LUMINESCENCE DATING OF FLUVIAL DEPOSITS FROM THE WESER VALLEY, GERMANY

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Abstract: Luminescence dating was applied on coarse-grained monomineralic potassium-rich feldspar and polymineralic fine-grained minerals of five samples derived from fluvial deposits of the River Weser in northwestern Germany. We used a pulsed infrared stimulated luminescence (IRSL) single aliquot regenerative (SAR) dose protocol with an IR stimulation at 50°C for 400 s (50 µs on-time and 200 µs off-time). In order to obtain a stable luminescence signal, only off-time IRSL signal was recorded. Performance tests gave solid results. Anomalous fading was intended to be reduced by using the pulsed IRSL signal measured at 50°C (IR50), but fading correction was in most cases necessary due to moderate fading rates. Fading uncorrected and corrected pulsed IR50 ages revealed two major fluvial aggradation phases during the Late Pleistocene, namely during marine isotope stage (MIS) 5d (100 ± 5 ka) and from late MIS 5b to MIS 4 (77 ± 6 ka to 68 ± 5 ka). The obtained luminescence ages are consistent with previous 230Th/U dating results from underlying interglacial deposits of the same pit, which are correlated with MIS 7c to early MIS 6.

Keywords: pulsed infrared stimulated luminescence, fluvial deposits, independent age control, Late Pleistocene, Weser valley, northern Germany.

1. INTRODUCTION

Optically stimulated luminescence (OSL) dating was applied to fluvial deposits in order to give insights into the timing of fluvial aggradation and degradation (e.g. Wallinga, 2002; Busschers et al., 2008; Cordier et al., 2010; Lauer et al., 2010). The major difficulty in dating sediments by means of luminescence is mainly caused by the occurrence of insufficient bleaching of the luminescence signal, which is considered a great challenge for especially fluvial deposits (e.g. Murray et al., 1995; Gemmell, 1997; Olley et al., 1999; Stokes et al., 2001). In a fluvial environment, insufficient bleaching can be caused by different environmental conditions, such as water depth, transport distance, and the mode of transport. In the water column, sunlight is being attenuated and therefore generally hampers the probability for the transported minerals to be sufficiently bleached. Furthermore, rapid erosion and transport due to storm, high-discharge and flooding events may also limit the time needed for resetting the luminescence signal (cf. Wallinga, 2002; Jain et al., 2004; Rittenour, 2008). However, luminescence dating of fluvial deposits has been successfully applied in many case studies (Lewis et al., 2001; Wallinga et al., 2001; Rittenour et al., 2005; Briant et al., 2006; Choi et al., 2007; Busschers et al., 2008; Frechen et al., 2008, 2010; Krbetschek et al., 2008; Lauer et al., 2010, 2011). Lauer et al. (2011) compared the quartz and feldspar luminescence ages from fluvial sand samples from the River Rhine intercalated with the Laacher See tephra (12.9 ka). Both quartz and feldspar

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ages agreed perfectly with the independent tephra age, suggesting that insufficient bleaching, if any, might not be a problem for Pleistocene samples.

In order to check if the problem related to insufficient bleaching exists, one can perform measurements of multiple luminescence signals with different bleachable properties and compare the obtained results with each other. Such comparison is normally done using quartz and feldspar signals (e.g. Murray et al., 2012). The use of quartz minerals for luminescence measurements is often restricted to younger deposits (<70 ka; e.g. Fuchs and Lang, 2001; Lewis et al., 2001; Wallinga, 2002; Briant et al., 2006; Busschers et al., 2008) due to the lower saturation level of quartz (about 100–200 Gy). The quartz luminescence signal is much more light-sensitive, thus faster to bleach than the feldspar luminescence signal, but feldspar minerals allow for dating comparably older (fluvial) sediments (e.g. Krbetschek et al., 2008; Lauer et al., 2011) due to the higher saturation limit of the luminescence signal. Yet, feldspar minerals may suffer from a certain signal loss over time, referred to as anomalous fading (Wintle, 1973; Aitken, 1985; Spooner, 1994). When the quartz OSL signal cannot be used, equivalent doses or ages obtained from the infrared stimulated luminescence (IRSL) signal measured at low temperatures and the post-IR IRSL signal has also been used for comparison to evaluate the bleaching degree of a sample (Buylaert et al., 2013).

However, in order to identify the limits of different dating methods, including their uncertainties, and to calibrate the chronological framework, independent age control can be substantially helpful. Independent age control can be provided e.g. by additional radiocarbon (14C) dating (e.g., Thomas et al., 2006; Frechen et al., 2008; Murray et al., 2012), electron spin resonance dating (ESR; e.g., Molodkov, 2012; Zhao et al., 2012), amino acid racemization (AAR; e.g., Novothny et al., 2009) or uranium-thorium (230U/Th) dating (this study) of (i) the sediment itself or of (ii) the under- and/or overlying deposits, depending on the availability of appropriate dating material (e.g. organic matter in case of 14C dating). Given the fact that results of all applied dating methods are consistent with each other, the accuracy and reliability of the performed dating technique(s) can be proven.

