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Applying luminescence methodology to key sites of Alpine glaciations in Southern Germany

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ABSTRACT

This paper investigates the properties of quartz sampled at different sites in the Riss, Iller, Mindel, and Isar Valley of the northern Alpine Foreland of Germany and ties in with a previous study of optically stimulated luminescence (OSL) dating of glaciofluvial sediments from the type regions of the Würmian, the Rissian, and the Mindelian Glaciations. The previous investigation showed severe underestimation of quartz ages compared to the existing tentative chronology based on morphostratigraphic evidence as well as fading uncorrected feldspar Infrared Stimulated Luminescence (IRSL) ages. In this paper we present laboratory experiments and data analyses such as pulse annealing and isothermal decay measurements, $D_e(t)$ and NR(t) plots as well as curve fitting analyses to demonstrate that the quartz has unfavourable luminescence characteristics. We conclude that the quartz from the study area is not suitable for OSL dating using standard single-aliquot regenerative-dose techniques.

1. Introduction

The northern Alpine Foreland is of major interest for the reconstruction of the Quaternary glacial history, with research reaching back to the early 19th century. The benchmark work in the region was provided by Penck and Brückner (1901/1909), who developed the system of four major glaciations reaching from the Alps onto the foreland. These four glaciations were named (from old to young) Günz, Mindel, Riss and Würm. For several decades, the fourfold system had been adopted for many other regions worldwide. The stratigraphic scheme was originally mainly based on morphological investigations and tentative correlation with Milanković forcing, as suggested already by Köppen and Wegener (1924). While correlation of the fourfold system with marine isotope stratigraphy (which is based on Milanković forcing) is still used today (van Husen and Reitner, 2011), others have suggested more complex histories of the Quaternary of the Alps (Doppler et al., 2011; Ellwanger et al., 2011; Preusser et al., 2011a). According to the available studies, the main advance for the Würmian Glaciation occurred ~25 ka ago. The last advance of the Rissian Glaciation is usually attributed to Marine Isotope Stage (MIS) 6, with an age between 160 and 130 ka, but there is controversy if further advances occurred during early MIS 6, MIS 8 or even during earlier stages. While the Mindelian Glaciation is correlated with MIS 12 (~450 ka) in Austria (van Husen and Reitner, 2011), its age is considered poorly known in other parts of the northern Alpine Foreland. In summary, systematic numerical dating of sediments from the Alpine Foreland is rare, mainly because glacial and glaciofluvial deposits that can be directly connected to glacier advances are not straightforward to date.

In theory, luminescence dating can be used for the dating of sediment deposition ages. Quartz optically stimulated luminescence (OSL) is usually chosen when dating glaciofluvial sediments because of the faster resetting of the luminescence signal during sediment transport compared to feldspar (Godfrey-Smith et al., 1988). While many studies report good luminescence characteristics of quartz, some authors point out that quartz samples can underestimate depositional ages under certain circumstances (Bonde et al., 2001; Choi et al., 2003; Tsukamoto et al., 2003; Li and Li, 2006; Steffen et al., 2009; Klasen et al., 2015). In this context, Smith and Rhodes (1994) showed that the OSL signal of quartz comprises of different components, which are characterised by different stability and bleaching behaviour. The three major signal components were later termed fast, medium and slow component

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(Bailey et al., 1997). In most dating studies, the single-aliquot regenerative-dose (SAR) procedure (Murray and Wintle, 2000, 2003) is used for equivalent dose (D_e) estimation and the technique is robust when the quartz OSL signal is dominated by the fast component (Wintle and Murray, 2006). Studies that report on difficulties when using quartz OSL have in common that the luminescence signal is not dominated by the fast component, or is supplemented by an ultrafast component (Choi et al., 2003). Bonde et al. (2001) applied OSL and Infrared Stimulated Luminescence (IRSL) dating of palaeosols of presumably aeolian or volcanic origin from Santorini and observed little thermal stability of the OSL signal between 200 °C and 300 °C and a clear age underestimation. Luminescence properties of volcanic quartz from Japan also have shown age underestimation due to higher recuperation in the medium and slow component (Tsukamoto et al., 2003, 2007), which affected the initial OSL signal and resulted in missing thermal stability and a thermal lifetime of the main OSL component of only a few thousand years. Similar results were published for alluvial sediments in southern Peru by Steffen et al. (2009), who observed discrepancies between quartz and feldspar ages. In this case, the age offset is explained by thermally unstable components in the quartz.

The advantage of feldspar is that the mineral has a much higher saturation dose than quartz, which in principle offers the potential to date much further back in time compared to standard quartz OSL. However, there are two phenomena that make feldspar less suitable as natural dosimeter. Firstly, zeroing of IRSL in feldspar requires more time compared to quartz OSL (Godfrey-Smith et al., 1988; Murray et al., 2012) and incomplete zeroing will cause age overestimation if not detected and corrected for. Secondly, the IRSL signal of many feldspar samples is unstable with time, a phenomenon called fading (Wintle, 1973). This will cause underestimation of IRSL ages. While approaches to detect and correct for fading have been suggested (Huntley and Lamothe, 2001; Auclair et al., 2003), these approaches have sometimes been shown to be problematic, for example in the Swiss Alpine Foreland (Gaar and Preusser, 2012; Lowick et al., 2012) because samples appeared overestimated compared to the expected ages. While the problem of fading can be reduced by using a more stable signal such as the post-infrared infrared stimulated luminescence (pIRIR) signal (Thomsen et al., 2008; Buylaert et al., 2009), it has been shown that this approach can significantly overestimate the real deposition age in proglacial settings because the pIRIR signal is more difficult to bleach (Blomdin et al., 2012; Lowick et al., 2012; Gaar et al., 2014).

