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ATTEMPTS TO DATE THE HÖTTING BRECCIA NEAR INNSBRUCK (AUSTRIA), A CLASSICAL QUATERNARY SITE IN THE ALPS, BY OPTICALLY-STIMULATED LUMINESCENCE

Alastair M.D. GEMMELL^{1)*)} & Christoph SPÖTL²⁾

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¹⁾ Geography and Environment, GeoSciences School, University of Aberdeen, Aberdeen AB24 3UF, Scotland;

²⁾ Institut für Geologie und Paläontologie, Universität Innsbruck, Innrain 52, 6020 Innsbruck, Austria;

" Corresponding author, a.gemmell@abdn.ac.uk

ABSTRACT

This paper presents the results of a programme of luminescence dating of the Hötting Breccia, an accumulation of up to 150 m of partly lithified (calcified) debris-flow and scree deposits interdigitated with thin (<20 cm thick) beds of poorly lithified material of loessic character, on the northern flanks of the Inn Valley in western Austria. Based on stratigraphical and macrofossil evidence these deposits were believed to be of Last or Penultimate Interglacial age but direct age control was lacking until recently.

Samples of loessic and of lithified debris-flow material were collected for dating. Optically-stimulated luminescence (OSL) analysis of all samples was undertaken using the single-aliquot regeneration (SAR) protocol using the Post infra-red OSL signal. Luminescence ages suggest an early- to mid-Würmian deposition of the Hötting Breccia. Deposition no later than an early Würmian cold phase is favoured on stratigraphic, sedimentological and palaeobotanic grounds and is consistent with recently published uranium-thorium dates of postdepositional calcite in fractures.

Die Höttinger Brekzie ist eine bis zu 150 m mächtige Ablagerung von teilweise lithifiziertem (calcifiziertem) Mur- und Hangschutt an der nördlichen Talflanke des zentralen Inntales bei Innsbruck (Österreich), der mit geringmächtigen (< 20 cm) gelbbraunen Lagen wechsellagert, welche nur schwach lithifiziert sind und einen lössartigen Charakter aufweisen. Aufgrund stratigraphischer Überlegungen und Makrofossilfunden wurde diese Abfolge bisher ins letzte Interglazial gestellt, ohne dass jedoch direkte Altersdaten vorlagen. In dieser Studie wurde nun versucht, das Sedimentationsalter der Höttinger Brekzie mittels optisch stimulierter Lumineszenz zu bestimmen.

Untersuchungen wurden sowohl an den lössartigen Lagen als auch am lithifizierten grobklastischen Material mit dem SAR Protokoll unter Verwendung des Post-IR OSL Signals durchgeführt. Die gewonnenen OSL Daten sprechen für ein Früh- bis Mittelwürm-Alter der Höttinger Brekzie, doch sollten diese Werte aufgrund des beobachteten fading-Phänomens nur als Minimalalter betrachtet werden. Stratigraphische, sedimentologische und paläobotanische Überlegungen im Verein mit Uran-Thorium-Altern an postsedimentären calcitischen Kluftfüllungen sprechen ebenfalls für eine Ablagerung der Höttinger Brekzie spätestens während der ersten Kaltphase des Frühwürms.

1. INTRODUCTION

The area north of Innsbruck, Tyrol, is a classic Quaternary research site in the alpine realm. Despite being located within the Inn valley, which harboured one of the major Pleistocene valley glaciers of the Alps, a thick succession of slope sediments known as the Hötting Breccia has been preserved on the southern slope of the Northern Calcareous Alps, locally known as Nordkette. Being under- and overlain by lodgement till and containing remains of warmth-loving plants this lithified talus has provided the key historical evidence for the multiple Quaternary glaciation of the Alps (Penck and Brückner, 1909; Ampferer, 1914; Penck, 1921). Because the detailed stratigraphy is still unresolved, however, there has been no consensus as to which interglacial period the breccia was formed in. Following the classical stratigraphy proposed by Penck and Brückner (1909), the breccia has heretofore been assigned either a Last (Rissian-Würmian) Interglacial age (Gams, 1954; Heuberger, 1975) or an earlier interglacial age (Gams, 1936; Ampferer, 1946; Paschinger, 1950; Patzelt and Resch, 1986).

Given the historic and palaeoclimatological significance of this deposit in the alpine Quaternary, this is a highly unsatisfactory situation. In consequence, a project has been launched to attempt dating of the Hötting Breccia utilising optically-stimulated luminescence (OSL) techniques. This article presents results from this study and discusses possible palaeoenvironmental implications.

2. GEOLOGICAL SETTING

The Hötting Breccia comprises an area of some 5.4 km² along the southern slope of the Nordkette (Fig. 1), although some authors consider that the original areal distribution was probably twice as extensive (e.g. Paschinger, 1950). The total thickness of the Hötting Breccia ranges from several metres up to about 150 m. The thickness of individual breccia beds ranges from less than 1 m to 7 m. The lower part of the Hötting Breccia sequence – known as the Red Breccia – is distinctively coloured due to the presence of sandy to silty matrix

material derived from the erosion of local Lower Triassic red beds. In contrast, the stratigraphically younger parts of the breccia higher on the valley side (up to an altitude of 2200 m and above the outcropping red beds) lack this colour and are generally matrix-poor (White Breccia). As a consequence, the Red Breccia shows a matrix-supported fabric, whereas the younger White Breccia is predominantly clast-supported. The angular clasts are overwhelmingly local in origin (limestone and dolostone). Crystalline rock components are present in very small amounts, implying glacial transportation from the Central Alps predating the breccia formation. This is substantiated by the presence of



FIGURE 1: Innsbruck and the adjacent slope of the Karwendel mountains (Nordkette), showing the location of Mayr's Quarry in relation to the red and white coloured Hötting Breccia deposits. Altitudes of mountain peaks are marked in metres.

rare clasts showing striations (e.g. Böhm, 1884).

