Procedures used for optically and Infrared Stimulated Luminescence Dating of Sediments in Heidelberg

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Abstract : *This paper describes the procedures used for Optically Stimulated Luminescence (OSL) and Infrared Stimulated Luminescence (IRSL) in sediment dating at the Forschungsstelle Archäometrie in Heidelberg, Germany. The topics discussed include sampling, sample preparation, luminescence measurement, equivalent dose estimation and determination of dose rate.*

Introduction

Since the findings of Huntley *et al.* (1985) and Hütt *et al.* (1988) OSL and IRSL dating has been frequently applied and has become a method of high demand in geoscientific research of late Quaternary sediments. Nevertheless, some methodical problems involving experimental parameters, such as laboratory illumination, mineral separation, preheat procedures, stimulation and detection wavelengths, equivalent-dose (D_E) determination and dose rate determination, are still to be optimised and are subject to lively discussions and controversies. Depending on the combination of these parameters significantly different age results may be obtained.

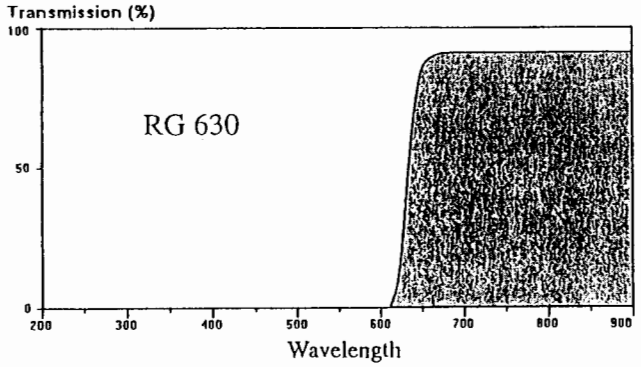
One of our research topics is to find out for which types of sediments optical dating may be applied successfully. In order to test the hypothesis that most waterlain sediments from Middle Europe can be dated, we use an empirical approach, i. e., dating of sediments for which independent age control is available (Lang and Wagner, 1997). This approach requires the specification of the experimental procedures applied. In this paper, we describe the techniques used for OSL and IRSL dating of sediments at our laboratory, particularly sampling, sample preparation, luminescence measurement, equivalent dose estimation and determination of dose rate, carried out on aeolian, alluvial, colluvial, limnic and littoral sediments with expected ages of less than 50 ka. Discussion of dating techniques may be found in Aitken (1992,1994), Wintle (1994), Mejdahl and Christiansen (1994) or Lang and Wagner (1996).

Sampling and sample preparation

In the field, sediments from outcrops are sampled with steel cylinders (100 cm^3, 200 cm^3 or 500 cm^3), which are hammered into the sediment. When recovering, both ends of the cylinder are immediately covered tightly with light-proof plastic lids. An additional sample of the sediment is watertight sealed in a plastic bag for determination of the actual interstitial water content and for radiometric analysis. In the laboratory's dark room, both ends of the sediment core, which may have been exposed to light, are removed. The sediment remaining in the steel cylinder is processed for luminescence analysis. If outcrops are absent, sampling is carried out by means of bore-cores. The core is split in half in the dark room and samples are taken from the inner part of the core. From fine grained sediments a minimum of 200 g is collected and for coarser grained sediments a minimum of 500 g.

Special attention is paid to optimal laboratory illumination. All lamps (halogen lamps with optical filters of 5 cm diameter as front window) are equipped with dimmers and are never focused directly onto the sample. Hence, during handling the samples are exposed at most to diffused light with very low intensity. Otherwise, the samples are strictly protected from light. During the separation procedure of quartz, red light of > 630 nm (Fig. 1) is used. The preparation of coarse grained feldspars and fine grained polymineral fractions is carried out under green-yellow light of wavelengths between ~ 500 and 600 nm (Fig. 1). This allows for minimum signal loss but high sensitivity of the human eye

