Residual luminescence signals of recent river flood sediments: A comparison between quartz and feldspar of fine- and coarse-grain sediments

M. Fuchs, J. Straub and L. Zöller

University of Bayreuth, 95440 Bayreuth, Germany

(Received 16 March 2005; in final form 1 June 2005)

Abstract
Sediments of the 2002 millenial flood in Saxony were investigated in respect to their residual luminescence signals. A comparison of residual palaeodoses in fine- and coarse-grain quartz and feldspar extracted from sediments was made. The fine-grain quartz was extracted by treating the sediment with H_2SiF_6 that selectively dissolved fractions other than quartz. Tests using H_2SiF_6 and HF for an optimum extraction of fine grain quartz were also carried out. A clear difference between quartz and feldspar based palaeodoses could be observed. Distinctly lower D_e values for quartz indicated better bleaching. Residual D_e values ranged from 0.5 Gy to 16 Gy for fine-grain quartz and coarse-grain K-feldspars, respectively.

Keywords: *residual doses, bleaching, fluvial deposits*

Introduction
One of the problems in optical dating of fluvial sediments is the confirmation of the extant of optical resetting of the geological luminescence signal at the time of deposition and burial (e.g. Fuchs and Lang, 2001; Olley et al., 1998; Stokes et al., 2001). The process of resetting strongly depends on the mineral type, the spectral quality and fluence of daylight. For a given spectrum, the quartz luminescence bleaches faster than that of feldspars (Godfrey-Smith et al., 1988). The daylight spectrum depends on the filtering and attenuation of the solar spectrum in water i.e. the depth and sediment load of the river (Berger, 1990; Ditlefsen, 1992). The foregoing implies that the bleaching characteristics of the grains should be dependent on the grain size since different grain sizes are transported differently. Typically the finer silt size fraction is more likely to be transported in suspension (i.e. closer to the water air- interface). In contrast, the sand and coarse silt fraction settle more quickly within the water column and are transported mostly as bed loads. Thus, there should be a higher probability of a better daylight-bleaching of the fine silt fraction. A complicating factor is that grains of the fine silt fraction tend to coagulate to form larger aggregates (Kadereit, 2000; Lang 1996), behaving like sand sized grains during their fluvial transportation and sedimentation. The grains within these aggregates are shielded from daylight by the outer grains and therefore tend to be insufficiently bleached.

Based on these considerations, sediments from the millennial flood in August 2002 in Saxony, Germany, were investigated in respect of their residual luminescence signals (Straub, 2004). Ideally, for these sediments equivalent doses should be close to zero, if sufficient bleaching of the sediments during their last reworking occurred. The extent of bleaching of different mineral types (quartz and feldspar) and different grain sizes (coarse- and fine-grain) were investigated.

Materials and methods
Fluvial sediments from the catastrophic 2002 flooding event at the rivers Elbe and Rote Weißeritz in Saxony, Germany, were investigated. Four fine-grained backswamp sediments from the river Elbe were taken north of Dresden (EL2: N51°03’53.88’’, E13°43’53.55’’, 110 m a.s.l.; EL5,6,7: N51°05’50.32’’, E13°39’01.23’’, 110 m a.s.l.), where the upstream catchment area is ca. 53,000 km^2. Sediments consisting almost entirely of sand-sized material from the river Rote Weißeritz, a tributary to the river Elbe, were sampled from a fresh point-bar (N50°57’3.76’’, E13°38’3.76’’, 300 m a.s.l.) in the V-shaped valley near Seifersdorf (Ore Mountains) at the upper part of the catchment, 1.5 km downstream of a dam. From this site, three samples (RW3, RW4, RW5) were taken (Figure 1). Sediments from both sites were sampled a few weeks after the flooding event.

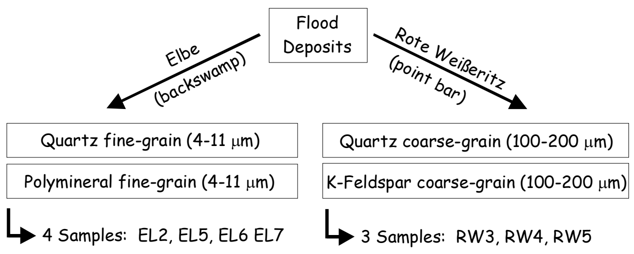


Figure 1: *Sediment samples from the river Elbe and Rote Weißeritz. From the river Elbe four fine-grain samples were investigated, from the river Rote Weißeritz three coarse-grain samples. From both rivers quartz and feldspars were used for D_e determination.*