The origin of the Red Breccia has generally been ascribed to debris flows probably triggered by heavy rainfall events (Penck, 1921; Paschinger, 1950). Such gravitational mass flows are not common on the slope north of Innsbruck today and imply a different (i.e. less dense) type of vegetation in conjunction with a higher amount of scree production. According to recent sedimentological studies the Hötting Breccia can be regarded as a clastic wedge composed of a lower alluvial fan unit (the Red Breccia) passing upwards into gully deposits and finally to lithified talus aprons (White Breccia; Sanders and Spötl, 2001; Sanders and Ostermann, 2006). The fan succession of the Red Breccia dips a few degrees toward the south and marks a palaeoelevation of the Inn valley floor ca. 200 m above the present one. The distal (i.e. more southern) facies of the debris-flow breccias has not been preserved, due to later glacial erosion in the Inn valley. In contrast, the White Breccia consists of rock-fall and megabreccias and thick-bedded breccias to conglomerates which grade up-section into steeply (up to 40°) inclined strata of lithified talus (Sanders and Spötl, 2001).

3. SILT LAYERS

An important aspect of the Red Breccia as exposed in the former Mayr's Quarry (Fig. 1) is the presence of yellow silt layers sandwiched between breccia beds (Fig. 2) and at the contact between the underlying till and the breccia. Within the quarry sections, seven discrete silt or loessic layers ranging in thickness from about 1 cm to 20 cm were identified. In contrast to the well indurated breccia beds these silt layers are poorly lithified and generally lack sedimentary structures. Faint lamination was observed only locally. The lower and upper boundaries of the silt layers are sharp and there is little evidence of erosion or reworking of these layers by the subsequent debris flow (Fig. 3). Powder X-ray diffraction analyses show a rather uniform mineralogical composition consisting of quartz, calcite, dolomite, K-mica and chlorite, together with small amounts of albitic plagioclase (Obojes, 2003; Ladurner, 1956), guite unlike the average composition of the Red Breccia. The carbonate content of the silts varies between 10 and 43% by weight. The grain size spectrum is dominated by the fine silt fraction. Scanning electron microscope images (Fig. 4) show angular grains and a high proportion of fine grained matrix. The heavy mineral spectrum of these silt layers matches that of today's River Inn, i.e. abundant garnet and opague minerals, together with chlorite, muscovite, biotite, staurolite, zircon, tourmaline, plus traces of hornblende, rutile and apatite (Obojes, 2003), again incompatible with an origin from the slope north of Innsbruck.

The origin of the silt layers has previously been attributed to dust sedimentation of local origin (Ladurner, 1956). Dust accumulation and the presence of poorly vegetated deflation areas in the Inn valley are difficult to reconcile with the classical interpretation of this site, based on the plant remains, as being interglacial. The apparently cool climate character of this sediment is also underscored by the presence of a fauna of "loess snails" in the same stratigraphic position in a temporary outcrop in the Hötting Graben west of Mayr's Quarry (Penck, 1921; Fig. 1).

4. OSL DATING - A BRIEF INTRODUCTION

Luminescence dating techniques - including OSL - are used to determine the period of time that has elapsed since quartz or feldspar mineral grains in sediments (amongst other constituents) were last exposed to sunlight or to heat, as heat and/ or sunlight exposure for even a short time can cause the luminescence signal to be zeroed. The OSL signal that is used for dating arises from the release of electrons which have previously become detached from their 'parent' atoms in consequence of exposure to naturally occurring ionizing radiation.



Floor of quarry

FIGURE 2: Simplified sedimentological section of Mayr's Quarry, showing yellow-brown loessic layers sandwiched between thick-bedded breccia layers. Grey and open asterisks mark the respective locations of drill cores in the loessic layers and of a core sample from the breccia matrix. OSL sample ages (in kyr) are indicated at the appropriate levels in the section. Note that the lowest loessic layer discovered is only visible in the southernmost part of the quarry, and hence has been omitted from this diagram.

Such ionizing radiation is produced by isotopes of uranium, thorium, their daughter nuclides, and potassium within the mineral structure of the rock or sediment, together with cosmic radiation. A proportion of the electrons detached by such radiation become 'trapped' at defects in the mineral structure and are unable to recombine with the 'parent' atom without the assistance of light or heat energy. Over time the number of trapped electrons in the mineral grains increases as a function of the ionizing radiation flux (the 'dose rate') and the availability of suitable defects in the mineral structure to act as traps. Thus in general the greater the period that has elapsed since the sediment was last heated or exposed to daylight, the greater the number of trapped electrons present in the mineral structure.

