

# ZEROING OF THE OSL SIGNAL AS A FUNCTION OF GRAIN SIZE: INVESTIGATING BLEACHING and THERMAL TRANSFER FOR A YOUNG FLUVIAL SAMPLE

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**Abstract:** Previous studies have indicated that the OSL signal from coarser grain-size fractions might be more completely reset during fluvial transport. In this study we investigate whether this feature is real, or rather an artefact of thermal transfer effects that might affect finer grains more due to smaller thermal lag during heat treatment. Our experimental results obtained on three grain-size fractions (90-125, 180-212 and 425-500  $\mu\text{m}$ ) clearly show that better bleaching of coarser grains is not caused by differences in thermal transfer. On the basis of our results we advise selecting a coarse grain size for analysis when dating young fluvial deposits.

## 1. INTRODUCTION

Optical dating allows determination of the timing of deposition and burial of sand and silt, providing that the material was exposed to light prior to burial (Aitken, 1998). Accuracy and precision of optical dating has been improved over recent years through development of novel methods and procedures for equivalent-dose determination on quartz separates (Murray and Wintle, 2000) and through improved equipment (Bøtter-Jensen *et al.*, 2000). Murray and Olley (2002) provide a review of the accuracy and precision attainable.

Application of optical dating to sediments deposited in the last few hundred years is of special interest, as the precision of radiocarbon dating in this age range is extremely poor due to plateaux in the calibration curve. However, optical dating in this age range can be problematic if light exposure prior to burial was insufficient to completely remove charge from the optically stimulated luminescence (OSL) traps used for dating. Such is especially likely in non-aeolian environments where light exposure prior to burial is often limited. Incomplete removal of charge from OSL traps in some or in all grains may result in an overestimation of the burial age; it is referred to as partial bleaching. Beside incomplete removal of charge from OSL traps, charge from light-insensitive traps may become mobilised during thermal treatment of the

sample prior to measurement of the OSL signal. Part of the mobilised charge may be re-trapped in the OSL traps used for dating. In a study of glacio-fluvial deposits from Greenland and the Himalayas, Rhodes (2000) demonstrated that such re-trapped charge may cause significant overestimation of the equivalent dose.

Optical dating is increasingly used to provide age control in studies of fluvial deposits (see Wallinga, 2002b for review). Rivers are important geomorphic agents, and much research is being carried out to investigate the rate at which fluvial processes occur and the recurrence interval of dramatic events (e.g. Kale *et al.*, 2000). However, partial bleaching and thermal transfer may affect optical dates for young fluvial deposits. Many researchers have discussed ways to detect partial bleaching (e.g. Clarke *et al.*, 1999; Colls *et al.*, 2001; Wallinga, 2002a), and methods for obtaining a burial age from scattered equivalent-dose results (e.g. Olley *et al.*, 1998, 1999). Avoiding partial bleaching has received far less attention in the literature because many researchers feel that partial bleaching cannot be completely avoided. Although this might well be true, the effect of partial bleaching on the equivalent dose obtained can and should be minimised for samples that were exposed to little light prior to burial. Opportunities to do so include: 1) selecting the best-bleached grain size for analysis, and 2) using a less-stringent thermal treatment to avoid thermal transfer.

Information available on the degree of bleaching as a function of grain size indicates that, for sand-sized grains, coarser fractions are better bleached (Olley *et al.*, 1998; Colls *et al.*, 2001; Wallinga, 2002b). Better bleaching of coarser grain sizes is surprising, seeing that coarser grains are more likely to be transported as bed load, i.e. at greater depths than the finer grains that may also be transported as suspended load. Therefore coarser grains may be expected to be exposed to less light during fluvial transport (see Wallinga, 2002b).

In this paper we investigate whether differences in heat treatment experienced by grains of different sizes prior to measurement may cause differences in equivalent doses obtained. The rationale behind our investigation is that grains might not always be heated to the desired temperature due to thermal lag between the grains and the thermal plate in the luminescence reader (hotplate). Especially when short preheats are used (typically 10 seconds), the temperature in the grains might be much lower than that of the hotplate. The difference between the desired preheat temperature and the actual temperature of the grains is expected to increase with grain mass, i.e. it will be largest for coarser grains. As a consequence, for a given preheat temperature (of the hotplate) the equivalent dose obtained on finer grains might be affected by thermal transfer while the equivalent dose obtained on a coarser grain-size fraction is not. If such is the case, the apparent better bleaching of coarser grain sizes might be an artefact of the (accidental) avoidance of thermal transfer effects.