In this study, we present new feldspar luminescence ages of fluvial deposits in northwestern Germany, which are supported by independent age control based on 230U/Th dating of underlying interglacial deposits. The obtained luminescence ages are of great importance as they shed new light on the previously established Middle to Late Pleistocene depositional model of the studied area.

2. STUDY AREA AND PREVIOUS RESEARCH

Study area

The study area is located in the southern Weser valley in northwestern Germany (Fig. 1A) and is characterised by up to 530 m high mountain ridges of the Central German Uplands (Fig. 1B). Here, the folded Variscan basement is unconformably overlain by Lower Permian red beds (‘Rotliegend’), Upper Permian marine evaporites and carbonates (‘Zechstein’), Lower Triassic sandstones (‘Buntsandstein’) and Middle Triassic shallow marine sediments (‘Muschelkalk’) (Lepper and Mengeling, 1990; Lepper, 1991). From the late Cretaceous to the Neogene, these sediments experienced uplift, which led to a subsequent incision of the River Weser that formed its isoclinal valley between the Buntsandstein anticlinal at its east and the steep cuestas of the outcropping Lower Muschelkalk at its west during the subsequent Neogene to Late Pleistocene (Grupe, 1912, 1929; Lepper, 1991). The Nachtigall pit is located at the western flank of the Buntsandstein anticlinal about 5 km southwest of Holzminden (Fig. 1B). The lowermost part of the sedimentary record, probably comprising Middle Pleistocene (Saalian) fluvial deposits of the River Weser (e.g. Rohde, 1989; Rohde et al., 2012), is not exposed in the studied Nachtigall pit but is assumed to occur at an altitude range from about 70–80 m a.s.l. (Rohde et al., 2012). Generally, the term “terrace” is geomorphologically defined as and associated with those deposits preserved above the present floodplain. In this paper, the terms “Older and Younger Middle Terraces” and “Lower Terraces” are used on a geochronological basis, referring to those fluvial
deposits that are considered to have been accumulated during the Middle and Late Pleistocene. Older and Younger Middle Terrace deposits are both considered to have been accumulated after the retreat of the Elsterian glaciation and prior to the advance of the Saalian Drenthe ice sheets (Middle Pleistocene), namely during the early Saalian (Older Middle Terrace) and during the late Saalian (Younger Middle Terrace). Deposition of the Lower Terrace is linked to the Weichselian glaciation (Late Pleistocene) (Rohde et al., 2012).

The unexposed fluvial deposits are referred to as Older Middle Terrace deposits and are overlain by 13–25 m thick fine-grained interglacial limnic and fen peat of the so-called Nachtigall-Complex. The Nachtigall-Complex ranges over an altitude of about 80–96 m a.s.l. (Rohde et al., 2012; this study). The interglacial deposits are unconformably overlain by 8 m thick coarse-grained fluvial sediments, occurring over an altitude range of 96–104 m a.s.l., deposited by a braided river system (Winsemann et al., 2015) (Figs. 2A and 2B). These fluvial deposits are referred to as Younger Middle Terrace deposits (e.g. Rohde, 1989; Kleinmann et al., 2011; Waas et al., 2011; Rohde et al., 2012).

In the western part of the pit, the lowermost 5 m of the braided river deposits (96–101 m a.s.l.) consist of gravel sheet deposits, which are overlain by up to 1 m thick fine-grained overbank deposits, consisting mainly of ripple cross-laminated and planar-parallel laminated silt and silty sand. These overbank deposits, which are intercalated with up to 0.4 m thick gravel sheet deposits, are truncated and overlain by about 2 m thick gravel sheet deposits (Winsemann et al., 2015) (Fig. 2A). The fluvial deposits in the western and eastern parts of the Nachtigall pit are separated by a major (erosional) bounding surface, characterised by a vertical erosion of about 9 m (Fig. 2B).
The fluvial sediments in the eastern part of the pit are at least 15 m thick and consist of channel belt and overbank deposits of a gravelly to sandy braided river system (Winsemann et al., 2015). Exposed fluvial deposits occur over an altitude range of about 90–103 m a.s.l. (Figs. 2A and 2B). Here, the lowermost part is characterised by about 2 m thick channel-fill deposits, passing upwards into lateral and downstream accretion macroforms as well as sandy bedforms, comprising planar-parallel stratified, planar or trough cross-stratified or ripple cross-laminated medium- to fine-grained sand. These deposits are truncated and overlain by about 4 m thick gravel sheets (Winsemann et al., 2015) (Fig. 2A). Locally, deposits are overlain by fine-grained floodplain deposits and draped by loess. The floodplain area of River Weser is expected to comprise Late Pleistocene (Weichselian) fluvial deposits (cf. Rohde et al., 2012). For further detailed information on the sedimentology of the Nachtigall deposits and the large-scale depositional architecture, which is being reconstructed from the outcrop section and digital elevation models, see Winsemann et al. (2015).

