

Luminescence dating has been widely applied to establish the timing of coversand deposition in Western Europe (reviewed by Koster, 2005). Several studies used quartz OSL dating to determine the age of coversands in the Netherlands; an overview of the stratigraphy and optical dating results is given in Fig. 9. Here we concentrate on the geological interpretations based on the optical dating. At the Lutterzand site, Bateman and Van Huissteden (1999) dated a phase of widespread permafrost degradation and aeolian deflation (represented in the Netherlands by the Beuningen Gravel Bed Complex) to 22-17 ka. After this phase sandsheet deposition took place between 17 and 14 ka and dune formation was dominant during the Younger Dryas. Van Huissteden et al. (2001) used the optical ages obtained at the Lutterzand site to infer environmental conditions and palaeowind directions at the end of the Weichselian Late Pleniglacial. The SAAD methods used by the authors are now out-of-date as they neglect OSL sensitivity changes. Therefore, the absolute timing of the different phases as determined by later studies using the quartz SAR method is probably more accurate.

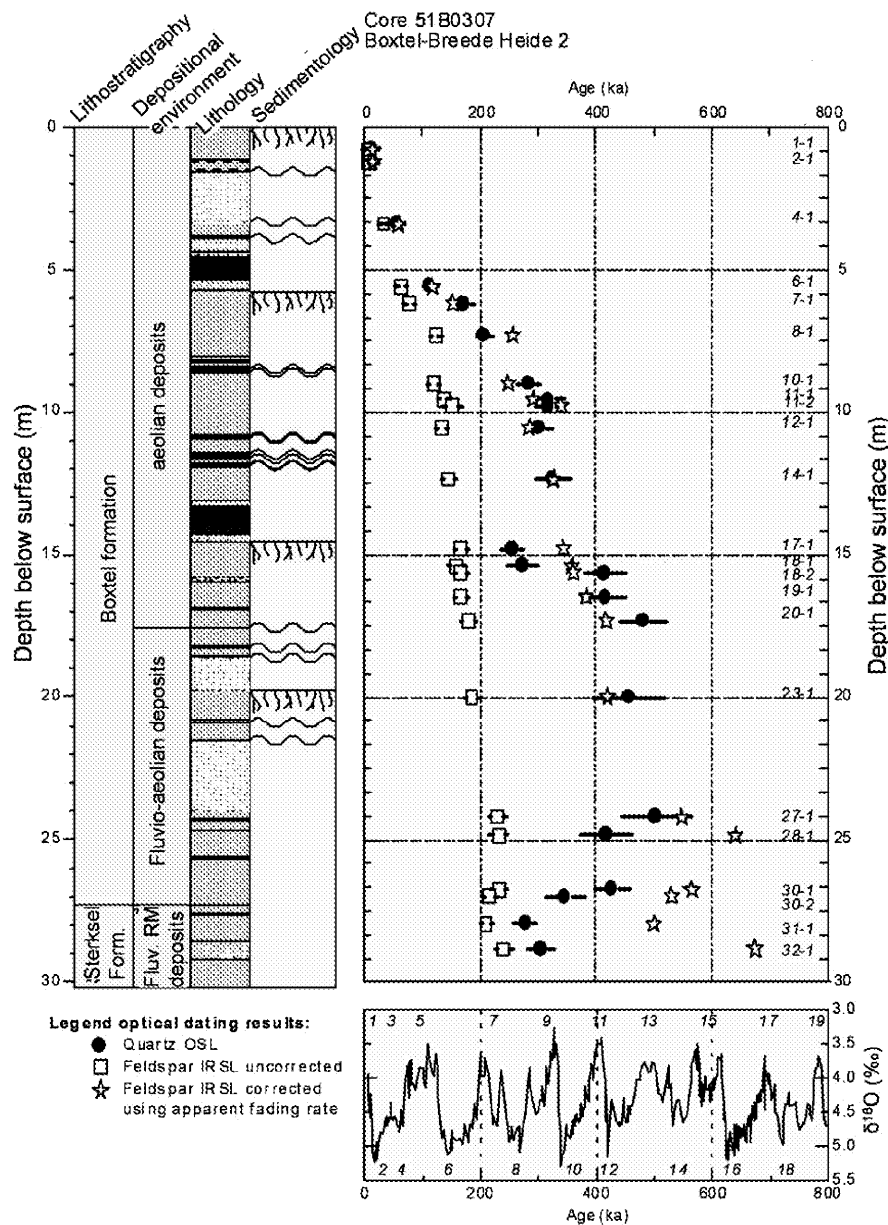


Fig. 8. Lithology, lithostratigraphy, quartz OSL and feldspar IRSL dating results for the Boxtel core penetrating Middle and Late Quaternary deposits in the Roer Valley Graben (southern Netherlands). Feldspar results are shown both uncorrected and after anomalous fading correction using the 'apparent fading' rate (after Schokker et al., 2005; Wallinga et al., 2007).