For luminescence measurements, quartz and K-feldspar minerals of the coarse-grain fraction (100-200 µm) from the river Rote Weißeritz were extracted, using standard techniques. The polymineral and quartz fine-grain fractions (4-11 µm) were extracted from the deposits of the river Elbe (Figure 1). To separate the fine-grain quartz fraction, the techniques suggested by Prasad (2000) using hydrofluoric acid (HF), by Jackson et al. (1976) and by Berger et al. (1980) using fluorosilicic acid (H_2SiF_6) were tested. The procedure suggested by Prasad (2000) was found to be insufficient as after etching in 5 % and 10 % HF for 80 minutes and 120 minutes significant feldspar could still be detected by IRSL and X-ray diffraction. Etching with pre-treated 34 % H_2SiF_6 was carried out after Berger et al. (1980), varying the pre-treatment time, the etching time with its stirring frequency and the acid / sample ratio (Table 1). The pre-treatment of H_2SiF_6 was carried out by adding commercial quartz (100-500 µm) to the acid at a ratio of 1:10 and occasionally stirring the mixture. After pre-treatment for 3 or 7 days, the commercial quartz was filtered out and the acid was then used for etching the sediment samples. Optimum results in the extraction of fine-grain quartz were obtained by using H_2SiF_6 pre-treated for 3 days, etching each sample twice for 3 days (with a washing step in between), and stirring the samples twice per day. The loss in the weight of the sediment after this procedure was circa 74 % - 79 % (Table 1). The purity of the quartz fine-grain extracts was tested by IRSL and X-ray diffraction (Mauz and Lang 2004). Absence of any IRSL signal indicated the high purity of the fine-grain quartz extracts.

To determine the equivalent doses (D_e) a single aliquot regeneration (SAR) protocol (Murray and Wintle 2000) was used for the quartz fraction. A single aliquot regeneration protocol was used for the K-feldspar and polymineral fraction (Kadereit 2000).

Typically up to 15 aliquots per sample were measured for D_e determination. Although 15 aliquots is too low a number for reliable estimation of D_e in dating applications, we feel that it is sufficient to get an order of magnitude estimate of the equivalent dose from such poorly bleached flood sediments.

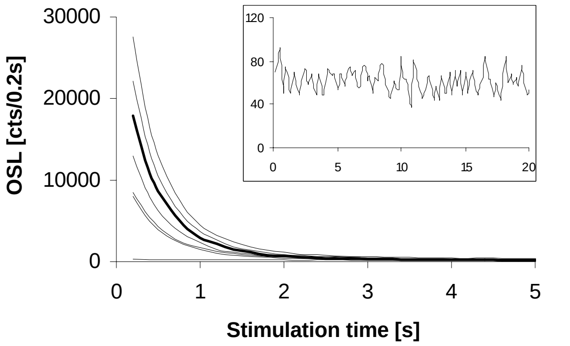
Pre-treatment [1]	Stirring-time [2]	Ratio [3] *Sediment / H_2SiF_6*	Loss [4] [%]
3 days	continuous	1:40	No yield
7 days	continuous	1:40	No yield
3 days	continuous	1:40	98
7 days	continuous	1:40	98
0 days	2 times / day	1:20	No yield
3 days	2 times / day	1:40	84
7 days	2 times / day	1:40	89
3 days	2 times / day	1:20	72
3 days	2 times / day	1:20	79
3 days	2 times / day	1:20	76
3 days	2 times / day	1:20	74
3 days	2 times / day	1:20	75

Table 1: *H_2SiF_6 etch procedures to extract fine-grain quartz from fluvial sediments.*
[1] *H_2SiF_6 pre-treatment with commercial quartz (100-500 µm) with a quartz:acid ratio of 1:10*
[2] *during sample etching with pre-treated H_2SiF_6*
[3] *ratio between sediment extract and H_2SiF_6 while etching*
[4] *Sediment loss after etching*

Optically stimulated luminescence from the quartz samples was measured for 20 s, using the first 0.4 s of the measurement for D_e determination, and subtracting a background signal from 16-20 s. For the polymineral and feldspar samples luminescence was measured for 240 s, using the first 100 s for D_e determination, and subtracting a background signal from 200-240 s. Typical quartz (OSL) and polymineral (IRSL) decay curves of the fine-grain fraction (EL5) are shown in Figure 2.

