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Thesis Abstracts

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Thesis Title: Luminescence dating of Romanian loess using feldspars
Grade: Ph.D.
Date: October 2011
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Romanian loess-palaeosol sequences are considered semi-continuous and extended archives of climate and environmental change during the Late and Middle Pleistocene. A reliable sedimentation chronology of these deposits would improve our understanding of the regional climatic context and of the link between similar deposits in Europe and Asia. Optically stimulated luminescence (OSL) dating of Romanian loess using quartz has been recently shown to have a significant potential to test the reliability of proxy-based methods and also to provide additional information regarding dust transport and deposition. At the same time, however, OSL dating studies of the loess section near Mircea Vodă (SE Romania) revealed an intriguing dependence of age results on the grain-size fraction of quartz that was used for dating.

This work started by confirming these observations. Single-aliquot regenerative-dose (SAR) OSL dating of silt (4-11µm) and sand-sized (63-90µm) quartz grains extracted from the loess-palaeosol sequence at Mostiştea (SE Romania) yielded ages that are grain-size dependent, indicating that the phenomenon may be characteristic for loess deposits in this region. Detailed investigations into the OSL characteristics did not allow identifying the origin of this discrepancy. The results point at a hitherto unexplained mechanism in OSL production at high doses and question the reliability of obtaining SAR-OSL equivalent doses in the high dose region when a second function is needed to describe the dose response.

The potential for dating of OSL and infrared stimulated luminescence (IRSL) signals from polymineral silt-sized grains was then investigated. To this purpose, archived material of samples from the loess-palaeosol sequence near Mircea Vodă was used, and all luminescence measurements were made using a SAR protocol.

A double-SAR approach – involving successive stimulation of the polymineral fine grains with IR and blue light, and detection of the resulting IRSL and post-IR OSL signals in the UV – was tested first. Although both signals exhibited significantly different fading rates, the corrected ages are mutually consistent and in agreement with OSL ages for purified silt-sized quartz. This indicates that it may not be necessary to isolate pure quartz to obtain reliable ages for Romanian loess. Owing to the limitations of the fading-correction method, however, the approach is limited to samples from the last glacial period.

A conventional approach, in which stimulation with IR was at 50°C and the detection window in the blue, was also tested. This procedure was not able to provide accurate depositional ages due to initial sensitivity changes induced by preheating at 250°C for 60s and/or contributions from thermally unstable components after less stringent preheat treatments.

Finally, a post-IR IRSL protocol was tested, in which a first stimulation at 50°C was followed by a second stimulation at either 225 or 300°C, and detection was in the blue. Although none of the two post-IR IRSL signals seems to be affected by anomalous fading, only the post-IR IR225 signal yields ages entirely consistent with both OSL ages for silt-sized quartz and independent age control over four interglacial/glacial cycles. The post-IR IR300 signal appears to suffer from dose dependent initial sensitivity changes that hamper its use for the oldest samples investigated.

The results obtained within this work show that silt-sized feldspars can be used to date Romanian loess, thereby providing additional age control for quartz OSL ages. Especially post-IR IR dating of polymineral fine grains is very promising. The obtained age results urge the long-established chronostratigraphical framework for Romanian loess to be revised; the two uppermost well-developped palaeosols can no longer be thought of as interstadial soils that developed during the Last Glacial, but have formed during MIS 5 and MIS 7, respectively.

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Thesis Title: Fundamental and sedimentological controls on the luminescence of quartz and feldspar
Grade: Ph.D.
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The optically stimulated luminescence (OSL) characteristics of a suite of quartz and feldspar samples from a range of modern glaciofluvial sediments have been explored to determine the use of OSL as a depositional pathway tracer. Paraglacial and subglacial source material and various glaciofluvial deposits have been analysed from the glacial catchments of Bergsetbreen, Fåbergstølsbreen, and Nigardsbreen as well as the Fåbergstølsgrandane sandur, Jostedalen, Norway.

The OSL distribution signatures have been characterised through exploration of sample skewness, kurtosis and overdispersion, and dose distributions of the different depositional settings and source materials are distinct for both quartz and feldspar. Residual ages are greatest for feldspar, indicating significant potential age overestimation where feldspar is used to date glaciofluvial deposits. Sample dose distributions and overdispersion characteristics are driven by source sediment properties, whereas residual ages are controlled by transport and depositional processes. Those transport and depositional processes which result in significant light exposure, also influence dose distributions, and processes that sort sediments least effectively have the highest residual doses.