Kasse et al. (2007) applied quartz optical dating using the SAR method to 22 samples from coversand deposits at the Grubbenvorst type locality (southern Netherlands). Optical ages are internally consistent (see Fig. 10) and in agreement with optical dating results on coversand sequences elsewhere as well as age information derived from palaeoclimate indicators and radiocarbon dated stratigraphic markers. The authors reconstruct the sedimentary environment and climatic evolution by combining the optical dating results with sedimentological investigation of the deposits. A declining fluvial activity of the River Meuse, followed by coversand formation is observed. The deflation phase leading to formation of the Beuningen gravel bed was bracketed between  $17.2 \pm 1.2$  and  $15.3 \pm 1.0$  ka and the authors suggest a correlation with Heinrich Event H1. The study by Kasse et al. (2007) clearly shows the added

value of multiple samples from the same level; ages obtained on these samples together provide a more accurate and precise age estimate for the deposits and comparison of the results gives information on whether the uncertainties on individual estimates are correct.

### Fimic soils

Before the introduction of chemical fertilizers, plaggen agriculture was the dominant land use system in the Pleistocene coversand area of the Netherlands. The quartz grains in fimic soils are brought in by both aeolian processes and the addition of sods; the former group of grain is likely exposed to light prior to deposition, the latter may be exposed to light during ploughing of the field. Bokhorst et al. (2005) applied quartz

Fig. 9. Chronostratigraphy and lithostratigraphy of the late Weichselian coversand deposits (after Van der Hammen, 1971; Van Geel et al., 1989). Optically Stimulated Luminescence (OSL) ages of (1) type locality Lutterzand (Bateman and Van Huissteden, 1999); (2) Ossendrecht (Vandenberghé et al., 2004); (3) type locality Grubbenvorst (Kasse et al., 2007). Weighted average ages between brackets. Figure adapted from Kasse et al. (2007).

cal BP	Chronostratigraphy		Lithostratigraphy		OSL age		
	(1)	(2)	(1)	(2)	(1)	(2)	(3)
10	HOLOCENE						
11							
12	WEICHELSELIAN	Younger Dryas	TWENTE FORMATION	Younger Coversand II	11.7-13.2 (12.5)	11.5-13.1 (12.3)	13.8
13		Allerød		Usselo Bed or Soil			
14		Older Dryas		Younger Coversand I			
15		Bølling s.s.		Lower Loamy Bed			12.7
16	LATE GLACIAL	LATE PLENIGLACIAL	LUTTERZAND MEMBER	Older Coversand II	13.9-17.5 (15.8)	14.2-15.3 (14.7)	14.1-16.3 (15.3)
17				Beuningen complex			
18				Older Coversand I	21.9		17.2-25.2
19							
20							

OSL dating using the SAR method to two samples taken from the fmic horizon of the profile at Dijkerakker (southern Netherlands). An age of  $1023 \pm 208$  years was obtained for the lower sample taken near the base of the fmic horizon, whereas an age of  $394 \pm 40$  years was obtained for the sample higher

up in the profile. For the lower sample, the equivalent dose distribution indicated that some aliquots might contain grains for which the OSL signal was not completely reset (Fig. 11). In spite of this heterogeneous bleaching, the results indicate that optical dating forms a viable alternative to the use of