Luminescence measurements were carried out on a Risø Reader TL-DA-15. Quartz extracts were stimulated at 125°C with blue light (470 Δ 20 nm; ca. 36 mW/cm^2) and detected in the 290-370 nm (Hoya U340) wavelength band. The K-feldspar and polymineral extracts were stimulated at room temperature with infrared (875 Δ 80 nm; ca. 67 mW/cm^2) and detected in the 390-450 nm (Schott BG39, 2 x BG3, GG400) wavelength band, as recommended by Krbetschek et al. (1996).

(a)



(b)

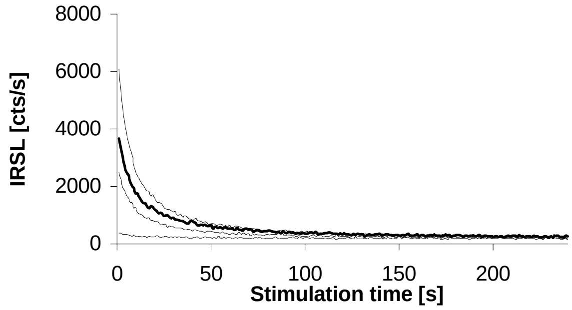


Figure 2: *OSL and IRSL decay curves for sample EL5 (figure a: quartz fine grains; figure b: polymineral fine grains. The graphs show the decay curves of the natural signal (bold line) and the regenerated signals. Inset of figure a: IRSL stimulated signal of the quartz fine grain fraction. The sample shows no IRSL signal, thus no feldspar contamination (for axis labels see large figure).*

Results and discussion

In Table 2 results of the equivalent dose determination (D_e) along with their standard deviation are given for the samples from the rivers Elbe (fine-grain) and Rote Weißeritz (coarse-grain). All samples show a $D_e > 0$, regardless of the grain size or mineral fraction used for D_e determination. The expected zero value for recently deposited sediments was not found. An indication of insufficient bleaching is also given by the high scatter of D_e values for the coarse-grain samples, expressed as the standard deviation. This is due to the number of grains per measured aliquot (ca. 500), which consist of heterogeneously bleached grains. The measured fine-grain aliquots do not show this high scatter, because of the much larger number of grains per aliquot (ca. 14,000) that averages out the heterogeneity (Fuchs and Wagner, 2003). Examples of dose distributions for coarse- and fine-grain quartz samples are given in Figure 3.

Sample	*Mineral*	*D_e [Gy]*	*SD [Gy]*	*SD [%]*
Fine-Grain (Elbe)				
EL2	Quartz	0.50	0.07	14
EL5	Quartz	0.75	0.06	8
EL6	Quartz	1.57	0.17	11
EL7	Quartz	1.32	0.11	8
EL2	Polyminerals	5.81	0.45	8
EL5	Polyminerals	4.49	0.28	6
EL6	Polyminerals	16.03	0.60	4
EL7	Polyminerals	10.19	0.65	6
Coarse-Grain (Rote Weißeritz)				
RW3	Quartz	2.32	1.43	62
RW4	Quartz	1.00	0.27	27
RW5	Quartz	1.67	0.49	29
RW3	Feldspar	7.41	8.75	118
RW4	Feldspar	7.72	6.64	86
RW5	Feldspar	16.35	17.90	109

Table 2: *Mean equivalent doses (D_e) of recent river flood sediments from the river Elbe and Rote Weißeritz for the minerals quartz and feldspar. D_e values are given with their standard deviation (SD).*