The aim of this paper is to establish whether the apparent better bleaching of coarser grain sizes is real or an artefact of different thermal treatments due to thermal lag. We carry out experiments on three different grain-size fractions from a sample taken from 300-year-old fluvial channel deposits from the River Waal in The Netherlands. Our goals are: 1) to establish the degree of bleaching as a function of grain size; 2) to quantify thermal transfer effects on the equivalent dose as a function of grain size; 3) to determine whether apparent grain size dependency of the degree of bleaching is an artefact of thermal transfer effects.

## 2. STUDY AREA AND SAMPLES

The Holocene Rhine-Meuse delta is located in The Netherlands, in the south-central part of the North Sea Basin (Fig. 1, see Berendsen and Stouthamer, 2000). The sample used for this study was collected from a fluvial channel deposit from the River Waal, the primary distributary of the River Rhine, which has been active since about 2150 years cal.  $^{14}\text{C}$  BP (Berendsen and Stouthamer, 2000; 2001). We took a sample from point-bar deposits at 2.75 m below the surface. According to historical maps, point bars at the sample location developed between AD 1688 and AD 1723 (Fig. 2; Middelkoop, 1997), giving an age of about 300 years. We chose not to use a modern sample for this study, because sediment transport mechanisms in the present-day system have been modified through locks and waterworks resulting in a different bleaching environment.

The sample was water washed, treated with HCl and  $\text{H}_2\text{O}_2$  and then sieved to different grain-size fractions. Three grain-size fractions (90-125, 180-212 and 425-500  $\mu\text{m}$ ) were density separated at  $2.58 \text{ g cm}^{-3}$ ; the denser fraction was treated with 40% HF for 40 minutes, and then washed with HCl to obtain clean quartz grains. Purity of the samples was checked using measurement of the OSL response to infrared stimulation.

## 3. EQUIPMENT AND PROCEDURES

The measurements were made on an automated Risø TL/OSL reader, using blue light-emitting diodes (LEDs) emitting at  $450 \pm 30 \text{ nm}$  and an infrared laser diode emitting at  $830 \text{ nm}$  (Bøtter-Jensen *et al.*, 2000). The reader was equipped with an internal  $^{90}\text{Sr}/^{90}\text{Y}$   $\beta$ -source, giving a dose of  $0.028 \text{ Gy s}^{-1}$  to samples mounted on stainless-steel discs. The SAR procedure (Murray and Wintle, 2000) was used for equivalent-dose determination. Optical stimulation by blue LEDs was for 20 s at  $110^\circ\text{C}$  for all measurements; the OSL signal was detected through 7 mm of Schott U-340 filters (detection window 250-390 nm). The OSL signal detected during the first 0.16 seconds of stimulation was used for calculations after subtraction of the background signal derived from the OSL signal measured during the last two seconds of stimulation. For the test-dose signal, the last two seconds of the previous stimulation was used for background subtraction (Murray and Wintle, 2000).