**Previous research**

Reconstruction of the fluvial terrace architecture of the River Weser is largely based on lithostratigraphy and morphology (Rohde, 1983, 1989, 1994). Up to 11 terrace levels were mapped, recording about 170 m of fluvial incision during the Pleistocene (Fromm, 1989; Rohde 1989, 1994).

The Nachtigall pit, which has long been exploited for brick production, must be considered as a key section for understanding fluvial sedimentation in the area. The deposits with other interglacial successions in Germany and France (e.g. Kleinmann et al., 2011). Studies dealing with the deposits of the Nachtgall pit go back to the 19th century and focused on the interglacial sediments (e.g. Dechen, 1884; Carthaus, 1886; Koken, 1901). The interglacial deposits were allocated either to the Holsteinian (based on pollen analysis; Gruepe, 1929) or to the Eemian (based on their stratigraphic position related to the Middle Terrace deposits; Siegert, 1912, 1921; Soergel, 1927, 1939). Much later, Mangelsdorf (1981) performed detailed palynological analysis on the interglacial deposits and proposed a late Cromerian age (Bilshausen/Rhume interglacial). Later pollen analysis of the interglacial sediments of the Nachtigall pit did not support such a late Cromerian age but tentatively pointed to a Saalian deposition (Lepper, 1998). Recently, 230U/Th dating and palynological studies on the interglacial limnic sediments support this finding and refer to a deposition during MIS 7c to early MIS 6 (227±8 ka to 177 ± 8 ka; Kleinmann et al., 2011; Waas et al., 2011). Based on these ages and stratigraphic relations, the underlying fluvial deposits were assumed to have been deposited during MIS 8 and are referred to as Older Middle Terrace deposits (Kleinmann et al., 2011; Rohde et al., 2012), whereas the overlying fluvial deposits were interpreted to have been deposited during MIS 6 (Kleinmann et al., 2011; Waas et al., 2011; Rohde et al., 2012) and form part of the so-called Middle Terraces that accumulated prior to the Saalian Drenthe glaciation.

So far, much research has been carried out on a lithostratigraphical and palynological basis. However, robust numerical ages only exist for the interglacial deposits and the 230U/Th ages published by Waas et al. (2011) only provide maximum ages for the overlying fluvial deposits. Reliable luminescence ages for the overlying fluvial sediments are still missing, thus hamper the establishment of a chronological framework for these deposits.

**3. METHODS**

**Sampling and preparation**

Five luminescence samples were taken in 2012 from the fluvial sediments of the Nachtigall pit (Figs. 2A and 2B). Samples NG1, NG2, NG3 and NG4 were taken from sandy bedform deposits from the eastern part of the Nachtigall pit, while sample NG5 was taken from overbank deposits from the westernmost part of the Nachtigall pit (Figs. 2A and 2B). The 230U/Th ages determined by Waas et al. (2011) were derived from interglacial deposits about 175 m northwest of sample NG1 (Fig. 2C).

Sampling and preparation was performed as described in Roskosch et al. (2015). For luminescence measurements, both monomineralic coarse-grained (150–200 µm) potassium-rich feldspar minerals and polymineralic fine-grained (4–11 µm) minerals were used (Table 1). For coarse-grained minerals, small-sized (2.5 mm) aliquots with about 100–120 grains were created by mounting coarse-grained minerals on 9.8 mm stainless steel discs with about 100–120 grains were created by mounting coarse-grained minerals on 9.8 mm stainless steel discs using silicone spray as an adhesive. Fine-grained minerals (~105 grains; Fuchs et al., 2005, 2013) were mounted on 9.8 mm aluminum discs from a suspension in acetone.