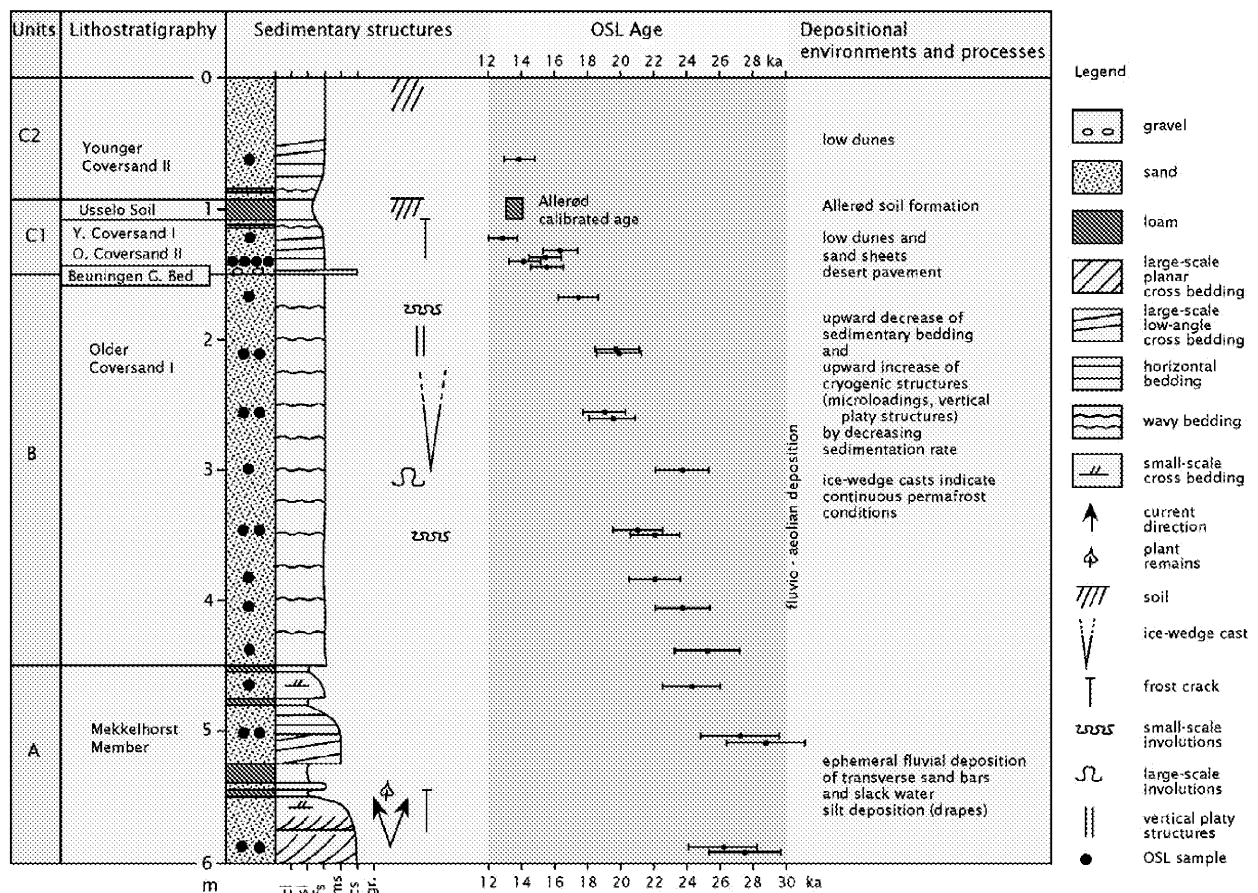


Fig. 10. Lithostratigraphy, sedimentary environments and OSL ages of the fluvial to aeolian succession in Grubbenvorst. Figure adapted from Kasse et al. (2007).

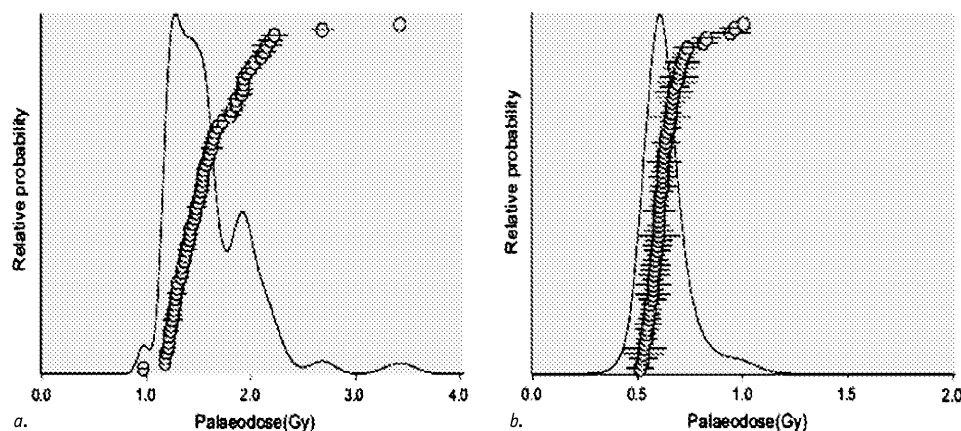


Fig. 11. Quartz OSL SAR equivalent dose (palaeodose) distributions obtained for two samples from a fmic horizon of the Dijkerakker. For the lowermost sample (a), some aliquots returned large palaeodoses, indicating that the OSL signal of some grains was not completely reset at the time of deposition. Graphs adapted from Bokhorst et al. (2005).