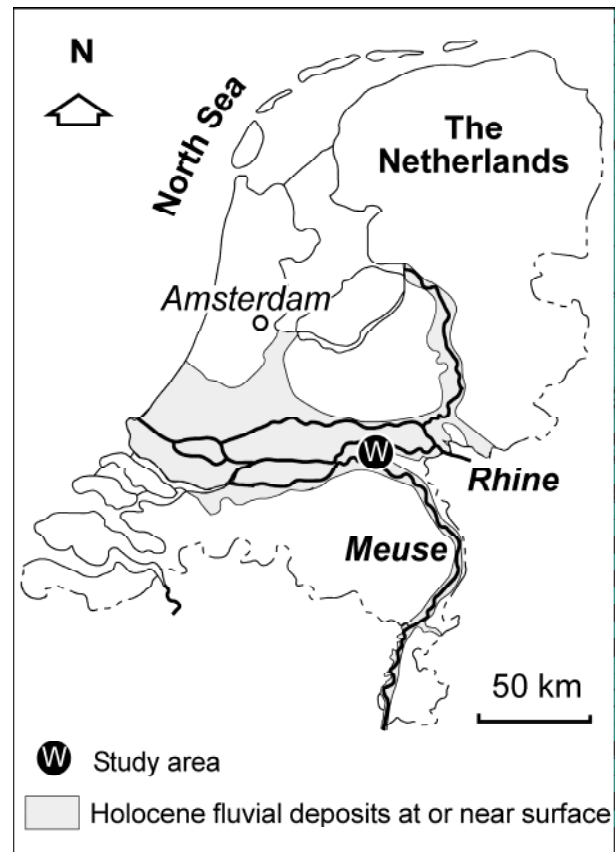
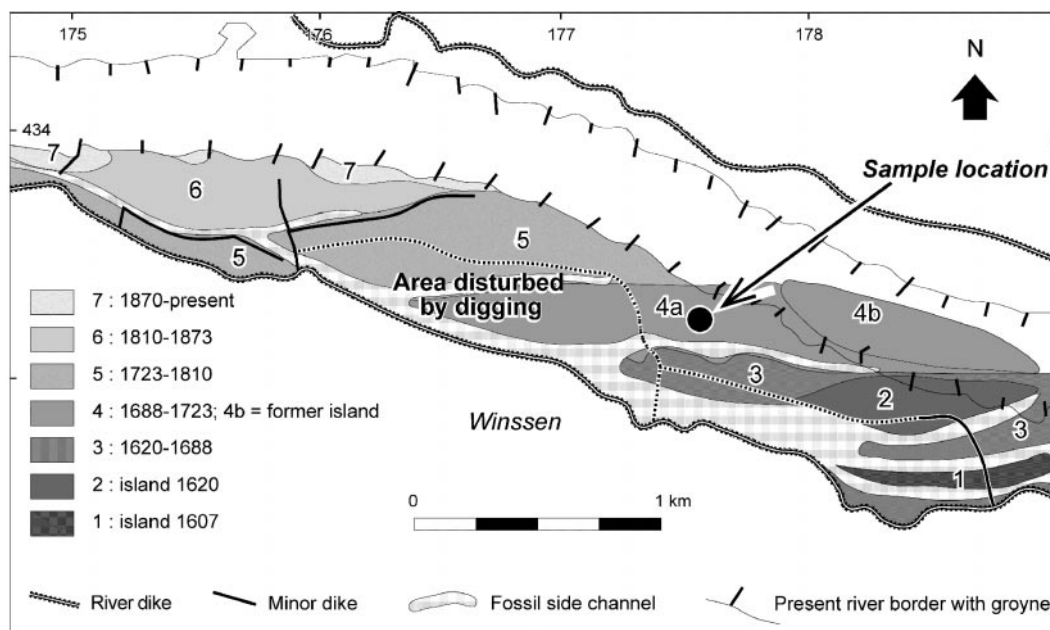


Fig. 1. The Rhine-Meuse Delta in The Netherlands and location of the sampling site.



**Fig. 2.** Sampling site and independent age control (local co-ordinates of sampling site: 177.560; 433.260, depth 2.75m; figure redrawn from Middelkoop, 1997).

A standard SAR procedure was followed, incorporating three regenerative doses, followed by a zero dose (to estimate the recuperation signal) and a repeated regenerative dose (to test whether our procedure satisfactorily corrects for sensitivity changes). We chose to repeat the second regenerative dose, because the dimness of the OSL signal resulting from the small first regenerative dose would have resulted in noisy recycling ratios. A relatively large test dose of 5.6 Grays was used to obtain a brighter test-dose response. The test dose was heated to a low temperature of 120°C, to allow measurements over a wide preheat range whilst still applying a more stringent heating to the regenerative doses than to the test doses prior to measurement.

The wide range of regenerative doses (0.6 to 22.4 Gy) was chosen to allow equivalent dose determination through interpolation even for aliquots with high equivalent doses. For aliquots where the natural OSL intensity was smaller than that from the first regenerative dose, we used interpolation between the origin and the first regenerative-dose point.

The natural dose rate of the sample was estimated using high-resolution gamma-spectroscopy (Murray *et al.*, 1987) on material obtained from just above the sample used for equivalent-dose measurements. The sample has been saturated with water since deposition, which reduces the dose rate experienced by the sample. Due to grain-size dependency of the grain-size attenuation, the dose rates for the different grain-size fractions differ slightly (Table 1). Radionuclide concentrations for the Winssen sample are presented in Wallinga *et al.* (2001).

**Table 1.** The Winssen sample used for this study.

Grain size ( $\mu\text{m}$ )	Natural dose rate (Gy/ka)	Independent age (year)	Expected burial dose (Gy)
90-125	$1.30 \pm 0.05$	$297 \pm 17$	$0.39 \pm 0.03$
180-212	$1.27 \pm 0.05$	$297 \pm 17$	$0.38 \pm 0.03$
425-500	$1.19 \pm 0.07$	$297 \pm 17$	$0.36 \pm 0.03$

#### 4. EXPERIMENTS

In our first experiment we aim at quantifying the dependency of the equivalent dose on the grain-size fraction used for analysis. The degree of scatter observed in equivalent doses obtained on individual aliquots of a heterogeneously bleached sample largely depends on the number of grains per aliquot (Li, 1994; Wallinga, 2002a). As we want to compare results obtained on the different grain-size fractions, we aimed at using an equal number of grains on each aliquot. In order to mount 200 to 250 grains per aliquot, we covered the centre 2, 3, or 8 mm of the disc for the finest, medium and coarse fraction, respectively. We measured 16 aliquots for each grain-size fraction. Experimental procedures are outlined in Table 2.