Sample OSL characteristics, transport distance and grain size distributions have been investigated using factor analysis, as a means of predicting sediment source, facies, depositional process and deposit type. Although the depositional processes of the quartz samples can be clearly differentiated based upon OSL characteristics, factor analyses of feldspar and grain size characteristics are inconclusive.

The application of quartz OSL to the Norwegian samples was limited by its very poor luminescence sensitivity. Quartz is the preferred mineral for OSL, however, despite the plethora of successful quartz OSL applications, the precise origin of the UV/blue luminescence emission, measured during OSL, remains unclear. The origins of this emission and controls on its intensity were explored using a variety of spectroscopic techniques including photo-luminescence, cathodoluminescence, radio-luminescence (RL), ionoluminescence (IL) and x-ray excited optical luminescence (XEOL).

Exciting sample luminescence at a range of energies enables exploration of the different donor centres responsible for the luminescence emission. Cathodoluminescence and RL emission spectra are similar, comprising broad emissions at 1.5, 2.0 and 2.7 eV (detection in the UV part of the spectrum was not possible for these experiments). Iono-luminescence emission spectra were dominated by the ~ 3.3 eV emission, which is a component of the signal conventionally monitored during OSL. This emission depleted as a function of dose, to the benefit of the red emission (1.8-2.0 eV) for all samples throughout IL, and similar observations were made for the 3.4 eV emission observed from the XEOL emission spectra. The XEOL spectra are dominated by an emission at ~ 3.8 eV, not widely reported for quartz, which has tentatively been attributed to peroxy linkages. Differences between the IL and XEOL emission spectra are interpreted as evidence for the presence of multiple excited states.

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Thesis Title: The timing and the environmental and palaeoclimatic significance of the late Quaternary dune encroachments into the northwestern Negev Desert, Israel
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This thesis studied the spatial and temporal characteristics of quartz-rich vegetated linear dune (VLD). The time of their encroachments from the northern Sinai Peninsula into the northwestern (NW) Negev desert, subsequent stabilization and the

triggering palaeoclimate, was established by optically stimulated luminescence (OSL) dating.

The dunefield was first divided into geomorphic units that were merged into dune encroachment corridors. Approximately twenty full dune sections at the western and eastern ends of each corridor were analyzed and sampled in outcrops or drills. GPR profiles using a 100-MHz antenna were not found to be a dependable tool for sampling-oriented identification of stratigraphic units.

Ninety-seven samples of aeolian quartz were dated by OSL. Dose recovery tests showed that in the preheat range of 220-280°C, the ratios between measured and given doses is 0.9-0.95. Recycling ratios and preheat plateaus further showed that the sediments are well-suited for the modified single aliquot regenerative-dose (SAR) protocol, that included a thermal cleaning step at the end of each measurement cycle. D_e distributions were usually normal. Tailing aliquots are attributed to contamination by bioturbation and minute contribution of underlying older sediments. Dose recovery tests showed systematic uncertainties ranging from 2% to 11%, with the greater scatter for the smaller (2 mm) aliquots. These values indicate that samples with D_e standard deviations lower than ~10% were probably well-bleached at the time of deposition. Disregarding dune crests that have high (>30%) over-dispersion, the age errors usually do not exceed ±15% and the ages are considered reliable.

Radiocarbon dates on charcoal and ostrich egg shells, and published TL and IRSL ages which were sampled at similar settings, usually agree with the OSL ages, supporting their reliability and significance, and place the entire study area within a single chronological framework.

The OSL ages cluster at ~24-10 ka, ~2-0.8 ka and 150-10 years, matching the chronostratigraphic units of the VLD axis, and representing the major dune mobilization episodes. VLDs accrete sand along their axis during mobilization (elongation) and undergo minute lateral migration. Between mobilization episodes, intermittent local reworking, regulated by strong wintertime storm winds, droughts, and vegetative and biogenic crust cover, resets the luminescence signal of the upper dune sands.

In global terms, the NW Negev dunefield is relatively young. The sand probably originated from the Nile Delta and its availability was limited by the exposure of Delta sands during glacial lower Mediterranean sea-levels. Although sand has been intermittently transported into the Negev for over 100 ka, the dunefield formed only at ~24 ka. Most of the ages cluster at ~16-13.7 ka and at ~12.4-11.6 ka, synchronous with the Heinrich 1 and Younger Dryas cold events, respectively, suggesting a link between global glacial and cold climates to dune mobilization. The earlier age cluster marks the main encroachment episode that deposited the main bulk of the Negev's sand. At 12.4-11.6 ka, dunes reached the easternmost extent of the dunefield. Based on OSL-dated stratigraphy, the episodes included several rapid incursion events. Encroaching dunes dammed wadis, forming standing-water bodies that supported short-term Epipalaeolithic camp sites.