The trapped electrons can be released in the laboratory by carefully controlled applications of heat [thermoluminescence (TL)] or light [optically stimulated luminescence (OSL)]. A proportion of these electrons will then move to so-called recombination centres and cause light to be emitted in the form of photons. The number of photons emitted (i.e. the OSL signal) is a function of the number of formerly trapped electrons evicted by the heat or light energy. Controlled doses of ionizing radiation (measured in Gray) are administered to samples of the material being studied, and the OSL signal measured and compared to the signal emitted when the natural sample is heated or exposed to light. The OSL signal can then be expressed as the Equivalent Dose (D,), the dose that the mineral grains have accumulated over time. Division of the D, value by the dose rate gives the time (in years) since the mineral grains were last exposed to light or heat, and hence the OSL age of the sediment:

$$OSL \ age \ (yr) = D_e \ (Gy) / Dose \ rate \ (Gy \ yr^{-1})$$
(1)

The OSL signal of mineral grains is usually reset ('zeroed') during transportation from one location to another, but in some cases the light exposure may be inadequate to completely reset the existing signal. This phenomenon is known as partial bleaching, and can lead to an overestimate of the number of electrons which have become trapped since the sediment was finally deposited. In such circumstances, calculated D_e values may be overestimated, leading to inflated age determinations.

For further information about OSL techniques the reader is referred to Aitken (1998), Bøtter-Jensen et al. (2003), Duller (2004) and Lian and Roberts (2006).

5. PROCEDURES

Investigations were focussed on the former Mayr's Quarry, currently the most extensive exposure of the lower lying Red Breccia. Samples from five locations in the east face of the quarry (Figs. 1, 2) were collected for analysis. At each location, five-centimetre diameter cores were extracted using a power drilling system. A truck-mounted hoist enabled access to sites up to 8 m above the floor of the quarry (Fig. 2). Wherever possible, coring into the exposure was continued to a depth of at least 1 m. Due to the friable nature of the loessic layers drilling was performed with compressed air rather than water. Each core was left in the steel casing (to avoid light contamination), wrapped in black polythene, sealed and stored in a wooden box. In the laboratory the core pieces were carefully extracted from the casing under red safelight conditions.

Material for dating was released from the core samples via

Sam

ABL

ABU_198

ABU 199

an initial immersion in 10% HCI (volume for volume dilution of concentrated HCI) for c. 20 min or until the outer surface of the core had been dissolved. After brushing the surface to remove any grains remaining which might have been exposed to light, the remainder of the core was then transferred to another bath of 10% HCI. The rest of the calcite was then dissolved, and all remaining mineral grains treated in H₂O₂, then dried and sieved. Poorly lithified loessic material was similarly treated with 10% HCl and then H₂O₂. For all samples, material was then prepared for analysis by settlement in acetone (4-11 µm polymineralic grains) or by HF etching followed by density separation in sodium polytungstate to isolate quartz grains (90-125 µm and 125-180 µm). Aliquots of each sample were produced by settlement in acetone on stainless steel discs (4-11 µm grains) or by dispensing quartz grains onto such discs and fixing them in position with silicone oil.

OSL analysis of the samples was undertaken using a Risø TL-DA-15 reader. Coarse grains of quartz (>90 μ m) were examined using a single-aliquot regeneration (SAR) protocol (Murray and Wintle, 2000; 2003) using stimulation with blue LEDs emitting at ~470 ± 30nm. Photon emissions were detected using a photomultiplier tube (9235QA) fitted with Hoya U-340 optical filters, removing all but the desired wavelengths.

For samples containing few or no quartz grains >90 μ m in size, the polymineralic fine grain fraction was analysed using the response to stimulation with blue diodes following infrared stimulation (830 Δ 10 nm), as per the protocols of Banerjee et al. (2001). In this protocol the infra-red stimulation is used to drain most of the signal from feldspar grains, so that the dominant response to subsequent stimulation with blue diodes should come from quartz grains.

Some doubts have been expressed as to whether this protocol can reliably isolate the quartz signal from a polymineralic mixture unless the sample has been chemically pre-treated to isolate the quartz fraction as far as possible (Roberts, 2007). Dose recovery tests, in which aliquots of the sample are bleached in sunlight, then given a known laboratory dose prior to being subject to SAR analysis using the Post IR-OSL protocol, have been conducted on some of the samples from Mayr's Quarry. If the protocol is successful, the measured dose determined using the blue LED stimulation should be the same as the administered laboratory dose. If infra-red stimulation of those same aliquots also yields a measured dose the same as the known laboratory dose, then it is possible that the signal from both stimuli comes from feldspar, and the protocol cannot be assumed to have isolated a quartz signal. If however, the IR-stimulated signal yields no accurate determination of the laboratory dose, yet blue stimulation gives the correct laboratory dose, there is much greater confidence that the protocol has successfully removed the majority of any feldspar signal, and is therefore capable of giving meaningful age determinations for that sample. Results indicate that the Post IR-OSL protocol could successfully recover the correct D_a value (within errors) for two of the three samples subjected to this test (Table 1).

ole I.D.	Test Dose (Gy)	Recovered Dose/Test Dose Ratio			
_196	20	1.029 ± 0.118			

All the dose recovery data in this table relates to the post-IR Blue signal **TABLE 1:** Results of OSL dose-recovery tests for selected samples from the Hötting Breccia.