radiocarbon dating in these environments. Application of radiocarbon dating to fmic soils is often problematic because old organic material is brought in with the sods and may lead to overestimation of the age of the start of fmic deposition.

### Coastal dunes and inland drift sands

Van Heteren et al. (2006) used the dataset Ballarini et al. (2003) supplemented with optical ages on eight more samples to reconstruct the coastal evolution of the southwest coast of Wadden Island Texel. The eight additional samples were taken from more inland (older) dune ridges, which were of unknown age (Fig. 6). The authors could distinguish four phases in the development of the southwest coast of the island: 1) Before 1600 AD, the island expanded southward through spit accretion; 2) Between AD 1600 and AD 1800, dune-ridge formation resulted in accretion at the western side of the island; 3) Between AD 1800 and AD 1925 the bulge formed during the previous period migrated southward. 4) After AD 1925 the coastline flattened through erosion on the west side and renewed accretion on the south side.

In addition to these successful applications to coastal dune deposits, preliminary results of a recent study indicate that quartz OSL dating is also a powerful tool for dating Late Holocene inland drift sands (Schilder et al., 2006).

### Fluvial deposits

Following validation of SAR based quartz OSL dating to fluvial deposits of known age (Wallinga et al., 2001) this method was used in a number of studies to investigate the response of the Rhine-Meuse system to external forcing during the past two glacial cycles.

Törnqvist et al. (2000) investigated an almost 50 m deep core penetrating Middle to Late Pleistocene Rhine-Meuse deposits near Leidschendam, close to the present coastline of the western Netherlands. Chronological information from quartz OSL dating was combined with sedimentological and stratigraphic analyses of the sediments and data on shell,

diatom and pollen content. The optical ages provided the first chronological information for these deposits, but interpreting the chronology of the core was not straightforward. Optical ages obtained showed a slight age reversal. Moreover, estuarine deposits at a depth of ~29 m -NAP which were optically dated to  $58 \pm 4$  ka were ascribed to the most recent OIS 5 highstand (OIS 5a, ~80 ka) based on the pollen content. Törnqvist et al. (2000) conclude that the quartz OSL age on this sample is underestimated. With respect to the response of the Rhine-Meuse system to sea level changes, the authors concluded that major incision related to sea-level fall took place after each of the OIS 5 sea-level highstands. Moreover, the authors conclude that most of the preserved deposits were formed in a period of relative sea-level fall.

The work on the Rhine-Meuse system was continued by Törnqvist et al. (2003) who reported quartz OSL ages on two more cores penetrating the Middle to Late Pleistocene deposits in the western Netherlands. Information from the cores was combined to provide a south-north transect perpendicular to the drainage direction of the Rhine-Meuse system (Fig. 12). Optical dating allowed the authors to ascribe deposits to OIS stages, although also in one of the new cores there was a reversal in the optical ages around the OIS 5 - 4 boundary (~80 ka). Törnqvist et al. (2003) concentrated on the timing of the last sequence boundary formation. They concluded that the formation of this sequence boundary was associated with the onset of sea-level fall associated with the OIS 5 - 4 transition (~80 ka). The sequence boundary was commonly cryptic both in the chronology (representing less than 10 ka) and in sedimentology.