Sample preparation and luminescence measurements were performed at the Leibniz Institute for Applied Geophysics (Hannover, Germany). For luminescence measurements, an automated Risø TL/OSL reader (DA-20) with a calibrated 80Sr/90Y beta source (1.48 GBq = 40 mCi) was used (Bøtter-Jensen et al., 2010). Feldspar

<table>
<thead>
<tr>
<th>Sample</th>
<th>Lab num.</th>
<th>Longitudes E</th>
<th>Latitudes N</th>
<th>Depth b.s. (m)</th>
<th>Altitude a.s.l. (m)</th>
<th>Grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG1</td>
<td>2665</td>
<td>09°24'11.16&quot;</td>
<td>51°48'30.83&quot;</td>
<td>9.50</td>
<td>94.00</td>
<td>150–200</td>
</tr>
<tr>
<td>NG2</td>
<td>2666</td>
<td>09°24'11.16&quot;</td>
<td>51°48'30.83&quot;</td>
<td>9.00</td>
<td>94.50</td>
<td>150–200</td>
</tr>
<tr>
<td>NG3</td>
<td>2667</td>
<td>09°24'10.90&quot;</td>
<td>51°48'31.90&quot;</td>
<td>6.70</td>
<td>98.60</td>
<td>150–200</td>
</tr>
<tr>
<td>NG4</td>
<td>2668</td>
<td>09°24'09.13&quot;</td>
<td>51°48'31.49&quot;</td>
<td>6.30</td>
<td>98.70</td>
<td>150–200</td>
</tr>
<tr>
<td>NG5</td>
<td>2628</td>
<td>09°24'07.90&quot;</td>
<td>51°48'31.69&quot;</td>
<td>2.50</td>
<td>98.00</td>
<td>4–11</td>
</tr>
</tbody>
</table>

Table 1. Basic information on fluvial samples that were taken for luminescence dating using feldspar minerals.
signals were stimulated by pulsing by IR light-emitting diodes (LED) either using an external pulsing box (Thomsen et al., 2008a) or a pulsed stimulation attachment (Lapp et al., 2009). A Schott BG39/Corning 7–59 filter combination was used and the feldspar signals were detected in the blue-violet (320–460 nm) during the off-periods of each pulse cycle, with a delay of 5 µs after the LED pulses switched off.

**Equivalent dose and dose rate determination**

For equivalent dose ($D_e$) determination, 10 aliquots per sample were measured using a pulsed IRSL single aliquot regenerative (SAR) dose protocol (Table 2). A preheat at 250°C for 60 s was used, followed by a pulsed IR stimulation at 50°C for 400 s with 50 µs on-time and 200 µs off-time. Only off-time signal was recorded because it was found to give a stable luminescence signal (Tsukamoto et al., 2006). The pulsed IRSL signal at 50°C (IR$_{50}$) was chosen over the elevated temperature post-IR IRSL signal (pIRIR; Thomsen et al., 2008b) because it appears to be more sensitive to light. Comparison of both pulsed IR$_{50}$ and pIRIR$_{290}$ results showed that pIRIR$_{290}$ $D_e$ values were generally higher by about 100–150 Gy than the pulsed IR$_{50}$ ones (see Fig. 3B in Roskosch et al., 2015). Jain et al. (2015) compared the residual dose obtained from a modern beach sample using continuous wave (CW) IR$_{50}$, pulsed IR$_{50}$, pIRIR$_{225}$ and pIRIR$_{290}$ signals and a much larger residual dose of ~10 Gy was obtained from the pIRIR$_{290}$ signal than all the other signals (less than 2 Gy). This was probably caused by the hard to bleach nature of the pIRIR$_{290}$ signal, as has been supported by results of a bleaching study performed by Kars et al. (2014). Based on the above mentioned findings, we focused on the pulsed IR$_{50}$ signal for $D_e$ determination of the fluvial sediments of this study.

The net feldspar luminescence signal was then calculated from the middle part of the decay curve (21–60 s) after subtracting a late background of the last 50 s (see Roskosch et al., 2015). The initial part of the decay curve (0–20 s) was actually reported to give considerably higher fading rates (up to 4.42 ± 0.46%), whereas the middle part was found to show only negligible anomalous fading (see Roskosch et al., 2015).

Aliquots were accepted when they passed for following criteria: recycling ratio limit within 10% of unity; maximal test dose error 10%; signal intensity larger than 3 sigma above background. We assumed a measurement error of ±2.0%. In order to calculate $D_e$ values, dose response curves were fitted using a single-saturating exponential function.

For dose rate determination, the radionuclide concentration of uranium ($^{238}$U), thorium ($^{232}$Th) and potassium ($^{40}$K) was determined by high-resolution gamma spectrometry. For coarse-grained feldspar minerals, an internal potassium content of 12.5 ± 0.5% was assumed (Huntley and Baril, 1997). The a-value was set to 0.15 ± 0.05 for monomineralic coarse-grains (Balescu and Lamothe, 1994) and 0.08 ± 0.02 for polynomineralic fine-grains (Lang et al., 2003), respectively. Cosmic radiation was corrected for altitude and sediment thickness after Prescott and Hutton (1994). Water content was measured using samples from the direct surroundings of the luminescence samples, and was 7% (NG4), 9% (NG3, NG5), 10% (NG1) and 11% (NG2). Based on these values, the overall water content was then set to an average value of 10 ± 5% for both the coarse-grained braided river and the fine-grained overbank deposits. Dosimetry results are provided in Table 3.