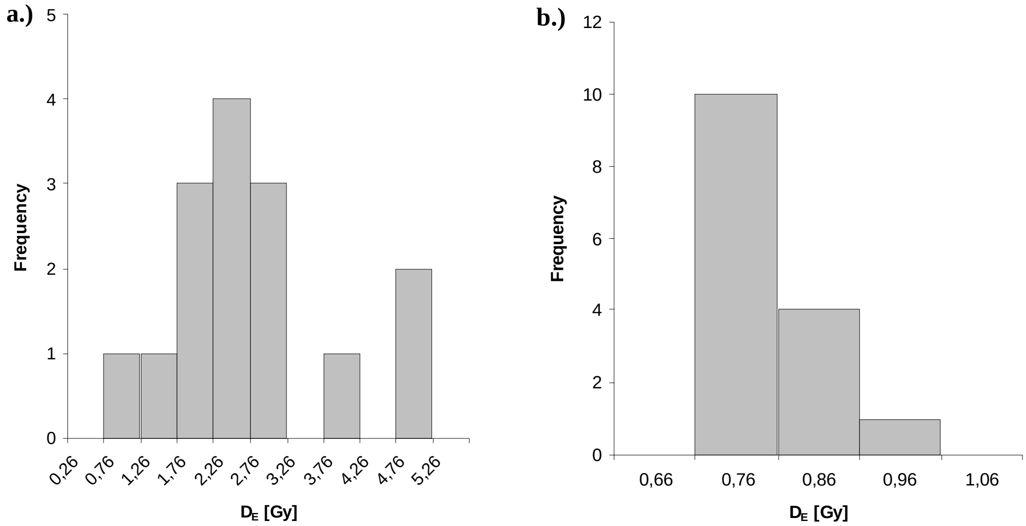


Figure 3: *Examples of typical dose distributions from coarse- (RW3, figure a) and fine-grain (EL5, figure b) quartz samples. Due to the small number of grains on the coarse grain aliquots (ca. 500) the distribution is much broader than that of the fine grain aliquots which have ca. 14,000 grains per aliquot.*

Even though none of the samples show a zero D_e, there are differences in the degree of luminescence signal resetting between the quartz and feldspar fraction of the fine- and coarse-grain extracts. Based on the values given in table 2, D_e values of feldspar and quartz are compared in Figure 4. Both, the fine- and coarse-grain quartz fractions are better bleached than the corresponding feldspar fractions. In the case of coarse grains, quartz shows a 3-10 times lower D_e than feldspar. For fine grains the differences in D_e values are even larger and quartz is showing an 8-12 times lower D_e than feldspar of the polymineral fraction. These large differences are higher than the difference due to the internal β dose rate of coarse K-feldspar grains (~0.5 Gy/a) or a lower a-value for fine quartz grains (typically ca. 0.03 rather than ca. 0.08 for feldspars), respectively. As residual D_e values rather than apparent ages were the purpose of this study, dose-rate measurements were not carried out from the samples. Assuming effective dose-rates of ca. 4 mGy/a for feldspar fine grains, ca. 3.5 mGy/a for quartz fine grains and for K-feldspar coarse grains and ca. 2.8 mGy/a for quartz coarse grains the following values would result as “residual” ages:

- Elbe-samples quartz fine grains:
  0.14 to 0.45 ka
- Elbe-samples feldspar fine grains:
  1.12 to 4.0 ka
- Rote-Weißeritz-samples quartz coarse grains:
  0.36 to 0.83 ka
- Rote Weißeritz-samples K-feldspar coarse grains:
  2.12 to 4.67 ka

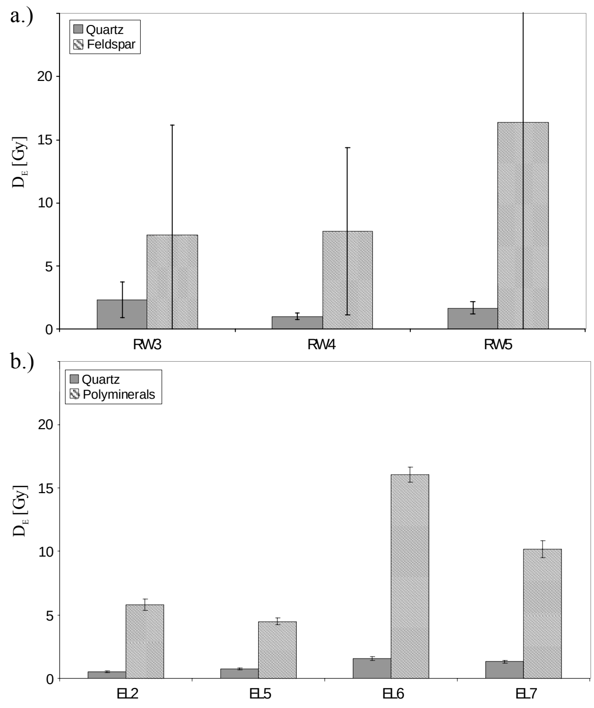


Figure 4: *Comparison of residual equivalent doses between quartz and feldspar (D_e) from recent river flood sediments from (a) Rote Weißeritz and (b) the river Elbe. D_e error bars are quoted at the 1σ level.*

We emphasize, however, that these values can only be taken as approximate first order estimates of apparent ages and indicate the amount of zero error in dating older samples of similar depositional history.

A comparison of different grain sizes for the same mineral is difficult, because there was not sufficient fine- and coarse-grain material available from the same samples. However, comparing quartz and feldspar of the fine- and coarse-grain fractions, there seems to be a trend towards lower D_e values for the fine-grains (Table 1, Figure 3). This could also be explained by extended transportation and thus better bleaching of the fine-grain sediment from the river Elbe in comparison to the coarse-grains from the river Rote Weißeritz. In the case of the Elbe site, the distance between the upper drainage system and the sample locality is some orders of magnitude larger than that of the Rote Weißeritz site (cf. Stokes et al., 2001).