Based on the same dataset, Wallinga et al. (2004) estimate an average aggradation rate of 8 cm/ka for Rhine-Meuse deposits formed during the past two glacial cycles in the western Netherlands. They ascribe this aggradation to tectonic subsidence and discuss reasons for deviations from the aggradation trend. High aggradation rates during OIS 4 and 3 (74 - 24 ka) were interpreted to reflect the relatively dry climate in combination with high sediment input. Incision around the Last Glacial Maximum (LGM) and a subsequent aggradation phase

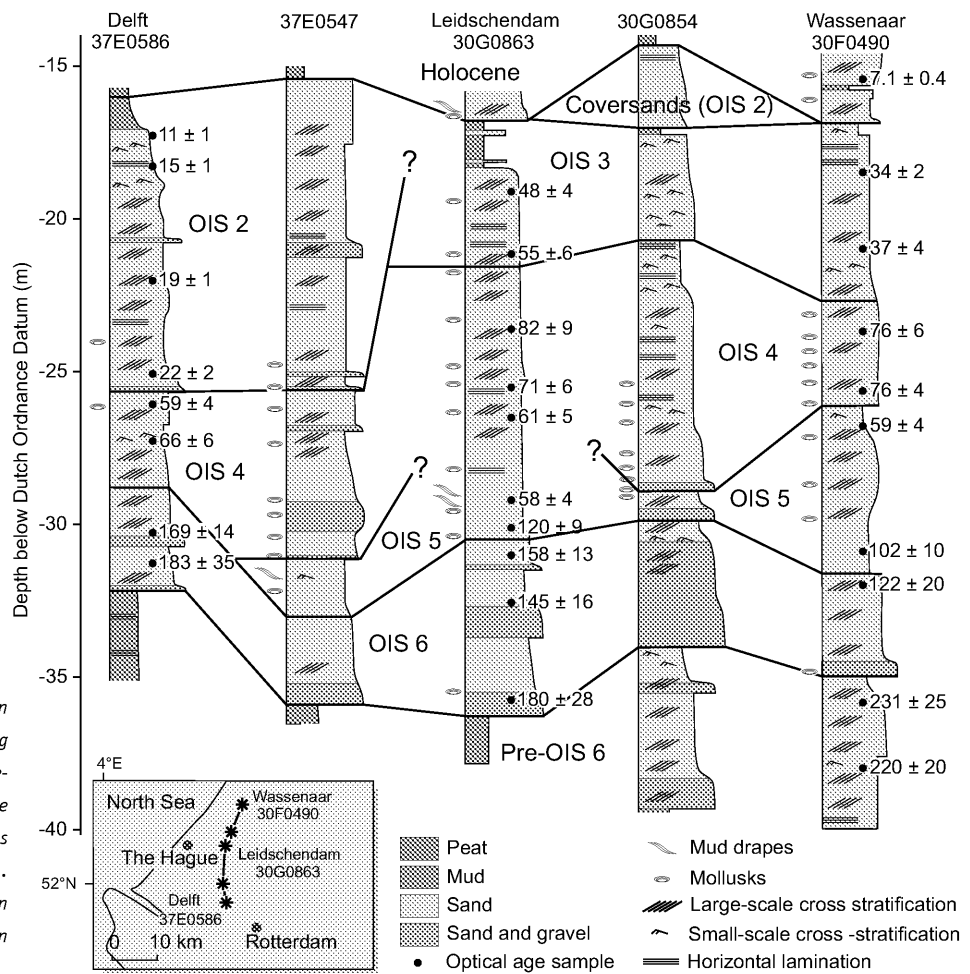


Fig. 12. South-North cross-section in the western Netherlands showing the chronostratigraphy of Rhine-Meuse deposits formed during the last two glacial interglacial cycles (after Wallinga et al., 2005). Interpretation is largely based on quartz OSL SAR ages indicated in the graph.

were ascribed to the build-up and collapse of a glacial-isostatic forebulge in this period. Wallinga et al. (2004) conclude that the sedimentary record is dominated by strata formed during time intervals when the study area was completely unaffected by sea-level control.

The quartz OSL chronology of the deposits was used by Busschers et al. (2005) who investigated sedimentology and lithology of the different fluvial units in the western Netherlands. They concluded that coarse-grained fluvial sediments are primarily deposited under cold climatic conditions, with low vegetation cover and continuous permafrost. Finer-grained sediments were generally deposited during more temperate climatic conditions with continuous vegetation cover and/or periods of sea-level highstand.