16

70

As part of the SAR protocol used for each sample, checks were carried out on every aliquot to ensure the ability of the protocol to return accurate results for that sample. Recycling ratios test the extent to which the sensitivity correction in the protocol is effective for a particular aliquot (Murray and Wintle, 2000). All aliquots for which the recycling ratio fell outside the range 1.0 \pm 0.1 were excluded from subsequent analysis, as the sensitivity correction was considered not to have been effective in such cases.

The SAR protocol also includes a step to test for recuperation (the build up of OSL signal without irradiation). If this is significant, the underlying assumption of the SAR protocol that the corrected dose-response curve passes through the origin (i.e. the corrected OSL signal is zero for zero dose) cannot be confidently upheld (Murray and Wintle, 2000). Accordingly any aliquots for which the corrected OSL signal following a zero dose is >5% of the natural signal have also been rejected for subsequent calculation of the D_e value for that sample.

Dosimetry for all samples was ascertained by neutron activation analysis (NAA, undertaken at Becquerel Laboratories, Australia), and by field determination of gamma radiation using a MicroNomad portable gamma spectrometer. A best estimate of the water content of samples was determined by weighing sediment at field moisture content, oven drying it at 60°C and then reweighing it. The cosmic radiation flux was determined following the method of Prescott and Hutton (1994). An a-value of 0.1 ± 0.01 was adopted for all fine grain



FIGURE 3: Detail of loessic layer at Mayr's Quarry. Note the stark difference in character between the fine-grained yellow silts and the bounding layers of breccia. Centimetre scale.

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 1.18 ± 0.07

 1.00 ± 0.06

dosimetry calculations following Wintle (pers. comm.) The resulting dosimetric data are presented in Table 2.

The calculation of the dose rate for the dating of samples has been based on certain assumptions:

- Deposition of individual debris flows took place rapidly
- Calcification of each flow deposit was largely complete prior to the deposition of any superposed loessic layer. This assumption is supported by two lines of evidence: (a) Holocene debris-flow deposits in the Northern Calcareous Alps commonly show evidence of rapid initial cementation (Spötl, unpublished data; D. Sanders, pers. comm. 2008), and (b) the small degree of cementation of the loessic layers argues against a more continuous calcification process throughout the post-depositional history of the Hötting Breccia.
- Burial of any loessic layer by a subsequent debris flow event or events took place soon after the emplacement of that layer.

If correct, these assumptions imply that mineral grains within each loessic layer were exposed to a full 4-pi radiation flux for most of the time since that layer was deposited.

For each sample the dose rate has been calculated using NAA data to calculate the alpha- and beta-contributions to the radiation flux, with the gamma-contribution being calculated from the portable gamma spectrometry. Any dose rate contribution from cosmic radiation has been calculated following the model proposed by Prescott and Hutton (1994). For all dose rate calculations, water content has been assumed to be that of the present day, though an uncertainty factor of $\pm 10\%$ has been built in to cover possible fluctuations in water content over time.

The approximate age of calcification and lithification of the debris flow deposits needed to be determined before the accuracy of the OSL ages for the deposition of the loessic layers could be assessed. There is no direct dating of the calcification process, but Spötl and Mangini (2006) published U/Th dates for flowstones (calcitic speleothems) which precipitated in two vertical cracks running through the breccia layers at Mayr's Quarry immediately to the west of the section sampled for OSL dating in the present study. These ages reveal several phases of flowstone deposition between 100.5 ± 1.5 kyr and 70.3 ± 1.8 kyr indicating that breccia deposition and lithification was complete by the time the oldest flowstone was deposited. Sanders and Ostermann (2006) briefly reported a slightly older radiometric date of isopachous calcite cementing components of the White Breccia (109 ± 7 kyr). The significance of this date is somewhat unclear, however, because of the analytical approach used (isochron based on leaching steps, significant scatter of the data) and because a second poorly defined isochron from an occurrence further west yielded an age of 21.5 ± 1.5 kyr (Ostermann, 2006), which is geologically virtually impossible (the Inn Valley was filled by ice during the Last Glacial Maximum). The oldest U/Th age obtained from flowstones (101 kyr - Spötl and Mangini, 2006) has therefore been used in this study as an approximation (minimum age) for the timing of calcification of the breccia layers.

6. RESULTS

Samples show clear growth of the OSL signal with increasing radiation dose (Fig. 5) without saturation effects becoming significant. Similarly, recuperation effects are very limited in scale (Fig. 6) and unlikely to have significantly distorted the age determinations. Figure 6 also shows an example of the results from a recycling test on the same sample. The correction procedures built in to the SAR protocol have been highly effective for those aliquots used for subsequent age calculations. As an additional check, dose recovery determinations

> were made on the 4-11 µm fractions of samples ABU_196, ABU_198 and ABU_199. In this procedure, aliquots were bleached by exposure to sunlight, then irradiated with a known dose in the laboratory. This dose is utilised as a 'natural' dose, and the aliquots are then subjected to the same sequence of measurements as were used to determine the luminescence age of the sediment, including the use of preheats at 20°C intervals from 160 to 300°C. Where sufficient material was available, three aliquots were tested at each preheat temperature. If the Post IR-OSL protocol is effective,