Conclusion

Fine-grain (4-11 µm) quartz could be successfully extracted from the Elbe sediments by etching the polymineral fine-grain fraction with H_2SiF_6. Etching with HF as suggested by Prasad (2000) was not successful. Regardless of the minerals or grain sizes used for D_e evaluation, no zero D_e values could be obtained for recently deposited sediments. This is explained by insufficient bleaching of the sediments. Quartz showed considerably lower D_e values than feldspar, and could be used for optical dating of such sediments from catastrophic floods. Typical 'zero error' on age is estimated so as to provide an estimate of the amount of uncertainty associated with older sediment of similar depositional history. The present study suggests that the 'zero-error' in dating such catastrophic flood events in the region will be in the order of 0.1 to 1 ka for quartz and 1 to 5 ka for feldspars, respectively.

Acknowledgements

We would like to thank Prof. A.K. Singhvi for discussion and helpful comments.

References

Berger, G.W., Mulhern P.J. and Huntley, D.J. (1980). Isolation of silt-sized quartz from sediments. *Ancient TL* **11**, 8-9.

Berger, G.W. (1990). Effectiveness of natural zeroing of the thermoluminescence in sediments. *Journal of Geophysical Research* **95**, 12375-12397.

Ditlefsen, C. (1992). Bleaching of K-feldspars in turbid water suspensions: A comparison of photo- and thermoluminescence signals. *Quaternary Science Reviews* **11**, 33-38.

Fuchs, M. and Lang, A. (2001). OSL dating of coarse-grain fluvial quartz using single-aliquot protocols on sediments from NE Peloponnese, Greece. *Quaternary Science Reviews* **20**, 783-787.

Fuchs, M. and Wagner, G.A. (2003). Recognition of insufficient bleaching by small aliquots of quartz for reconstructing soil erosion in Greece. *Quaternary Science Reviews* **22**, 1161-1167.

Godfrey-Smith D.I., Huntley, D.J. and Chen, W.-H. (1988). Optical dating studies of quartz and feldspar sediment extracts. *Quaternary Science Reviews* **7**, 373-380.

Jackson, M.L., Sayin, M. and Clayton, R.N. (1976). Hexafluorosilicic acid reagent modification for quartz isolation. *Soil. Sci. Am. J.* **40**, 958-960.

Kadereit, A. (2000). *IR-OSL-datierte Kolluvien als Archive zur Rekonstruktion anthropogen bedingter Landschaftsveränderungen.* Ibidem-Verlag, Stuttgart.

Krbetschek, M.R., Rieser, U. and Stolz, W. (1996). Optical dating: Some luminescence properties of natural feldspars. *Radiation Protection Dosimetry* **66**, 407-412.

Lang, A. (1996). *Die Infrarot-Stimulierte Lumineszenz als Datierungsmethode für holozäne Lössderivate*. PhD thesis, University of Heidelberg, 137pp.

Mauz, B. and Lang, A. (2004). Removal of the feldspar-derived luminescence component from polymineral fine silt samples for optical dating applications: evaluation of chemical treatment protocols and quality control procedures. *Ancient TL* **22**, 1-8.

Murray, A.S. and Wintle, A.G. (2000). Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiation Measurements* **32**, 57-73.

Olley, J., Caitcheon, G. and Murray, A.S. (1998). The distribution of apparent dose as determined by optically stimulated luminescence in small aliquots of fluvial quartz: Implications for dating young sediments. *Quaternary Geochronology* **17**, 1033-1040.

Prasad, S. (2000). HF treatment for the isolation of fine grain quartz for luminescence dating. *Ancient TL* **18**, 15-17.
Stokes, S., Bray, H.E. and Blum, M.D. (2001). Optical resetting in large drainage basins: tests of zeroing assumptions using single-aliquot procedures. *Quaternary Science Reviews* **20**, 879-885.
Straub, J. (2004). *Studien zur Datierung von Hochflutsedimenten mittels Optisch-Stimulierter Lumineszenz am Beispiel von Ablagerungen der Flutkatastrophe in Sachsen 2002.* Unp. Dipl. Thesis. University of Bayreuth, Germany.

Reviewer
Ashok Singhvi

Comments
This study provides useful new data that will be of potential use in future studies on fluvial systems in the region.