OSL



IRSL

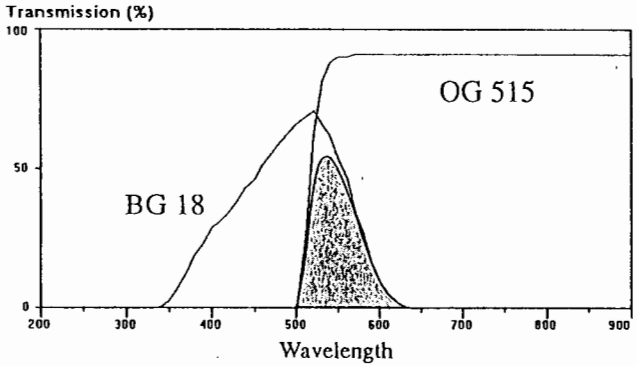
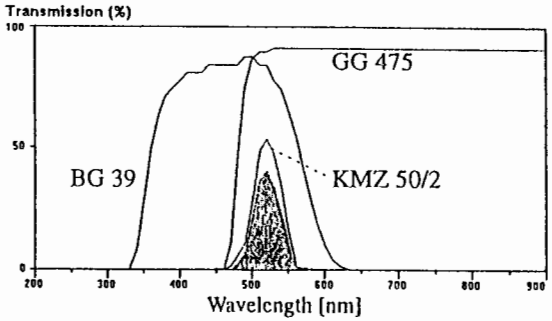


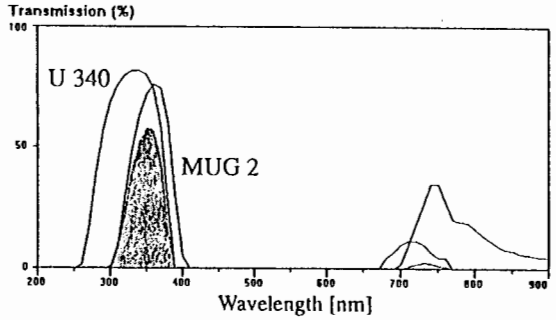
Figure 1 *Wavelength regions used for laboratory illumination. Transmission in % is plotted versus wavelength for the filters put in front of the lamps during sample preparation for IRSL and OSL measurements. The total transmission of the filter combinations is marked as shaded area. Filter used for OSL sample preparation: RG 630. Filters used for IRSL sample preparation: BG 18 and OG 515 (Appendix A).*

OSL

Stimulation:



Detection:



IRSL

Detection:

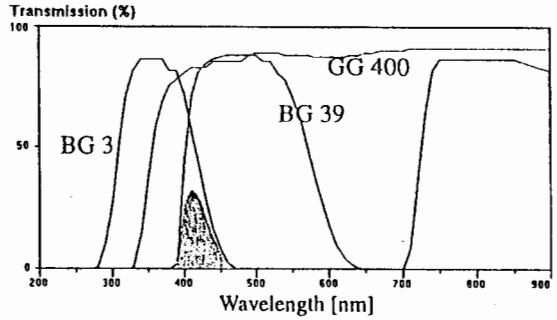


Figure 2 *Wavelength regions used for stimulation and detection. Transmission in % is plotted versus wavelength for the filters used for stimulation and detection of OSL (stimulation: BG 39-, GG 475- and KMZ 50/2 filters; detection: MUG 2- and U 340 filters) and for detection of IRSL (BG 39, 2 BG 3 and GG 400 filters) (Krbetscheck, et al., 1996). The total transmission of the filter combinations is marked as shaded area (Appendix A).*

when adapted to darkness (Wiggenhorn, 1995; Appendix B).

To prepare polymineral fine grains (4-11μm), the sample is treated with H_2O_2, then sieved and settled in 0.01 n NH_4OH. Subsequently, the sample is treated with HCl (10%), rinsed several times and dispersed in acetone. Finally, between 80 and 100 aliquots of each sample are prepared by settling in acetone on aluminium discs. Each disc carries equally 1 to 2 mg of silt. For preparing coarse grains, the preferred 90 to 125 μm fraction (if not available then 90 to 200 μm or 200 to 315 μm) is achieved by sieving. The sieved grains are treated with H_2O_2 and HCl. The quartz (using densities of 2.62 and 2.75 g/cm^3) or a K-feldspar-rich fraction (2.53 to 2.58 g/cm^3) are obtained by density separation. The quartz fraction is etched in 40% HF for 40 min and subsequently treated with HCl. As for the fine grain aliquots, 80 to 100 aliquots are prepared but in this case the weight is 2 to 4 mg. Steel discs or rhodium plated bronze cups are used as sample carriers and the grains are fixed on the discs with silicon spray. Because of the different response of quartz and feldspar to the wavelengths of the laboratory light it is advisable to obtain both mineral fractions in seperate sieving runs.