> then it should be able to determine

the laboratory 'natural' dose accu-

rately. Sample D, determinations ty-

pically show quite extensive scat-

Sample I.D.	U ^a (ppm)	Th ^a (ppm)	Rb ^a (ppm)	K ^a (%)	Cosmic radiation flux ^ь (Gy/kyr)	Dose Rate ^c (Gy/kyr)
ABU_196	1.32	0.95	n.d.	0.367	0.030±0.0015	1.24±0.07
ABU_197	1.72	1.14	n.d.	0.363	0.030±0.0015	1.39±0.09
ABU_198	2.39	1.02	n.d.	0.17	0.032±0.0016	1.79±0.12
ABU_199	0.69	1.5	n.d.	0.416	0.032±0.0016	1.35±0.05
ABU_200	3.25	13.7	n.d	1.87	0.048±0.0024	2.64±0.13
ABU_201	3.78	13.8	n.d.	1.85	0.048±0.0024	2.71±0.16
ABU_202 (90-125µm)	3.35	12.6	n.d.	2.29	0.048±0.0024	3.08±0.21
ABU_202 (125-180µm)	3.35	12.6	n.d.	2.29	0.048±0.0024	3.00±0.2
ABU_219	3.05	13.8	145	2.36	0.052±0.0026	4.73±1.12
ABU_223	3.52	12.3	126	1.8	0.034±0.0017	2.58±0.13

n.d. = Rubidium content not determined for this sample

* Concentration determined by NAA at Becquerel Laboratories, New South Wales, Australia.

^b Calculation following Prescott and Hutton (1994)

[°] Dose rate compiled from NAA data for alpha- and beta-activity, and portable gamma spectrometry for the gamma-component of the dose rate.

TABLE 2: Dosimetric data for samples from the Hötting Breccia

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ter about the mean, a pattern often replicated in the dose recovery data (Fig. 7). The results of such a dose recovery test, plotted as the ratio between the given dose and the dose determined using the SAR protocol, should cluster around a ratio of 1 if the protocol is effective. Results (Table 1) confirm the efficiency of our sensitivity correction procedures and of the reproducibility of the dose response characteristics of the sediment. They also indicate that the post-IR OSL protocol is capable of returning accurate D_e values for samples such as these.

7. DISCUSSION

7.1 OSL DATING

The calculated ages for the majority of samples collected from the Hötting Breccia conform to the concept that the basal, conspicuously red coloured layers of the deposit (the Red Breccia) were most probably laid down during Marine Isotope Stage (MIS) 5d or 5e. Subsequent debris-flow events appear to have occurred in relatively rapid succession, such that within error margins the entire deposit could have been laid down as an almost continuous event, broken only by short interregna during which the loessic layers accumulated. The thickness of the individual loessic layers (<20 cm) is commensurate with their accumulation in a relatively short time.

Although the majority of the ages determined are consistent within error margins given the constraints of stratigraphy (Fig. 2), there are some samples (e.g. AB_200 and ABU_201) for which the calculated age would indicate deposition considerably after the calcification of the breccia (Table 3), a situation which is logically impossible given their location. All such samples come from loessic bands within the sequence (Fig 2), and all are overlain by calcified sediment layers.

Although the flowstone U/Th-data represent a minimum age for the consolidation and cementation of the breccia beds, a possible underestimation of the timing of this calcification process is not thought likely to be a source of significant error in the determination of the luminescence ages. If the U/Th ages as determined by Spötl and Mangini (2006) are indeed underestimates, then more of the OSL ages become impossibly young for the reason mentioned above – that the sediments would postdate the calcitic infill events affecting them. If that is the case then there are clearly problems with at least some of the OSL age determinations.

Possible reasons for the anomalously young ages for these samples include:

- Accidental exposure of the sample to light during collection
- Limitations on accuracy of dosimetric determinations, in particular the impact of possible radioactive disequilibrium
- Anomalous 'fading' of the OSL signal over time, a problem not known to be associated with the OSL signal from quartz (Roberts et al., 1994)
- Loessic material being emplaced at a later date than the bounding breccia horizons

This last suggestion seems inherently unlikely, given that the



FIGURE 4: Scanning electron micrograph of the fines from a loessic layer. The angularity of the principal minerals visible - quartz and K-mica - suggests a local source for the grains, i.e. alluvial sediments of the River Inn.

sampled face is located as much as 40 m behind the original hillslope prior to the start of working at the quarry. It is impossible to imagine aeolian dust blowing that far into narrow fissures between the breccia layers, even had such cracks existed in the original slope.

Significant accidental exposure to light during sample collection ought to be reflected in abnormally low D_e values. While the D_e values for ABU_199 and ABU_200 are the lowest of any of the samples, they are not significantly low compared with the others, once errors in D_e determination have been taken into consideration (Table 3). Nevertheless accidental exposure cannot be ruled out as a possible reason for the anomalously young ages for these samples.



FIGURE 5: Luminescence growth curve for ABU_199, showing good recycling characteristics. The beta laboratory irradiation rate was 0.043 Gy sec⁻¹. The growth curve is for an aliquot which has been given a preheat of 160° C for 10 seconds prior to each shine in order to eliminate any 'unstable' luminescence signal from that measurement. The horizontal dashed line shows the intensity of the 'natural' luminescence signal, and where it intercepts the growth curve, the vertical dashed line marks the beta dose (D_e) required to match that intensity of OSL.