Calculated dune transport rates that incorporate the range of the OSL age errors are in the range of ~5-25 m/yr. The Negev VLDs encroached in a windier but probably wetter climate than today, providing better conditions for vegetation growth, although when dunes elongated, vegetation was suppressed. It is suggested that the decrease in global windiness between the LGM and the Holocene, as indicated from dust records in ice cores, resulted in global lower-latitude dune stabilization.

The late Holocene (2-0.8 ka) mobilization episode varied from 10 m-thick transverse dune formation to VLD incursion in the western dunefield, and deposited 1-2 m of sand in other parts of the dunefield. Dune erodibility may have resulted from man-induced decimation of vegetation and biogenic crusts. OSL ages, compatible with anthropogenic land-use changes, mark intermittent sand activity and stabilization in the last 150 years, though dune elongation did not ensue.

Sand redness, spectroscopically defined by the redness index (RI) ($RI=R^2/(B*G^3)$), reflecting the amount of iron-oxide quartz-grain coatings, does not vary greatly down the Negev dune sections and across encroachment corridors. RI intensities and the OSL ages of the sand are not correlated and sand grain rubification may have been minimal since deposition or inherited from its Nile source.

The recurring discontinuous aeolian sedimentation pattern found in OSL-dated VLDs provides new and important chronological and sedimentological insight into dune mobilization and stabilization processes, while demonstrating the sensitivity of dunes located along the fringe of the sub-tropical desert belt to climate change and sediment supply. The suggested link between global reductions in cold-climate windiness and low-latitude dune stabilization episodes emphasizes the dominant effect of windiness on major dune mobilizations in low-latitude dunefields even if they are partially vegetated.

This thesis is available as a PDF on the Ancient TL web site www.aber.ac.uk/ancient-tl

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Title: Numerical simulations and modeling of dosimetric effects in Quaternary sediments: application to luminescence dating methods.
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Luminescence dating methods are based upon the following age equation:

$$\text{Age (a)} = \text{Palaeodose (Gy)} / \text{Dose-rate (Gy.a}^{-1}\text{)}$$

Whereas research on the determination of palaeodose has made significant progress during the last decade, research on dose rates has severely lagged behind. It has long been known that spatial variations in the distribution of radioactivity in sedimentary media at different scales (the range of the different particles/rays involved: alpha, beta, gamma) is limiting the precision and accuracy of luminescence dating methods. However the difficulty in obtaining experimental data has prevented progress in this area of research. In order to resolve this issue, the particle-matter simulation toolkit Geant4 was used in this thesis to study the effects of heterogeneities in sedimentary media.

A series of simulations were designed to refine a field gamma spectrometry technique, with the aim of improving accuracy and precision while reducing measurement times. The results were then used for the experimental calibration of a gamma ray probe. Comparisons between experimental and numerically simulated results of gamma ray spectrometry were very satisfactory and placed the new technique on a secure footing. GEANT4 was then used to define a non-invasive approach, based on surface measurements, which would be adapted to archaeological excavations.

On a grain scale, numerical simulations of dosimetric effects with GEANT4 revealed the limited validity of the widely used concept of infinite matrix in palaeodosimetric dating methods. The initial step consisted of studying the effect of water on dose rates received by sedimentary grains, an exercise that was conducted in simple geometries. This became the starting point for a review on the use of the infinite matrix assumption and associated concepts. Adequate tools for quantifying a number of identified microdosimetry effects, such as the consequence of radioelement hotspots (e.g. potassium in feldspar grains), were developed and are anticipated to form the basis for a better accuracy in single grain OSL dating methods.

The final part of the thesis focussed upon a chronological study of the Mousterian sequence of Roc de Marsal, which is one of the key sites for the Middle Palaeolithic in South West France. Complex gamma and beta dose rate patterns were identified and their consequences on the ages obtained from heated flints (TL) and quartz samples (OSL) were discussed. The results were then viewed in their palaeoclimatic and palaeoenvironmental context, documented both at a local scale by faunal remains and at a regional scale by pollen studies from a marine sediment core. This chronological study helped placing human occupations on an absolute time scale together with the environment in which the studied Neanderthals were evolving.

The thesis manuscript is partly written in French; however the main results have been published in international journals. As a result, seven chapters consist of papers published in English. This thesis is available as a PDF on the Ancient TL web site www.aber.ac.uk/ancient-tl. Supplementary data is available on request to the author.