Gouw and Erkens (2007) discuss the external controls that determined the architecture of the Holocene Rhine-Meuse delta (the Netherlands). Based on 130  $^{14}\text{C}$  dates they draw timelines in five cross-valley sections through the delta deposits. The radiocarbon dataset is supplemented with eight OSL dates. Four OSL samples taken from Weichselian deposits of the Kreftenheye formation near Bommel returned ages of  $\sim 11$  ka, indicating that the pleniglacial terrace is absent at that

site. The OSL ages confirmed that the deposits post-dated the Laacher See eruption as evidenced by the occurrence of pumice in the deposits. At the nearby Elst site, four OSL samples were taken from Holocene channel deposits. These samples returned ages of  $2.69 \pm 0.13$  to  $3.21 \pm 0.19$  ka and indicated that the Holocene channel had incised to a depth of at least 10.5 m below the present surface.

De Moor et al. (2007) used OSL and  $^{14}\text{C}$  dating to reconstruct the Holocene landscape development and fluvial dynamics of the Geul River (southern Netherlands). Five OSL samples returned ages ranging from  $12.4 \pm .7$  to  $0.54 \pm 0.04$  ka. Based on the combined  $^{14}\text{C}$  and OSL chronology the authors concluded that deforestation during the Roman Period and High Middle ages led to severe erosion and increased floodplain sedimentation in the study area, indicating that the Geul catchment is highly sensitive to changes in land use.

### Sedimentary records of the Roer Valley Graben

Due to tectonic subsidence the Roer Valley Graben in the southern Netherlands is an ideal setting for the formation of relatively continuous sediment records in a continental setting.



Schokker et al. (2004; 2005) have explored this archive through a multidisciplinary investigation including quartz OSL dating using the SAR method. Schokker et al. (2004) discuss the upper seven metres of a core taken near the town of Boxtel. Four samples were dated and provided stratigraphic consistent results. The authors discuss the clastic sediment flux and reconstruct the local groundwater-level history of the site.

In their second paper, Schokker et al. (2005) discuss the sediment provenance, the depositional processes and the continuity and timing of deposition of sediments in the Roer Valley Graben. The study is based on the Boxtel core which penetrates Pleistocene deposits down to 30 m depth and the Heusden core which penetrates the upper 25 m of Pleistocene deposits. A total of 37 quartz OSL samples were dated from the two cores. The dataset from the Heusden core is internally consistent and shows a large hiatus between 250 ka and 30 ka. It is not clear whether this hiatus is due to large-scale erosion or a long period of non-deposition. Optical ages obtained on the Boxtel core showed some inconsistencies (Fig. 8). Firstly, at a depth ~15 m two samples yielded ages that are somewhat younger compared to ages obtained for overlying and underlying samples. The causes for this apparent age underestimation remain unclear. Secondly, there is an age reversal below ~20 m depth, which the authors attribute to saturation of the quartz OSL signal. Schokker et al. (2005) propose that only dating results on samples with equivalent doses smaller than 200 Gy (~ upper 17.6 m) are reliable. Owing to the low dose rate at the site, this allows OSL dating up to 400 ka i.e. far beyond the usual limit of ~125 ka. From their multidisciplinary investigation, the authors conclude that the depositional environment gradually changed from fluvial to aeolian. Sedimentation and preservation of deposits occurred under humid surface conditions.

Using feldspar IRSL dating and a new anomalous fading correction procedure Wallinga et al. (2007) showed that, most probably, the quartz OSL age of the lowermost samples severely underestimates the burial age (Fig. 8). However, no uncertainties on the IRSL age estimates could be given because of assumptions made for the anomalous fading correction. The IRSL age estimates are only crude approximations of the burial age of the deposits and geological implications are not discussed by the authors.

### ***Sediments related to fault activity***

Optical dating can be used to determine the activity of tectonic faults. The average displacement rate can be established from the depositional age of a stratigraphic unit and the magnitude of the displacement. The timing of earthquakes can be determined by dating colluvial or slope-wash deposits on the hanging wall. Several studies in the Netherlands have taken these approaches.

Frechen and Van den Berg (2002) attempted to date sediments from the colluvial wedge of the hanging wall around the Peel boundary fault near Neer (southern Netherlands) using MAAD IRSL and TL methods in order to establish the timing of Late Weichselian and Holocene earthquake events. According to the authors, the colluvial deposits accumulated rapidly and were not sufficiently exposed to sunlight prior to deposition to completely reset the IRSL signal. As a consequence the ages obtained on these deposits ( $12.0 \pm 1.2$  and  $6.9 \pm 0.7$  ka) are interpreted as maximum ages of the deposits, and the authors suggest that an earthquake event probably occurred during the Middle or Late Holocene. Surprisingly, TL ages obtained on the same samples were similar to the IRSL ages whereas one would expect gross age overestimation for the TL method when light exposure was limited.