Natural fading of the luminescence signal (leakage of electrons from traps at ambient temperatures) may explain some possible age underestimation. This phenomenon, which particularly affects minerals such as feldspars and zircons (Aitken, 1998), means that the total number of electrons trapped within the mineral structure is less than might be expected given the Dose Rate, and will also tend to reduce further growth over time. The consequence is that stimulation would cause a smaller luminescence signal to be emitted than the age of the sediment might justify. Fading tests have been run to determine whether or not fading could possibly have had a significant impact on the OSL ages determined in the present study. As the signal from quartz is not known to fade, any such behaviour in these samples would support Roberts' (2007) concerns that the post IR-OSL protocol is not always effective in eliminating the feldspar contribution to the OSL signal when polymineralic samples are analysed. Certainly, fading might explain OSL ages of some sediments postdating, rather than predating, the U/Th ages of calcitic infills in cracks in those same sediments.

Simple fading tests involved zeroing of any stored signal in a sample by exposure to daylight, then irradiation of that material with a known laboratory dose prior to storage in the dark at room temperature for 14 days. Next, the aliquots of the sample were shone using IR stimulus, followed by OSL (Blue light) stimulus at 125°C for 100 seconds to measure any signal remaining. The aliquots were then re-irradiated with the same laboratory dose, but then shone immediately, using the same duration and intensity of stimulus as before. In each case, measurement was normalised using the response to a standard test dose. The ratio of the signal for the 'delayed' shine to that for the 'immediate' shine gives the percentage fade of signal over the period of storage. Results show that all samples tested in this way showed a degree of fading (Table 4) even for the OSL signal.

As a cross-check on these findings, a more complex set of measurements, based on procedures devised by Auclair et al. (2003), were undertaken on samples ABU_196 and ABU_199. In this protocol, samples are put through cycles of irradiation and measurement with different degrees of delay. The loss of signal can be monitored over different time periods, with storage of aliquots and then reapplication of the routine being used to extend the duration of storage – in these case of ABU_196 to 42 days, but for ABU_199 the maximum storage period over which fading was assessed was 4 days. The results of these tests show considerable differences in fading patterns between IR stimulated signals and blue LED stimulated signals, and between fading rates for individual aliquots, giving rise to mean fading rates with very large error terms (Table 4).

The fading of the blue OSL signal would suggest that the IR-BSL protocol has, in the case of the samples from Mayr's Quarry, failed to completely remove all the feldspar signal prior to the measurement of the quartz signal. It is interesting therefore that the dose recovery tests undertaken on ABU_199 showed that the protocol was nevertheless capable of recovering accurate D_e values, even in the presence of a feldspar contribution to the luminescence signal. Of course this is an immediate measurement following irradiation, and should not be taken to imply that fading of the blue-stimulated signal would not occur over time.

It is clear from these experiments that fading has occurred

Sample I.D.	Number of aliquots used in age determination	Number of aliquots rejected for use in age determination	D _e (Gy)	Dose Rate ^a (Gy/kyr)	Age Determination (kyr)
ABU_196 (4 – 11 µm)	17	7	123.85±19.7	1.24±0.07	99.9±16.9
ABU_197 (4 – 11 μm)	4 – 11 μm) 1°		147.39±13.87	1.39±0.09	105.9±14.8
ABU_198 (4 – 11 μm)	16	18	53.01±22.66	1.79±0.12	29.7±12.8
ABU_199 (4 – 11 μm)	10	14	109.51±87.92	1.35±0.05	81.1±65.2
ABU_200 (125 – 180 µm)	1	5	117.64±18.12	2.64±0.13	44.5±6.9
ABU_201 (125 – 180 µm)	2	1	165.26±87.37	2.71±0.16	60.9±32.4
ABU_202 (90-125µm)	3	6	278.58±22.15	3.08±0.21	90.4±9.5
ABU_202(125-180µm)	4	2	263.55±111.77	3.00±0.2	87.8±37.7
ABU_219 (4 – 11 μm) [OSL signal]	17	7	256.09±103.61	4.73±1.12	54.1±25.4
ABU_219 (4 – 11 μm) [IRSL signal] 20 4		4	264.59±9.3	4.73±1.12	55.9±13.4
ABU_223 (90 – 125 µm)	3	15 ^d	195.09±6.28	2.58±0.13	75.6±4.5

^a Determined by NAA and field gamma spectrometry measurement. An a-value of 0.1 ± 0.01has been used in dose-rate calculations

^b Determined using U/Th dating (Spötl and Mangini, 2006)

^c One aliquot rejected as D_e/D₀ >2 (Wintle and Murray, 2006)

^d Includes 5 aliquots rejected as low-dose outliers

TABLE 3: Details of age calculations

to some degree with all samples, and that therefore the OSL ages calculated have to be regarded as minima. To obtain some idea of the consequences of the fading, corrected ages have been calculated for several of the samples, following the procedures detailed by Huntley and Lamothe (2001). The adjusted ages (Table 4) should not be regarded as highly accurate, because of the inherent assumption in the correction procedure that the signal strength follows a logarithmic decay pattern. As Aitken (1985, p.279) has observed "even for a mineral that has been shown to fade with logarithmic dependence during laboratory storage tests, the validity of extrapolating into the past is unproven". Such tests do however provide at least an order of magnitude impression of the impact which fading could have had on the OSL ages derived in the present study, but should be regarded as no more than that.