In the second experiment, we repeat the SAR procedure described above using a range of preheat temperatures (10 s at 125°C to 300°C, in steps of 25°C) on the three grain-size fractions (Table 2). Three aliquots are measured for each temperature and grain-size fraction. For this and subsequent experiments we covered the centre three millimetre of the discs with material, resulting in aliquots consisting of about 600, 200 and 40 grains for the finest, medium and coarse grain size, respectively. We chose to cover a three-millimetre area to obtain a usable OSL signal intensity without using too much of our sample material. For the finer grain-size fraction increasing the number of grains per aliquot has the added advantage that inter-aliquot scatter is somewhat reduced.

We developed a third experiment to investigate whether the dependency of equivalent dose on preheat temperature used is a result of thermal transfer. In this experiment we take four natural aliquots from each grain-size fraction. Prior to the measurement routine, we empty the OSL traps by optical bleaching using blue LEDs (20s at 110°C). We then submit each aliquot to seven SAR cycles without applying regenerative doses (see Table 2 for experimental details). In the first SAR cycle, we use a preheat temperature of 125°C, and in subsequent cycles

**Table 2.** Experimental procedures

Step	Experiment 1 Dependency of $D_e$ on grain-size fraction	Experiment 2 Dependency of $D_e$ on PH	Experiment 3 Quantify thermal transfer (TT)	Experiment 4 Quantify TT from test dose	Observed
Pre	–	–	Optical bleaching	Thermal & optical bleaching	
1	Regenerative dose <sup>1</sup>	Regenerative dose <sup>2</sup>	–	–	
2	Preheat (10 s at 150°C)	Preheat (10 s at 125 - 300°C)	Preheat <sup>3</sup> (10 s at 125 - 275°C)	Preheat <sup>3</sup> (10 s at 125 - 275°C)	
3	OSL natural/regenerative	OSL natural/regenerative	OSL natural/regenerative	OSL natural/regenerative	$L_i$
4	Test dose (5.6 Gy)	Test dose (5.6 Gy)	Test dose (5.6 Gy)	Test dose (5.6 Gy)	
5	Cut heat (120°C)	Cut heat (120°C)	Cut heat (120°C)	Cut heat (120°C)	
6	OSL test dose	OSL test dose	OSL test dose	OSL test dose	$T_i$
7	Repeat step 1-6 <sup>1</sup>	Repeat step 1-6 <sup>2</sup>	Repeat step 2-6 <sup>3</sup> Using different preheat	Repeat step 2-6 <sup>3</sup> Using different preheat	
8			SAR routine <sup>4</sup>		

<sup>1)</sup> No dose applied in the first cycle for measurement of the natural. Regenerative doses used in subsequent cycles: 0.6, 5.6, 28, 0 (recuperation measurement), 5.6 (recycling measurement) Gy. Sixteen aliquots were measured for each grain-size fraction.

<sup>2)</sup> No dose applied in the first cycle for measurement of the natural. Regenerative doses used in subsequent cycles: 0.6, 5.6, 22.3, 0 (recuperation measurement), 5.6 (recycling measurement) Gy. Three aliquots were measured for each grain-size fraction at each preheat temperature.

<sup>3)</sup> The preheat temperature applied is increased with 25°C in each cycle, i.e. in the first cycle a preheat temperature of 125°C is applied, in the second 150°C etc. up to 275°C in the seventh cycle. Measurements are made on three aliquots for each grain-size fraction.

<sup>4)</sup> A SAR dose-response curve was constructed using regenerative doses of 1.1, 3.3 and 6.7 Gy on the three discs per grain-size fraction. We checked recycling through repeating the second regeneration point.

we increase the preheat temperature with 25°C each time, until the maximum preheat temperature of 275°C is reached in the seventh cycle. Because we do not give any regeneration doses, any OSL signal detected in the OSL measurement of step 3 (see **Table 2**) is expected to be caused by thermal transfer. The test dose administered in each cycle is used to monitor and correct for sensitivity changes during the measurement procedure. After completion of seven SAR cycles without regenerative doses, a SAR dose-response curve is created using a preheat temperature of 175°C.