Equivalent-dose (D_E) determination

Routinely, the multiple aliquot additive dose methpd is used. Irradiation is carried out at room temperature using $^{90}Sr/^{90}Y$-β- and ^{241}Am-α-sources. 5 β-irradiator systems are available (dose rates between ~ 1 and ~ 10 Gy/min). Feldspar and polymineral aliquots are stored after irradiation for at least 4 weeks at room temperature. Quartz samples are stored at least 1 week at room temperature. Preheating of 220°C for 5 min (Smith *et al.*, 1986; Godfrey-Smith, 1994; Lang, 1996) is usually carried out on all samples. For determination of the growth curve 10 'natural' discs and 6 groups of artificially dosed discs (5 each) are measured. Additive doses are chosen subsequent to initial tests of the sample's dose response, in order to obtain at least a threefold increase in luminescence intensity.

Shine-down curves of 60 s are measured at room temperature using a 'Risø reader TL-DA-12' (Bøtter Jensen *et al.* 1991) equipped with an EMI 9635Q photomultiplier and a Xe-lamp (Kuhn, 1993). The light of the Xe-lamp filtered by a combination of BG 39-, GG 475- and KMZ 50/2 filters (transmission 500 - 550 nm, Fig. 2, Appendix A) is used for OSL stimulation of quartz. The OSL is detected with MUG 2- and U 340 filters (transmission 300 - 380 nm, Fig. 2, Appendix A). TEMT 484 diodes are used for IR stimulation (880 Δ 80 nm) of feldspars and IRSL is detected with a filter combination of BG 39, 2 BG 3 and GG 400 (transmission 390 - 450 nm, Fig. 2, Appendix A) (Krbetscheck, *et al.*, 1996). By restricting the detection to the 410 nm IRSL emission it is ensured that the sampled signal is more stable than the signal sampled by the broad detection window of a BG 39 filter only (Lang and Wagner, 1996).

Data handling is done using G. Duller's ANALYSE software. R. Grün's SIMPLEX ROUTINE is used for D_E calculation applying error weighting proportional to variance. For IRSL analysis, the subtraction technique is applied (Aitken and Xie, 1992), using the mean IRSL-intensity of the interval 50 s to 60 s as the 'late light'. This is necessary because the 410 nm emission seems to have a dose dependent hard-to-bleach-component (Lang and Wagner, 1997).

Fading tests are carried out for all polymineral fine grain and potassium-feldspar-rich fractions. An additional set of natural and maximum dosed subsamples, which have been irradiated and preheated contemporary to the discs used for the growth curve fixing, are measured after 3 months storage. The ratios of 'natural' intensities to 'artificially irradiated' intensities for the first and second measurement are compared to check for fading.

Dose rate determination

α-counting (Littlemore ELSEC 7286), β-counting (Risø GM-25-5) and low-level-γ-spectrometry (Ge-detectors) are routinely applied. Whenever possible, *in situ*-measurements are carried out using portable γ-spectrometry (NaI-detectors). α- and β-dose rates are calculated from α- and β-counting results respectively and from the U-, Th- and K-contents determined with low-level-γ-spectrometry and using the dose conversion factors given by Nambi and Aitken (1986). Although α- and β-counting are more precise than low-level-γ-spectrometry, systematic errors such as under- or overcounting may occur. Therefore, α- and β-counting are only used for age calculation if they do not differ significantly from the γ-spectrometric results. The α-dose rate for fine grains is calculated using the a-value system (Aitken and Bowman, 1975). The external α-dose rate for coarse grained feldspars is estimated as discussed in Appendix C. β-counting is used to determine the K-content of the potassium rich feldspar fraction. β-attenuation factors for dose rate determination of coarse grains are taken from Mejdahl (1979). To check for radioactive equilibrium, low-level-γ-spectrometry is used. If no *in situ*-measurements are

available, the γ-dose rate is calculated from the radionuclide contents derived from low-level-γ-spectrometry. Additionally, these radionuclide contents are used for modelling the dose rate if the sample exhibits radioactive disequilibrium. The contribution of cosmic rays to the dose rate is calculated following Prescott and Hutton (1994). All errors quoted are standard errors. Errors are calculated using the Gaussian error propagation law.