A further likely contribution to any discrepancy is the impact of calcification of the breccia on the constancy of the gamma flux experienced by the mineral grains in the loessic layers. Olley et al. (1997) indicate that in a situation not dissimilar to that found at the Mayr's Quarry section, reliance on NAA or other determinations of parent nuclide concentrations could lead to an error of the order of 6-8% in the age determinations. This of itself is clearly not enough to account for the degree of age underestimation suggested by the results. Possibly one or more of the initial assumptions used in the calculation of the dose rate is erroneous, or may not apply to every stratum for which sample ages have been determined. Further investigation is required to determine the reason(s) for the anomalously young ages of such samples, but it is most likely a combination of the factors detailed above.

7.2 PALAEDENVIRONMENTAL IMPLICATIONS

The OSL dating provides the first direct information about the age for formation of the Red Breccia, largely confirming the age suggested nearly a century ago on the basis of its being both over- and underlain by till (e.g. Penck, 1921). Although the initial OSL dates appeared to rule out an age older than the Last Interglacial (Eemian; MIS 5e), the impact of signal fading, together with the error margins on the dates, precludes unequivocal assignment of the breccia formation to a specific palaeoclimatic period or event. Two lines of observation suggest, however, that the Red Breccia was probably not formed during the Last Interglacial. First, the basal, near horizontal strata of the Red Breccia lie high above the present floor of the Inn valley, a position difficult to reconcile with an interglacial origin. It is well known that rivers worldwide show aggradation contemporaneous with cold periods (e.g., Fuller et al., 1998; Litchfield and Berryman, 2005). In the Alps this is a result of a lowered timberline, more extensive periglacial and slope erosion, and of concomitant glacier advances (van Husen, 2000). This cold climate mode contrasts with effective river incision during interglacials when sediment supply is low while annual discharge is high (higher precipitation). We note that the Inn valley preserves prominent remnants of such a palaeovalley floor some 300 m above the present valley floor dated to MIS 3 and MIS 2 (e.g., Patzelt and Resch, 1986).

Secondly, in addition to the aeolian dust layers intercalated with the debris-flow deposits forming the Red Breccia at Mayr's Quarry, there is a similar layer of dust at the interface between the breccia and the underlying till (Penck, 1921). The existence of these layers is incompatible with an interglacial vegetation cover similar to that of today. The Inn valley, the most likely source of the locally derived dust, is presently densely vegetated and most probably would have been so during the Last Interglacial. No aeolian sediments of Holocene age are known from the Inn valley, a consequence of, among other things, the lack of areas susceptible to deflation. The presence of the loessic layers at Mayr's Quarry therefore argues for a sparsely vegetated valley floor. The implication is that the climate would have been cold and stormy at times, with winds capable of entraining silt-size sediment. Two addi-

Sample I.D.	OSL Age (kyr)	g (%/decade) determined after 14 days of storage.	g (%/decade) determined by Auclair <i>et al.</i> (2003) protocol	% Age shortfall due to fading	Approximate Corrected Age (kyr)	% Age shortfall predicted by Auclair <i>et al.</i> (2003) protocol	Approximate corrected age (kyr) predicted using Auclair et al. (2003) protocol
ABU_196	99.9±16.9	10.723	25.14±1000.6	64.2	326.2±55.2	138.5	No solution
ABU_197	105.9±14.8	12.78	-	76.8	965.8±134.9	-	-
ABU_198	29.7±12.8	12.76	-	69.6	135.0±58.2	-	-
ABU_199	81.1±65.2	14.45	0.4±1.1	85.2	No solution	2.6	84.7±68.1
ABU_200	44.5±6.9	9.53	-	25.1	244.4±37.9	-	-
ABU_201	60.9±32.4	6.03	-	34.8	148.4±78.9	-	-
ABU_202 (90-125µ)	90.4±9.5	4.05	-	24.1	158.8±16.7	-	-
ABU_219	54.1±25.4	7.05	-	19.2	160.7±75.4	-	-

Calculations of fading based upon models by Huntley and Lamothe (2001)

Errors for corrected ages are calculated pro rata from errors in OSL age determinations

TABLE 4: Fading data and corrected ages (blue-stimulated signal)



FIGURE 6: Details of the recycling and recuperation characteristics for sample ABU_199 as a function of preheat temperature. The arrows point to the relevant axis for each variable.

tional observations corroborate a rather cold-climate origin of these silts: the lack of pedogenic features in these beds, and the occurrence of "loess snails" in a temporary outcrop in the same unit 1.5 km west of Mayr's Quarry (Penck, 1921; Fig. 1).