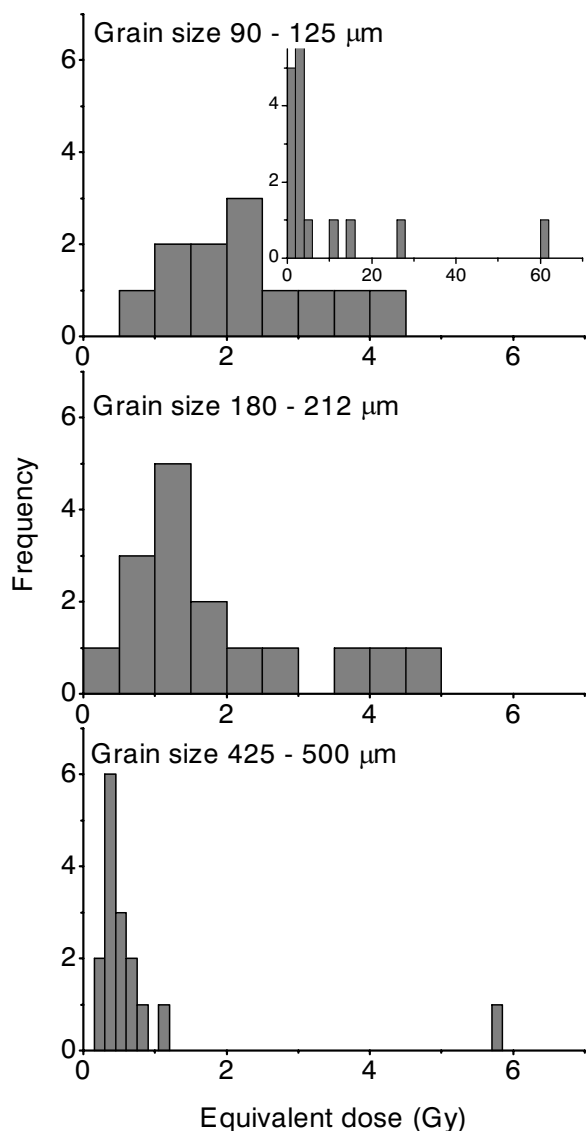
The thermal transfer as obtained in the third experiment might be slightly overestimated due to a contribution of thermal transfer from the test doses. In a standard SAR procedure such as experiment 2, thermal transfer from a test dose may slightly add to the subsequent OSL measurement of the regenerative dose. This could theoretically lead to a minor underestimation of the equivalent dose, but effects are likely to be negligible. However, in the case of our experiment 3 any contribution from thermal transfer from the test doses over the seven SAR cycles will be added up and might considerably add to our estimation of the effects of thermal transfer on the equivalent dose estimation. To quantify this effect, a fourth experiment was carried out (**Table 2**) in which the third experiment was repeated on the same discs. The difference with the third experiment is that all thermally unstable traps are now empty at the start of the experiment. Hence there will be no thermal transfer from the natural dose, i.e. all observed thermal transfer must arise from the test doses applied to the samples during the experiment.

## 5. EXPERIMENTAL RESULTS

Dose distributions as a function of grain size (experiment 1) are shown in **Table 3** and **Fig. 3**. Results on the finest fraction are widely scattered, and both the mean and the median equivalent doses on this fraction are far greater than the expected value (**Table 1**). Scatter in equivalent doses obtained on the medium fraction is less, and the mean and median equivalent doses are lower for this fraction. The tightest dose distribution and the lowest mean and median equivalent doses are obtained on the coarsest grain-size fraction, even though the standard deviation and standard error are affected by a single outlier at about 6 Gy. The median equivalent dose for the coarsest grain-size fraction (0.46 Gy) is close to the expected value (0.36 Gy).

Equivalent doses obtained on the three grain-size fractions as a function of preheat temperature (experiment 2) are shown in **Fig. 4**. Differences in equivalent doses are again found for the different grain-size fractions. Furthermore, for the medium and coarse grain sizes, an increase in equivalent dose is observed at preheat temperatures above 200°C. This trend is likely a result of charge transfer during heating, and is further investigated in the subsequent experiments.

In our third experiment we quantify the offset in equivalent dose caused by thermal transfer. In consecutive SAR cycles four aliquots of each grain-size fraction were heated to higher temperatures and the OSL signal resulting from heating over that temperature interval recorded. We projected the test-dose corrected OSL signal



**Fig. 3.** Equivalent dose distributions obtained on three different grain-size fractions of the Winssen sample (experiment 1, Table 2). Each aliquot was mounted with 200-250 grains. Sixteen aliquots were measured for each grain-size fraction. Only for the finest grain size were equivalent doses above 6 Gy obtained on some aliquots; these are shown in the inset of the top graph.

on the SAR dose-response curve obtained after completion of the experiment. Thereby we obtained a quantitative offset in equivalent dose due to thermal transfer for that preheat-temperature interval. To obtain an offset in equivalent dose due to thermal transfer for a given preheat temperature, we sum the offsets for all preheat-temperature intervals up to that temperature. The linear nature of the dose-response curve in the dose region of interest allows us to do so. Results of this exercise are presented in Fig. 5.