Houtgast et al. (2003) applied quartz OSL SAR dating to sediments around the Geleen Fault in the southern Netherlands. The Geleen Fault is part of the Feldbiss Fault Zone in the Roer Valley Rift System. A trench was dug to expose the fault and allow the researchers to investigate the sedimentology and the chronology of the deposits. Samples were collected from local slope-wash deposits of the hanging wall and a single sample from aeolian deposits at the top of the sequence. Two samples from the lowermost unit of slope wash deposits showed a large age reversal and, therefore, were rejected by the authors. However, although stratigraphically inconsistent, the dates support the assumed Weichselian Pleniglacial age. The deposits above this unit all gave optical ages of ~15 ka which provided evidence for rapid successive deposition of these deposits. Based on their investigation, the authors propose that at least one earthquake event occurred at ~15 ka and was followed by a period of high fault displacement rate.

In a similar trenching study of the Feldbiss Fault (Roer Valley Rift system, southern Netherlands) Houtgast et al. (2005) applied quartz OSL SAR dating to establish the activity of this fault. For the lowermost samples only minimum ages (>112 and >89 ka) could be determined, likely due to the quartz OSL signal approaching saturation although this is not mentioned by the authors. Weichselian Late Pleniglacial ages were obtained for two samples from sandy slope-wash deposits. A single sample from the sandy loess cover indicated a Weichselian Late Pleniglacial to Lateglacial age. In their paper, the authors mention two additional samples for which OSL dating failed. No reasons for this failure were provided and no results are presented in the paper. Results of their study confirmed Weichselian activity of the fault, although age control was too limited to draw firm conclusions on whether or not the Feldbiss Fault showed the same activity phases as the Geleen Fault discussed above. The authors tentatively correlate the increase in activity of the main border faults of the Roer Valley Rift System around 10 - 15 ka to glacial unloading of the crust.

## Synthesis and future perspectives

We discussed luminescence dating results of over 200 samples from Netherlands' sediments in the previous sections. The tests and applications of quartz OSL dating clearly show the possibilities for geological use of OSL dating in a wide range of depositional environments and over a wide age range. We envisage that the use of optical dating will increase in coming years and will play an important role for providing chronologies for a wide range of applications. The increasing use of luminescence dating is illustrated by the more than 100 sediment samples that are now yearly processed by the Netherlands Centre for Luminescence dating (NCL). OSL dating results of the samples discussed in this review and all samples processed by the NCL are stored in an online database that is regularly updated (LumiD; [www.LumiD.nl](http://www.LumiD.nl)). In collaboration with TNO all luminescence dates on Netherlands' sediments will also be incorporated in the online databank of DINOLoket.nl.

Several methodological challenges are clear from the review of tests and applications of luminescence dating. The most important of these are discussed below together with expected methodological developments in these areas.

### Improved accuracy and precision for optical dating

It is difficult, if not impossible, to obtain an optical age with an overall uncertainty of less than ~5% of the age (Murray and Olley, 2002; Vandenberghe et al., 2004). This overall uncertainty arrives from a combination of systematic and random effects. Sources of systematic uncertainty include calibration of the beta source (~1%, Bos et al., 2006), conversion of radionuclide concentrations to dose rates (~3%), calibration of concentration measurements (~3%) and the beta attenuation factor (~2%). Additional sources of systematic uncertainty are those associated with the cosmic radiation dose rate and water and organic contents during geological burial. Random uncertainties are associated with the measurement of equivalent doses and radionuclide concentrations (both arising from counting statistics). No significant reduction of the size of both random and systematic uncertainties is to be expected in the next few years and, consequently, neither is a substantial increase in the precision of optical dating.

Some studies show internal inconsistencies in the quartz optical dating results (e.g. Wallinga et al., 2004; Schokker et al., 2005). Some of these may be caused by inadequate sampling for dose-rate estimation. Especially for bedded sediments the radionuclide concentration may vary considerably with depth, and the dose rate derived from the sample collected for dose rate analyses may not be representative of that effectively experienced by the grains used for the luminescence measurements. In particular near lithological transitions this may lead to inaccurate dating results (e.g. Aitken, 1985). To avoid such problems, samples should be taken from deposits that are

homogeneous in a sphere of ~30 cm. If this is not possible, the dose rate should be measured in-situ or, alternatively, modeled based on the layer to layer variations in the radionuclide concentrations.