Conclusions

A large number of sediments ranging from aeolian sands, loess, limnic sediments, salt marshes, some fluvial sands to colluvial sediments and anthropogenic deposits have been dated successfully using the procedures described. The ages obtained range from less then 200 a up to 70 000 a with precisions from 7% to 13%.

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Appendix A: optical filters

All optical filters mentioned are 3 mm thick, except the KMZ 50-2 which is 4 mm thick. They are all produced by Schott except the U 340 which is supplied from Hoya.

Addresses:

Schott Glaswerke, Hattenbergstr. 10, D-55122 Mainz, Germany.

Hoya Corporation, optics sales division, 3-3-1 Musashino, Akishima-Shi, Tokyo, 196, Japan.

Appendix B: green-yellow light for IRSL sample preparation

The use of wavelength shorter than red for IRSL sample preparation goes back to personal communications with N. A. Spooner, G. Hütt and U. Rieser. Consequently, Wiggenhorn (1995) weighted the curve of spectral efficiency of the human eye (Gobrecht, 1977) with the spectral curves of energy required for bleaching the blue IRSL-emission of different types of feldspars (microcline and sanidine) as given by Spooner (1993 and 1994). The calculated curves show minimum signal loss but maximum sensitivity of the human eye when adapted to darkness between 500 and 550 nm, depending on feldspar type. The visibility (especially for contrasts) is higher than it is under longer wavelength. Consequently, sample handling can be carried out under fairly low light intensity. Following Wiggenhorn (1995) the laboratory lamps were equipped with dimmers and the filter combination shown in Fig.1. We checked for potential occurrence of accidental bleaching: Sample aliquots were split in two groups: Group 1 aliquots accompanied samples in preparation during the whole preparation procedures. Group 2 aliquots were kept in the dark. After finishing the sample preparation, the mean IRSL-intensities of the two groups were compared. In between statistical errors (3% to 6%) no signal loss occurred. The test is repeated from time to time.

Appendix C: external α-dose rate for coarse grained feldspars

Contrary to quartz, the surface of a feldspar grain is not regularly etched by HF (Duller, 1992). Etching occurs preferentially along crystal plains. Therefore, it is unlikely that the outer α-irradiated layer will be removed totally during etching. The luminescence signal of a HF-treated feldspar grain may still contain luminescence induced by α-particles - an amount which can hardly be determined. We think that estimating the external α-dose rate is more precise, even when the estimate is only rough.

For this rough estimate the percentage (x) of a grain's volume influenced by α-particles is calculated, assuming spherical grains, homogeneous irradiation of the grains, an α-penetration depth of 10 ± 5 μm and using the mean diameter of the grain size fraction separated. The α-dose rate for x is calculated as for fine grains, using an a-value of 0.1 ± 0.05. This procedure has the advantage that as soon as a precise method is available, the α-dose rate can be recalculated.

Reviewers

Martin Aitken, Rainer Grün

Comments

This detailed account of laboratory procedures is greatly welcomed; it is in line with one of the original objectives of Ancient TL. My only comment is in respect of Appendix C where the optimistic impression is given that there are no hidden problems associated with the HF etching of quartz grains. Investigations by Bell & Zimmerman (1978) showed there to be similar uncertainties with quartz grains; this was for grains extracted from baked clay; maybe the situation for grains from sediment is better. The failure of 45 minutes of HF treatment (at 40°C) to eliminate completely the alpha dose from quartz grains has been demonstrated by Valladas & Valladas (1982); they propose that evaluation is to be preferred and describe an appropriate experimental technique.

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