To correct results of the third experiment for thermal transfer arising from the test doses, the experiment was repeated after thermally emptying all traps (experiment 4). Results (Fig. 5) show that this contribution is significant, especially for the coarser grain size.

**Table 3.** Equivalent doses obtained on different grain-size fractions (experiment 1)

Aliquot number	Equivalent dose (Gy)		
	Finest 90-125 µm	Medium 180-212 µm	Coarse 425-500 µm
1	1.98	0.87	0.49
2	2.84	0.35	0.29
3	1.90	1.98	1.17
4	3.40	3.98	0.55
5	11.05	1.02	0.41
6	2.15	2.64	0.74
7	1.03	4.04	5.74
8	3.95	1.13	0.38
9	0.99	1.19	0.61
10	2.07	1.09	0.28
11	15.06	0.76	0.43
12	2.11	4.78	0.38
13	60.47	2.48	0.44
14	1.02	1.83	0.82
15	27.09	0.88	0.48
16	4.29	1.18	0.39
Mean	8.84	1.89	0.85
sd	15.42	1.34	1.32
se	3.86	0.33	0.33
Median	2.50	1.19	0.46
	Age (years)		
Mean ± se	6800 ± 1720	1490 ± 380	710 ± 180 (440 ± 120) <sup>1</sup>
Median	1920	930	390 (370) <sup>1</sup>

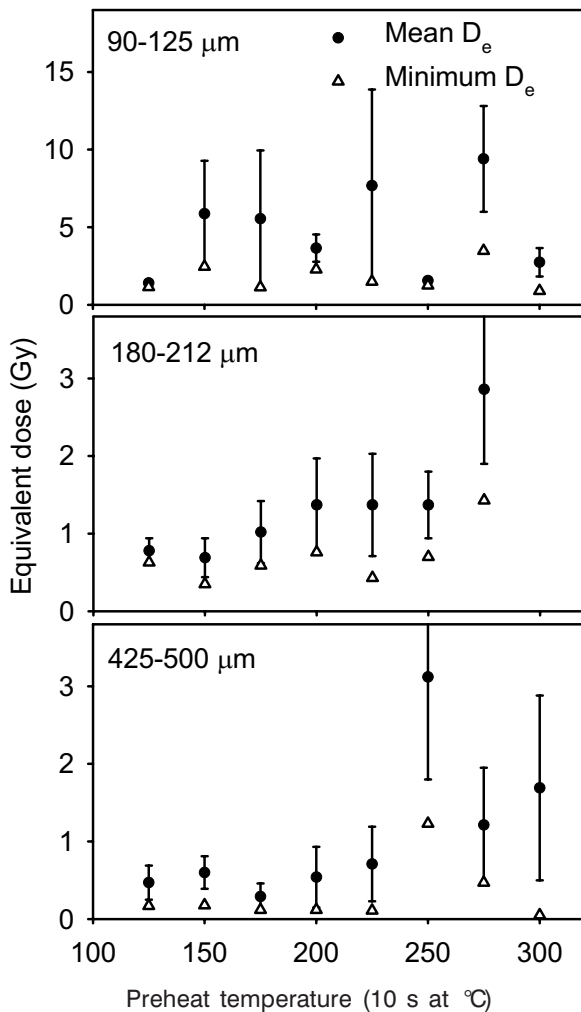
<sup>1</sup>) When omitting the single outlier at 6 Gy (disc 7).

## 6. DISCUSSION

### *Thermal transfer effects on the equivalent dose*

From experiments 3 and 4, we calculate the offset due to thermal transfer (TT) from the natural only ( $TT_{\text{natural}} = TT_{\text{total}} - TT_{\text{test dose}}$ ). Subtracting this from the equivalent doses obtained in experiment 2 gives an equivalent dose corrected for thermal transfer. Results (Fig. 6) indicate that after this correction, no clear dependency of equivalent dose on preheat temperature is found for any of the grain-size fractions, although inter-aliquot scatter remains greater for higher preheat temperatures.