**Performance tests**

Dose recovery experiments on three aliquots of each sample were performed prior to $D_e$ measurements to check for the suitability of the applied SAR protocol under laboratory conditions. Within the Risø TL/OSL reader, aliquots were bleached by IR diodes and then given a similar beta dose that was close to the natural expected one (271 Gy for samples NG1, NG2, NG3 and NG4, 401 Gy for sample NG5). Afterwards, the same
SAR protocol was applied to check if the given dose could be accurately recovered.

Recycling ratio tests were conducted by applying the same dose twice (namely at the beginning and at the end of the measurement). A recycling ratio value that is within 10% of unity (0.9–1.1; Wallinga et al., 2000) indicates that sensitivity changes which might occur during measurement were successfully corrected. Dose recovery and recycling ratios are presented in Table 4 and Fig. 3.

Recuperation was calculated from a zero-dose point in order to check if thermally-transferred charge from light-insensitive traps to the luminescence traps occurred. A recycling ratio value that is with-

### Table 3. Dosimetry results, dose rates and total dose rate of coarse-grained monomineralic potassium-rich feldspar and polynuclear fine-grained minerals. The a value was 0.15 ± 0.05 for monomineralic coarse-grains (Balescu and Lamothe, 1994) and 0.08 ± 0.02 for polynuclear fine-grains (cf. Lang et al., 2003). The average water content for all samples was 10 ± 5%.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Dosimetry</th>
<th>Dose rates</th>
<th>Total dose rate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Uranium (ppm)</td>
<td>Thorium (ppm)</td>
<td>Potassium (%)</td>
</tr>
<tr>
<td>NG1</td>
<td>1.20 ± 0.01</td>
<td>4.91 ± 0.03</td>
<td>1.97 ± 0.01</td>
</tr>
<tr>
<td>NG2</td>
<td>1.73 ± 0.01</td>
<td>7.04 ± 0.03</td>
<td>2.08 ± 0.01</td>
</tr>
<tr>
<td>NG3</td>
<td>2.16 ± 0.01</td>
<td>8.98 ± 0.03</td>
<td>2.57 ± 0.01</td>
</tr>
<tr>
<td>NG4</td>
<td>2.43 ± 0.02</td>
<td>8.03 ± 0.03</td>
<td>2.23 ± 0.01</td>
</tr>
<tr>
<td>NG5</td>
<td>2.65 ± 0.02</td>
<td>11.04 ± 0.04</td>
<td>2.49 ± 0.01</td>
</tr>
</tbody>
</table>

Fading tests and age calculations

Feldspar minerals have been observed to show an instability of the luminescence signal, which is also known as anomalous fading (Wintle, 1973; Aitken, 1985; Spooner, 1994). This signal loss over time results in (significantly) lower, thus severely underestimated IRSL ages. Huntley and Lamothe (2001) proposed a fading correction model, which was applied to three aliquots of each of our samples to obtain fading rates (g-values). Based on Thiel et al. (2011) and Buylaert et al. (2012), g-values below the threshold of ~1.5% per decade were considered to be laboratory artefacts, thus samples with g-values above this threshold called for fading corrections. For comparison, we calculated g-values for the pIRIR225 and pIRIR290 signals of sample NG5 (n = 3). In both cases, g-values were above the threshold of 1.5% per decade. At the same time, they were higher than the pulsed IR50 g-value of sample NG5 of 0.6 ± 0.2% per decade, namely 2.8 ± 0.2% per decade (pIRIR225) and 2.0 ± 0.3% per decade (pIRIR290; Table 4). This additional test proved that the use of the pulsed IR50 signal did not only benefit from a more stable and faster to bleach signal but also used that part of the signal that showed comparably less fading at least for this sample. Fading rates, fading uncorrected and corrected pulsed IR50, pIRIR225 and pIRIR290 ages are shown in Table 4.

Final ages were calculated taking into account the mean pulsed IR50 D_e values of all accepted aliquots.

### Table 4. Results of luminescence measurements using the (A) pulsed IR50 signal, (B) the pIRIR225 signal, and (C) the pIRIR290 signal, including number of measured aliquots (n_m) and number of aliquots taken for age calculation (n_c), mean recycling ratios, dose recovery ratios, mean recuperation, total dose rates, fading rates (g-values), mean D_e values, and fading uncorrected and fading corrected ages. Final ages are written in bold.