The classical key argument put forward in favour of an interglacial origin of the Hötting Breccia is its famous plant assemblage, most notably Rhododendron sordellii (formerly R. ponticum; now R. ponticum var. sebinense - Denk, 2006) and Vitis silvestris (formerly V. vinifera; Wettstein, 1892; Murr, 1926; Hantke, 1983; recently questioned by Denk, 2006). Wettstein (1892) in his monograph of the flora already emphasized, however, that virtually all the specimens were recovered from the one site (Roßfalllahner, 1150 m - Fig. 1), located 2.1 km north-west of Mayr's Quarry. The plant remains are preserved in a light grey micritic limestone overlain by the White Breccia but unknown from other outcrops. Unfortunately, stratigraphic details at this site are poorly known and cannot be verified because the area is densely vegetated. Sanders and Ostermann (2006) recently studied available specimens from this site and concluded that the former are incompatible with the thick talus succession of the White Breccia above. The Red Breccia, despite active quarrying over several decades, yielded only a few poorly preserved plant remains. A detailed study by Murr (1926) showed that this flora - dominated by Pinus needles and rare Salix leaves -



FIGURE 7: Dose recovery statistics for sample ABU_199, showing the spread of results for individual aliquots.

lacks warmth-loving elements, suggesting a rather cool climate. More recently, Denk (2006) re-examined plant fossils from the different occurrences of the Hötting Breccia and dismissed earlier claims according to which the plant fossils indicate temperatures higher than today (e.g., Heuberger, 1975; Hantke, 1983). Contrary to Murr (1926) he maintained that available palaeobotanical data do not necessarily indicate that the floras from Roßfalllahner and the Red Breccia (including Mayr's Quarry) were deposited under different climatic conditions. They may reflect different taphonomic types of floral assemblages (Denk, 2006).

In essence, palaeobotanic evidence is still inconclusive as to when the Hötting Breccia formed, although one observation appears robust: even the flora from Roßfalllahner fails to support the previously held view of an interglacial warmer than today's climate. This effectively rules out an Eemian age of this flora, as there is ample evidence from many sites in the circumalpine and central Europe realm that Last Interglacial temperatures were on average ca. 2°C higher and winters were milder than today (Drescher-Schneider, 2000; Klotz et al., 2003; 2004; Kühl et al., 2007). In conjunction with stratigraphic and sedimentological data, there is now sufficient evidence to call into question the classic interglacial interpretation of this deposit. Although the OSL dates could be consistent with a Last Interglacial age for the Hötting Breccia, we suggest that deposition of the Red Breccia was initiated either during glacial inception or during MIS 5d. High-resolution palaeoclimate records show that MIS 5d was composed of two (North Atlantic sediments - McManus et al., 1994) or three cold events (Greenland ice cores - North Greenland Ice Core Project Members, 2004) separated by warmer intervals. Palaeovegetation data indicate a steppe-tundra ecosystem existing on the northern fringes of the Alps during MIS 5d cold phases in a climate with winter temperatures of c. -13°C and summer temperatures no higher than 14°C (Klotz et al., 2004). Precipitation was also significantly reduced (by c. 50%) when compared with the preceding late Eemian conditions. The climatic deterioration during MIS 5d resulted in deforestation of the alpine valleys (the forest re-expanded during MIS 5c; Drescher-Schneider, 2000; Müller et al., 2003) and pronounced aggradation of the river beds. Under such conditions the extent of bare ground, the potential source area of fine-grained material for aeolian redistribution in the region, would be markedly increased compared with the preceding interglacial.

8. CONCLUSIONS

Examination of the fine-grained sediments intercalated with debris-flow deposits in the Hötting Breccia shows that it possesses a similar mineral suite to that of sediment presently undergoing transportation by the River Inn. This suite is very different from that found in sediments sourced on the northern slopes of the Inn valley. This points towards aeolian transport, most probably from the floor of the Inn valley, having been the primary agency supplying fine-grained sediment to the site. Such aeolian transport provides near-optimal conditions for optical resetting of the luminescence signal prior to final deposition (e.g. Aitken, 1998, Singarayer et al, 2005). Tests conducted on the luminescence characteristics of such sediments at Mayr's Quarry have demonstrated that the procedures adopted are capable of determining reliable OSL ages from these materials if quartz grains are isolated, but that fading problems affecting both the IR and OSL signals mean that fine-grain samples give ages which are best regarded as significant underestimates of the true age of deposition. This conclusion is supported by the relationship between several of the OSL ages and those obtained from the same site by U/Th analysis.

The calculated OSL ages indicate that the bulk of the Red Breccia was laid down during the late Eemian or early Würmian, at a time when the level of the valley was higher than today and ice free. Aeolian deflation and transport would have been at its most effective during dry, cold periods of restricted vegetation cover. For this reason it is considered that the bulk of the loessic sediments in the central Inn valley would probably have accumulated during MIS 5d rather than in the warmer, wetter and better vegetated conditions that prevailed during the Last Interglacial (MIS 5e). A recent re-evaluation of the classic Hötting flora (Denk, 2006) is at least partially consistent with this interpretation.

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Alastair M.D. GEMMELL^{1)*)} & Christoph SPÖTL²⁾

- ¹⁾ Geography and Environment, GeoSciences School, University of Aberdeen, Aberdeen AB24 3UF, Scotland;
- ²⁾ Institut f
 ür Geologie und Pal
 äontologie, Universit
 ät Innsbruck, Innrain 52, 6020 Innsbruck, Austria;
- " Corresponding author, a.gemmell@abdn.ac.uk

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