### Poorly-bleached deposits

Overestimation of the burial age due to incomplete resetting of the quartz OSL signal has been reported for samples from coastal dune (Ballarini et al., 2003), fluvial (e.g. Wallinga et al., 2001), colluvial / slope wash (e.g. Houtgast et al., 2005) and fimic soil (Bokhorst et al., 2005) settings. Especially very young deposits are easily affected by poor bleaching as for these the effects of some residual trapped charge at the time of deposition will be most pronounced.

The most promising development for the dating of poorly-bleached deposits is the measurement of individual grains. Analysis of the equivalent-dose distribution of individual grains allows the selection of well-bleached grains for age estimation (Lamothe et al., 1994; Murray and Roberts, 1997; Olley et al., 1999). Although successful applications of such methods have been reported (e.g. Olley et al., 2004), there is still a large debate among specialists how the large amount of data generated by these analyses should be processed and interpreted. The use of age models as proposed by Galbraith et al. (1999) is promising, but these can not be applied to very young deposits where the measured equivalent dose distributions overlap zero dose. For young deposits, Ballarini et al. (2007a) developed a modified SAR protocol. Using this protocol they obtained ages in agreement with independent age information on 300 years old coastal dune sands from Texel, but results were less precise than those obtained using conventional methods. Application of the procedure to a poorly-bleached dune sand of only one year old showed similar age overestimation as obtained with single aliquot methods (Ballarini et al., 2007b).

We envisage that in the next few years better methods will be developed to measure and analyze single-grain equivalent-dose distributions. The Netherlands Centre for Luminescence dating will play an active role in this development. We plan to develop single-grain methods to accurately date fluvial deposits from embanked floodplains as well as storm surge deposits in the western Netherlands, both formed during the past millennia to decades.

### Extending the age range

One other obvious challenge is the dating of deposits beyond ~125 ka. The quartz OSL signal usually saturates around this age, although the age range can be longer for deposits with low dose rates (e.g. Schokker et al., 2005). Quartz OSL ages obtained using the dose range where the OSL signal is close to saturation tend to underestimate the burial age (e.g. Wallinga



et al., 2007). For some samples, the age underestimation problems may be related to slight contamination of the quartz extracts with feldspar. This will lead to age underestimation if the feldspar is affected by anomalous fading and will be most pronounced for older samples where the quartz OSL signal approaches saturation. This problem may be prevented by applying the infrared wash methodology proposed by Wallinga et al. (2002).

Some improvement may be expected from methodological advances in sensitivity change correction for quartz equivalent-dose determination. These would allow accurate equivalent dose determination in the region of the dose response curve that approaches saturation. However, these improvements are not expected to push quartz OSL dating further back in time. Further extension may be possible through the use of a recuperated OSL signal from quartz as has recently been reported for Chinese loess (Wang et al., 2006).

An alternative approach is the use of feldspar because the feldspar IRSL signal saturates at far higher doses than the quartz OSL signal (Fig. 4). Unfortunately, several studies have reported that feldspar IRSL ages underestimate the burial age of deposits (e.g. Wallinga et al., 2001; 2007). So far, no reliable feldspar luminescence-dating procedure for Netherlands' deposits has been developed. Newly developed radioluminescence dating methods using feldspar (e.g. Trautmann et al., 1999), or use of pulsed IR stimulation (Tsukamoto et al., 2007) could open up new possibilities. However, it should be noted that these techniques may be (indirectly) affected by anomalous fading as well (Wallinga et al., 2007).

## Conclusions

Luminescence dating allows earth scientists to determine the time of deposition of sediments formed during the last glacial cycle and sometimes beyond that. Accuracy of luminescence dating techniques has been greatly improved over the last decades, and applicability of the method has widened. TL dating of sediments proved to be problematic in many respects. OSL dating of sand-sized grains of quartz using the single-aliquot regenerative dose (SAR) method is the luminescence dating method of choice both in terms of accuracy and precision. A number of tests on Netherlands' sediments showed that quartz OSL dating results agreed with independent age information for samples ranging in age from a few years to ~125 ka. Quartz OSL dating methods have been successfully applied in the Netherlands to various types of sediments and over a wide age range. Remaining challenges are the dating of sediments with limited light exposure prior to deposition and the development of accurate methods for the dating of sediments deposited before the last glacial cycle (> 125 ka).

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