<table>
<thead>
<tr>
<th>(A) pulsed IR50</th>
<th>n_m/n_c</th>
<th>Mean recycling ratio</th>
<th>Dose recovery ratio</th>
<th>Mean recuperation (%)</th>
<th>Total dose rate (mGy/a)</th>
<th>g-value (% per decade)</th>
<th>Mean pulsed IR50 D_e (Gy)</th>
<th>Uncorr. pulsed IR50 age (ka)</th>
<th>Corr. pulsed IR50 age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG1</td>
<td>10/10</td>
<td>1.04 ± 0.04</td>
<td>0.95 ± 0.00</td>
<td>5.3</td>
<td>3.04 ± 0.13</td>
<td>2.5 ± 0.1</td>
<td>177 ± 3</td>
<td>58 ± 3</td>
<td>73 ± 3</td>
</tr>
<tr>
<td>NG2</td>
<td>10/10</td>
<td>1.03 ± 0.04</td>
<td>0.95 ± 0.00</td>
<td>5.1</td>
<td>3.39 ± 0.14</td>
<td>2.7 ± 0.4</td>
<td>202 ± 4</td>
<td>59 ± 3</td>
<td>77 ± 6</td>
</tr>
<tr>
<td>NG3</td>
<td>10/10</td>
<td>1.03 ± 0.04</td>
<td>0.94 ± 0.04</td>
<td>4.6</td>
<td>4.06 ± 0.14</td>
<td>2.1 ± 0.4</td>
<td>227 ± 3</td>
<td>56 ± 2</td>
<td>68 ± 5</td>
</tr>
<tr>
<td>NG4</td>
<td>10/10</td>
<td>1.03 ± 0.04</td>
<td>0.94 ± 0.02</td>
<td>4.8</td>
<td>3.77 ± 0.14</td>
<td>2.6 ± 0.2</td>
<td>216 ± 2</td>
<td>57 ± 2</td>
<td>73 ± 4</td>
</tr>
<tr>
<td>NG5</td>
<td>10/09</td>
<td>1.02 ± 0.04</td>
<td>1.01 ± 0.01</td>
<td>2.8</td>
<td>4.57 ± 0.22</td>
<td>0.6 ± 0.2</td>
<td>456 ± 5</td>
<td>100 ± 5</td>
<td>105 ± 6</td>
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</tbody>
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<table>
<thead>
<tr>
<th>(B) pIRIR225</th>
<th>n_m/n_c</th>
<th>Mean recycling ratio</th>
<th>Dose recovery ratio</th>
<th>Mean recuperation (%)</th>
<th>Total dose rate (mGy/a)</th>
<th>g-value (% per decade)</th>
<th>Mean IR225 D_e (Gy)</th>
<th>Uncorr. IR225 age (ka)</th>
<th>Corr. IR225 age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG5</td>
<td>6/6</td>
<td>1.02 ± 0.06</td>
<td>-</td>
<td>1.74</td>
<td>4.57 ± 0.22</td>
<td>2.8 ± 0.2</td>
<td>412 ± 5</td>
<td>90 ± 4</td>
<td>119 ± 7</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>(C) pIRIR290</th>
<th>n_m/n_c</th>
<th>Mean recycling ratio</th>
<th>Dose recovery ratio</th>
<th>Mean recuperation (%)</th>
<th>Total dose rate (mGy/a)</th>
<th>g-value (% per decade)</th>
<th>Mean IR290 D_e (Gy)</th>
<th>Uncorr. IR290 age (ka)</th>
<th>Corr. IR290 age (ka)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NG5</td>
<td>6/6</td>
<td>1.02 ± 0.06</td>
<td>-</td>
<td>2.19</td>
<td>4.57 ± 0.22</td>
<td>2.0 ± 0.3</td>
<td>474 ± 27</td>
<td>103 ± 7</td>
<td>125 ± 12</td>
</tr>
</tbody>
</table>
The age error of an uncorrected pulsed IR$_{50}$ age was calculated by taking the 1-sigma standard error of the obtained $D_e$ value. The age error of a corrected pulsed IR$_{50}$ age was calculated by adding the uncorrected age error to the mean age error.

4. RESULTS

For all luminescence samples, dose response curves and frequency-$D_e$ histograms as well as radial plots were
created based on the accepted aliquots (Figs. 4 and 5). Dose response curves are characterised by single saturating exponential growth. For the frequency-\( D_e \) histograms, bin widths are close to the median of \( D_e \) values as suggested by Lepper et al. (2000). Frequency-\( D_e \) histograms are characterised by very narrow and tight \( D_e \) distributions (Fig. 4) and radial plots are characterised by \( D_e \) values which are all within the 2-sigma range of the mean \( D_e \) value (Fig. 5).

Results of dose recovery and recycling ratio tests are all satisfying and in the acceptable range of 10% of unity (0.9–1.1; Fig. 3; Wallinga et al., 2000). Dose recovery ratios range between 0.94 ± 0.04 (NG3) to 1.01 ± 0.01 (NG5; Table 4). These results indicate that the applied SAR protocol is able to reliably recover a given dose, creating consistent \( D_e \) values. Recycling ratios range between 1.02 ± 0.04 (NG5) and 1.04 ± 0.04 (NG1; Table 4). Sensitivity changes that might occur during measurements were successfully corrected by the chosen SAR protocol. Recuperation values were all ≤5% of the natural signal (Table 4) and therefore in an acceptable range (Wallinga et al., 2000).