A first attempt to systematically date key sites of Alpine glaciations in the Riss, Iller, Mindel, and Isar Valley of Southern Germany using luminescence dating was carried out within the dissertation of Klasen (2008). This study found large differences between quartz OSL and feldspar IRSL age estimates, the latter being much higher and in better agreement with expected ages. Presented here is a summary of the key findings of Klasen (2008), only available in German so far, supplemented by a suite of additional experiments such as preheat plateau tests of natural aliquots, $D_e(t)$ and NR(t) plots, pulse annealing and isothermal decay experiments, curve fitting analyses and lifetime calculations. These results are used for a more detailed discussion of the quartz luminescence characteristics compared to Klasen (2008).

2. Sampling sites

During a field campaign in 2004, samples for luminescence dating were taken from sand lenses found within glaciofluvial terrace deposits in the northern Alpine Foreland in the area of the Rhine Glacier, the Iller Glacier and the Isar–Loisach Glacier (Fig. 1). Most of the sampled sites are within the type regions of the classical glaciations in the northern Alpine Foreland (Penck and Brückner, 1901/1909). Two outcrops of sediments of the Low Terrace ('Niederterrasse' = Würmian Glaciation, sites FIG and GLK) were collected in the Iller Valley (Ellwanger, 1983) and on the Munich Gravel Plain (Unger, 1995). Sediments of High Terrace ('Hochterrasse'), attributed to the Rissian (Brunnacker, 1964; Schreiner, 1985; Habbe, 1986; Jerz, 1987; Schreiner, 1989) have been sampled in the Isar Valley (PRO, SAT, ZDF), the Riss Valley (ECS, SHS, BAL) and the Iller Valley (BUX). Further samples were collected from the 'Jüngere Deckenschotter' of the Mindel Valley (GÄR), which is the type locality of the Mindelian Glaciation (Becker-Haumann, 2002; Rögner, 2002).

3. Methods

All samples were dry-sieved to isolate the 100–150 μm, 150–200 μm, 200–250 μm and 250–300 μm grain size fractions, depending on the composition of the sediment. Sample preparation included chemical treatment with HCl (10%) to remove carbonates, H_2O_2 (30%) to remove organic material and $Na_2C_2O_4$ (0.01 N) to remove clay as well as density separation ($\rho = 2.58$ g cm^{-3} and $\rho = 2.65$ g cm^{-3}) to isolate quartz and potassium feldspar. The quartz fraction was treated with hydrofluoric acid (37%, 40 min) plus a final HCl wash (10%, one hour). All measurements were carried out on an automated Risø TL/OSL DA 15 reader equipped with a calibrated ^{90}Sr beta source.

Multi-grain potassium-rich feldspar aliquots (1 mm) were stimulated with infrared diodes (870 nm, FWHM 40 nm) with the signal being detected through the combination of a Schott BG39, a Schott GG 400, and a Corning 7–59 filter (transmission 400–480 nm). The single-aliquot regenerative-dose protocol was applied (Wallinga et al., 2000; Preusser, 2003). This measurement included infrared stimulated luminescence at 50 °C ($IRSL_{50}$) for 300 s. To test the performance of the feldspar protocol, dose recovery tests were carried out. For these experiments, the samples were bleached using $IRSL_{50}$ stimulation for 1000 s and subsequently a laboratory dose similar to the natural dose was applied. The laboratory given dose was measured using different preheat temperatures between 210 and 290 °C. To investigate the influence of anomalous fading on the IRSL signal, fading tests were carried out (Auclair et al., 2003).

Blue-light emitting diodes (470 nm, FWHM 20 nm) and a Hoya U 340 filter (7.5 mm) transmitting wavelengths of 330 ± 40 nm were used for optical stimulation and signal detection of the quartz multi-grain aliquots (8 mm aliquots for laboratory experiments and 2 mm aliquots for equivalent dose measurements). The single-aliquot regenerative-dose (SAR) measurement procedure (Murray and Wintle, 2000, 2003) was used for all measurements. Standard laboratory experiments included dose recovery tests using preheat temperatures between 160 and 300 °C for one sample of each profile (Klasen et al., 2006). Cathodoluminescence (CL) was used to characterise the quartz (Preusser et al., 2006). Linear modulated optically stimulated luminescence (LM-OSL) measurements (Bulur et al., 2000) were used to split OSL into its different signal components. All LM-OSL measurements were made on natural and regenerative (after a laboratory dose of 60 Gy) aliquots at an elevated temperature of 125 °C during a stimulation of 1000 s (4000 datapoints, LED power from 0 to 80%).

For quartz and feldspar samples we have used the SAR criteria for the recycling ratio limit (15%) and test dose error (10%) to reject aliquots that have luminescence signals that are not appropriate for the SAR procedure. Equivalent doses have been calculated with the minimum dose approach of Preusser et al. (2007) to account for potential partial bleaching of the glaciofluvial sediments prior to