For the finest grain-size fraction, results are widely scattered and thermal transfer is shown to contribute only marginally to the large offsets in equivalent dose observed for some aliquots. For the medium grain-size fraction the dependency of equivalent dose on preheat temperature is largely explained by greater thermal transfer for the higher preheat temperatures; the rising trend in equivalent doses as a function of preheat temperature is largely removed by correction for thermal transfer. However, even after such correction, equivalent doses are greater than those expected for this deposit by a factor of two.

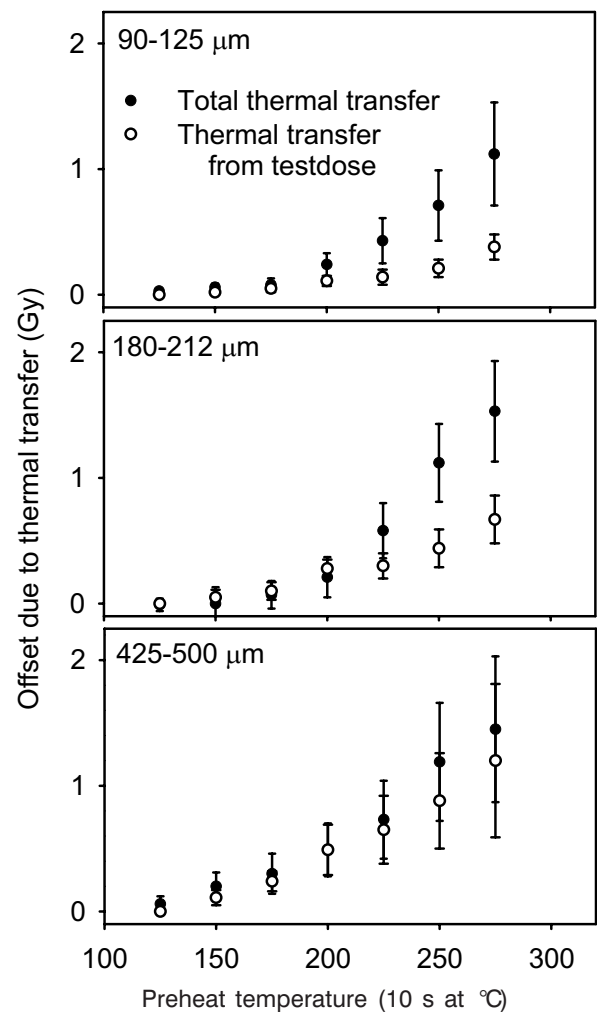


**Fig. 4.** Equivalent doses on three grain-size fractions as a function of preheat temperature applied (experiment 2, Table 2). Note the different scale for the top graph. Three aliquots are measured for each grain size and preheat combination. The mean equivalent dose and standard error on the mean are shown, as well as the minimum equivalent dose obtained on the three aliquots.

For the coarse grain-size fraction a flat preheat plateau is found up to preheat temperatures around 225°C. Thermal transfer is found to be negligible up to this level. For preheat temperatures of 250°C and higher the scatter increases, possibly due to aliquot-dependent thermal transfer. Also note that if we had not taken into account the thermal transfer from the test doses for our estimation of thermal transfer, correcting equivalent doses for thermal transfer would have resulted in near zero equivalent dose values for the coarsest grain-size fraction.

#### Can thermal transfer explain the grain-size dependency?

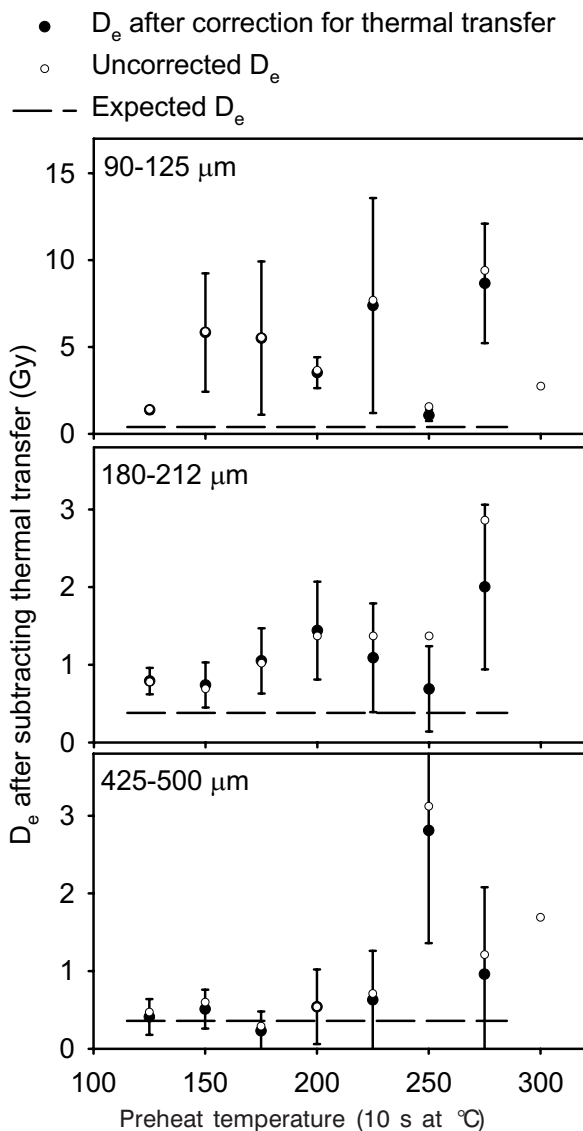
In the introduction we hypothesised that differences in equivalent dose obtained on different grain-size fractions might be an artefact of lower preheat temperatures experienced by coarser grains due to thermal lag. Based on the experiment results presented in this paper we conclude that the dependency of equivalent dose on grain-size fraction used is due to differences in the degree of