The obtained g-values for all samples were between 2.1 ± 0.4% per decade (NG3) and 2.7 ± 0.04% per decade (NG2) for monomineralic coarse-grains and 0.6 ± 0.2% per decade (NG5) for polymineralic fine-grains. Due to their higher g-values, the determined fading uncorrected pulsed \( IR_{50} \) ages of the coarse-grained samples NG1 to NG4 needed a subsequent fading correction (Thiel et al., 2011; Buylaert et al., 2012). Fading uncorrected and corrected pulsed \( IR_{50} \) ages are presented in Table 4. Dose rate results gave values ranging from 3.04 ± 0.13 mGy/a (NG1) to 4.57 ± 0.22 mGy/a (NG5; Table 3). Final depositional ages point to two major depositional phases. Sample NG5 gave a fading uncorrected pulsed \( IR_{50} \) age of 100 ± 5 ka, indicating a Late Pleistocene (Early Weichselian) deposition, correlating with MIS 5d. Samples NG1 to NG4 gave fading corrected pulsed \( IR_{50} \) ages ranging from 77 ± 6 ka (NG2) to 68 ± 5 ka (NG3), pointing to a Late Pleistocene (Early Weichselian to Early Pleniglacial) deposition which can be correlated with late MIS 5b to MIS 4. These ages reveal a chronological gap of about 12 ka between the Late Pleistocene MIS 5d and MIS 5b to MIS 4 fluvial sediments which seems to coincide with the major (erosional) bounding surface of about 9 m, separating the western and older from the eastern and younger fluvial sediments (Figs. 2A and 2B). Interpretation of the large-scale terrace architecture led to the assumption that the fluvial deposits display laterally attached terraces (Winsemann et al., 2015), which form when either both rates of fluvial aggradation and degradation are balanced or the generation of accommodation is low (Archer et al., 2011).
5. DISCUSSION

Luminescence results: reliable and robust?

Since feldspar minerals are known to suffer from anomalous fading, it is recommended to use only those parts of the IRSL signal which are less fading-dependent (e.g., Thiel et al., 2011). We followed the approach by Roskosch et al. (2015) who stated that the middle part of the decay curve of the pulsed IR$_{50}$ signal is characterised by a more stable, thus less fading-dependent luminescence signal when compared to other (parts of the) signals. The results of the additionally applied fading test of sample NG5 using the pulsed IR$_{50}$, pIRIR$_{225}$ and pIRIR$_{290}$ signals suggests that the pulsed IR$_{50}$ signal is more stable than the pIRIR$_{225}$ and pIRIR$_{290}$ signal (Table 4), confirming the use of the pulsed IR$_{50}$ signal. However, the applied fading tests for the other four samples indicated that some effect of anomalous fading was still present within our samples and fading correction seemed to be necessary for most of the samples. Table 4 shows that fading uncorrected pulsed IR$_{50}$ ages underestimated the fading corrected pulsed IR$_{50}$ ages by up to about 18 ka (NG2). So far, correction models for older samples (e.g. Lamothe et al., 2003; Kars et al., 2008) have not been tested on an accurate basis. Huntley and Lamothe (2001) strongly advise against using their correction model for (comparably) older deposits because their model is just applicable to the ‘linear’ part of the decay curve, thus (comparably) younger sediments. However, we followed the promising studies of Buylaert et al. (2011) and Roskosch et al. (2015), who successfully generated fading corrected ages of Middle Pleistocene (Elsterian, Saalian, Eemian) sediments. Consequently, we believe that the effect of age underestimation based on the occurrence of anomalous fading was minimized as far as possible by both using a more stable luminescence signal (Tsukamoto et al., 2006) and applying a suitable fading correction model (Huntley and Lamothe, 2001).

Age overestimation is commonly linked to the occurrence of insufficient bleaching of the luminescence signal prior to deposition. We additionally performed bleaching tests for the CW IR$_{50}$ (obtained as a part of the pIRIR$_{225}$ sequence) and pIRIR$_{225}$ signals and the pulsed IR$_{50}$ signals of samples NG2 and NG5. Natural aliquots of both samples were bleached in a Hönle SOL2 solar simulator for different bleaching durations between 0 and 6 hours and the remaining sensitivity-corrected signal intensity was plotted against the natural signal intensity. The results clearly demonstrate that the pulsed IR$_{50}$ signal is much faster to bleach (sample NG5) or bleaches in a similar way (sample NG2) as the pIRIR$_{225}$ signal, although this signal is harder to bleach than the CW IR$_{50}$ signal (Fig. 6). Taking the remaining signal after 30 minutes bleaching as an example, the pulsed IR$_{50}$ signal for both samples bleached to ~4–6% of the natural, whereas the pIRIR$_{225}$ signal has 6–11% remaining signal. Since the pulsed IR$_{50}$ signal is considered to be much more light-sensitive than other elevated temperature pIRIR signals (e.g. Jain et al., 2015; Roskosch et al., 2015) but has not been used widely so far (e.g. Roskosch et al., 2015), our objective was to use this stable, less fading-dependent and faster bleachable signal in order to provide new pulsed IR$_{50}$ ages. However, we only conducted comparative bleaching measurements of different IRSL signals on one coarse-grained sample (sample NG2) which is probably more prone to insufficient bleaching than the fine-grained sample of NG5. A definite exclusion of insufficient bleaching for all of the coarse-grained samples NG1 to NG4 can therefore not be made. However, as the last depositional ages of the coarse-grained samples are consistent within their age errors, we conclude that insufficient bleaching does not seem to be of great significance for these samples. The comparison of ages obtained from different IRSL signals for sample NG5 also demonstrated that although the fading corrected pIRIR$_{225}$ and pIRIR$_{290}$ ages slightly overestimated the pulsed IR$_{50}$ age, the three ages agreed within their 2-sigma uncertain-
ties (Table 4B and 4C). Again, this suggests that the bleaching condition prior to deposition does not seem to have been a major problem for sample NG5.

Stratigraphic significance of the luminescence results

Our ages are stratigraphically agreed with each other and are also consistent with the $^{230}$U/Th ages of the underlying interglacial deposits, which were correlated with MIS 7c to early MIS 6 (Waas et al., 2011; Fig. 7). The obtained luminescence ages are of great value when evaluating the previously established Middle to Late Pleistocene fluvial depositional model (e.g. Rohde et al., 2012). On the one hand, the occurrence of fluvial sediments which were assumed to be younger than the underlying interglacial sediments could be proven. However, fluvial deposition did not occur during the (Middle Pleistocene) Saalian, as had previously been assumed (e.g. Rohde et al., 2012), but during the (Late Pleistocene) Early Weichselian to Early Pleniglacial (MIS 5d, late MIS 5b to MIS 4; Table 4). This is comparable to the study of Cordiere et al. (2014) who also found by using luminescence dating techniques that deposits of a presumably Saalian age were actually deposited during the Weichselian.

On the other hand, the occurrence of Late Pleistocene (Weichselian) fluvial deposits was expected to occur only in the floodplain area of River Weser (cf. Rohde et al., 2012). The previous depositional model has to be revised due to the obtained luminescence ages of samples NG1 to NG4, pointing to an Early Weichselian to Early Pleniglacial deposition for this part of the pit. It is, however, likely that the adjacent valley area is characterised by gravelly and sandy fluvial sediments (referred to as Lower Terrace deposits, cf. Rohde et al., 2012), which had been deposited afterwards and which may be underlain by Saalian fluvial deposits, as has been described by Rohde et al. (2012). So far, these fluvial sediments have not been dated. Therefore, the Late Pleistocene sedimentary complex seems to have been subdivided into two fluvial sediment bodies.

The first phase of fluvial aggradation occurred around 100 ± 5 ka, correlating with MIS 5d, whereas the second phase of fluvial aggradation was found to have occurred from 77 ± 6 to 68 ± 5 ka, mainly correlating with late MIS 5b to MIS 5a (Table 4). It may, however, have continued until early MIS 4. The timing of vertical erosion (incision) of about 9 m (Figs. 2A and 2B) is difficult to determine but is likely to have occurred somewhere during MIS 5d to MIS 5c, which would be in accordance with data from France (Moselle and Meurthe: e.g. Cordiere et al., 2010; Somme, Seine, Yonne: Antoine, 1994; Antoine et al., 2007) and Germany (Leine valley: Winsemann et al., 2015). The luminescence dating results have shown that comparison with independent age control is of great importance. Given the important value of the obtained luminescence ages, additional numerical dating approaches need to be performed and thus complement the chronostatigraphic framework of the deposits of the Nachtigall pit.

6. CONCLUSIONS

We present new luminescence ages from five fluvial samples of the Nachtigall pit in the southern Weser valley in northwestern Germany. Luminescence measurements on monomineralic feldspar coarse-grains and polymineralic fine-grains were performed using a pulsed IRSL SAR protocol. Luminescence ages are consistent with $^{230}$U/Th ages of underlying interglacial deposits (Waas et al., 2011).

- Luminescence samples passed required performance tests, and results of dose recovery and recycling ratio tests as well as recuperation values were satisfyingly acceptable.
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