**Fig. 5.** Offset in equivalent dose due to thermal transfer as a function of preheat temperature used (experiment 3, Table 2), and the thermal transfer arising from the test doses used in experiment 3 (experiment 4, Table 2). Subtracting the thermal transfer from the test doses from the total thermal transfer gives the thermal transfer from the natural.

bleaching rather than to thermal transfer. The evidence for this conclusion is threefold:

- 1) Experimental results show no indications for different thermal lag; thermal transfer occurs at similar preheat temperatures for the three grain-size fractions (Fig. 5).
- 2) Equivalent doses are not affected by thermal transfer up to preheat temperatures of 200°C for 10 seconds (Fig. 5, 6). Thereby we have demonstrated that differences in equivalent doses obtained on the three grain-size fractions using a 150°C preheat (experiment 1) cannot be explained by differential thermal transfer effects.
- 3) Comparison of Figs. 4 and 5 shows that inter-aliquot scatter is greatly reduced by optical bleaching, and that equivalent doses obtained after light exposure are very small. This suggests that the offsets for the finer grain-size fractions are caused by inadequate light exposure prior to deposition and not by thermal transfer.



**Fig. 6.** Equivalent doses after correction for thermal transfer following the procedure described in the text. Uncorrected equivalent doses are shown for comparison. Note that thermal transfer effects are negligible up to preheat temperatures around 200°C. Also note the different scale for the top graph.

It is important to note that no clear correlation was found between the amount of thermal transfer and the degree of bleaching of a sample. Hence the absence or existence of thermal transfer for a given sample does not yield reliable information on the degree of bleaching of that sample.

#### *Why are coarser grains better bleached?*

Optical ages obtained on the finest and medium grain size overestimate the burial age of the deposit, whereas optical ages obtained on the coarsest grain-size fraction are in agreement with the independent age if the single outlier from experiment 1 is omitted (Table 3, Figure 6). The data shows that the median is in better agreement with independent age control, as is to be expected for a skewed distribution on a poorly-bleached sample. Moreover, the median is less affected by the single outlier than the mean of the distribution (Table 3).

This study reinforces previously published results that coarser grain-size fractions in fluvial systems may be better bleached (Olley *et al.*, 1998; Colls *et al.*, 2001). Reasons for this are still unclear, but are most likely to lie in different transport mechanisms for the different grain sizes. Possible reasons include: 1) Better light exposure of coarse grains on e.g. mid-channel bars; 2) Shorter burial periods for coarser grains (leading to smaller equivalent doses prior to erosion); 3) Mud-coating of finest grains (preventing light penetration).

Single-grain experiments on different grain-size fractions are needed to obtain more insight in the dose distributions and identify reasons for the better bleaching of the coarser grain sizes. We plan to carry out such research in the near future.

In spite of our poor understanding of the reasons for better bleaching of the coarser grain-size fraction, researchers can make use of this feature when dating fluvial deposits. We advise selecting a relatively coarse grain-size fraction for optical dating of fluvial sediments.

## 7. CONCLUSIONS

Experiments on three different grain-size fractions (90-125, 180-212 and 425-500 μm) from a 300 year old fluvial channel deposits have shown that:

- thermal transfer is negligible up to preheat temperatures of 200°C for 10s
- the coarsest grain-size fraction is best bleached and gives a reasonable estimate of the burial age of the sample
- apparent better bleaching of the coarser grain size-fraction is not an artefact of thermal transfer effects

Although reasons for the more complete resetting of the OSL signals of coarser grains in fluvial sediments are not understood, our results suggest that using a coarser grain-size fraction may facilitate optical dating of fluvial deposits.

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