

Thesis Abstracts

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Ingrid Bejarano Arias

Determination of depositional environment and luminescence dating of Pleistocene deposits in the Biely Váh Valley, southern foothills of the Tatra Mountains, Slovakia

June 2020

Lund University, Lund, Sweden

Degree: M.Sc.

Supervisors: Helena Alexanderson, Isa Doverbratt and Juraj Janočko

The Tatra Mountains have had several glaciations during the Quaternary. The deposits located in the southern foothills were classified as glaci-fluvial, based on geomorphology, but its sediments have not been studied in detail. Therefore, this project focused on establishing the depositional history, processes, and age of the deposits in a gravel pit, located in the Biely Váh Valley in these foothills. The combination of luminescence dating with sedimentological analysis, including clast shape and maximum particle size, were the methods of choice. For the luminescence dating eleven samples were collected from the units composed mainly of sand.

Initially optically stimulated luminescence (OSL) dating was used, but after applying the standard Single Aliquot Regeneration (SAR) protocol quality tests, it was evident that the quartz was poorly behaved with low signal intensity. The dose estimation gave bad results, even after attempting pulsed OSL and differential OSL. Therefore, it was decided to move on to feldspar grains. Given that there is a risk of fading and the fact that feldspar grains take longer to bleach, the corrective measurements were done by calculating the *g*-value and use of IR₅₀ and pIRIR₂₂₅ signals for the bleaching.

The sedimentological description yielded 13 units from the lower and upper outcrops in the study area. The results of the sedimentological analysis indicated that the process of deposition had a high energy component, representative of

subaerial flows. Given the features of the sediments it was identified as a hyperconcentrated flow. Moreover, for the age determination the most likely water content of the sediments was chosen, the *g*-values and the equivalent dose were determined, using both IR₅₀ and pIRIR₂₂₅ signals. Subsequently, four ages were obtained per sample, one for each signal, and with and without correction for fading. From these, the uncorrected pIRIR₂₂₅ ages were selected, since this signal has a lower fading rate which is supported by the obtained *g*-values, which were smaller than 1 – 1.5%.

The results were compared to the known glacial history of the Tatra Mountains. The obtained luminescence ages, which range from ca 200 to 260 ka, would correspond to the Riss glaciation. Both ages and sediment characteristics match those of the other deposits from the northern and southern Tatra foothills that also have been correlated to the Riss glaciation. Taken together, these results suggest a glacial advance to the present-day foothills of the High Tatras during the Riss glaciation and extensive deposition of glaci-fluvial sediments outside the ice margin.

Galina Faershtein

Extending luminescence dating into the early Pleistocene – method development and application to the eastern Mediterranean coastal plain

January 2020

Hebrew University, Jerusalem, Israel

Degree: Ph.D.

Supervisors: Naomi Porat, Ari Matmon

The aim of the thesis was to explore the extended-range luminescence dating techniques that have the potential of dating sediments throughout the middle to early Pleistocene. Numerical dating of continental clastic sediments of Pleistocene age, particularly early Pleistocene, has always been a challenge. The luminescence dating techniques, and particularly the optically stimulated luminescence (OSL), are strong tools for dating late Pleistocene to late Holocene sediments from different geological settings. However, the common OSL technique is usually limited to 100-200 ka due to signal saturation. Recent developments of new extended-range luminescence methods show great potential for dating older sediments of middle and even early Pleistocene. For quartz, the techniques include the thermally transferred OSL (TT-OSL) and violet stimulated luminescence (VSL). Using these signals, ages of up to 1 Ma have been reported. For alkali-feldspars, the post-infrared-infrared stimulated luminescence at elevated temperatures (pIRIR) protocols have been used

to obtain ages of over 500 ka. There are unsolved issues concerning the optimal use of these techniques such as exact measurement protocols, accuracy, and effective time range. This thesis deals with some of these problems in an attempt to resolve them.

The Israeli Coastal Plain sequence spans the entire Quaternary and comprises mainly aeolian sediments. The mineral grains (such as quartz and alkali-feldspar) are all derived from a single source: the Nile river. This setting is a perfect natural laboratory for investigating of the different luminescence signals. This study focussed on two representative sections: Kerem Shalom (sand) and Ruhama (loess). The extended range signals in quartz and feldspar were systematically investigated; the upper limits of the signals were defined through natural and laboratory saturation and thermal stability of their source traps.

In nature, luminescence signals usually reach saturation at lower doses than in the laboratory. Natural saturation profiles (created by plotting the natural normalized signals of samples against their depth) constructed for OSL and TT-OSL (for Kerem Shalom and Ruhama), as well as VSL and pIRIR₂₅₀ (for Kerem Shalom), were found to be a powerful tool for detecting saturated samples in the studied sections. Semi-natural dose response curves (DRC), constructed by plotting natural normalized signals against their laboratory equivalent doses (for OSL, TT-OSL, pIRIR_{250,290}), using samples from these two sections, as well as from other sites in the region, characterize Nilotic quartz and feldspar and define their typical DRC's. The semi-natural OSL DRC is unique to the quartz of Nilotic origin and indicates that the upper limit for reliable OSL dating of this quartz is 140 Gy due to signal saturation, corresponding to 45–280 ka (for typical environmental dose rates of 0.5–3 Gy ka⁻¹).

The TT-OSL semi-natural DRC is also uniform for the Nilotic quartz. This signal is limited by the low thermal stability of its source trap as was indicated for the Nilotic quartz by combination of field data and laboratory techniques (varying heating rates, isothermal decay, alongside several models). Isothermal decay data exhibits significant departures from first-order kinetic behavior; however, extrapolations of these models imply first-order behavior over geological timescales. The TT-OSL lifetime is assessed at ~550 ka for the eastern Mediterranean Pleistocene climate. Age underestimation simulations demonstrate that the environmental dose rate of the sediment significantly affects the upper limit of the attainable TT-OSL ages. Nevertheless, for a specific storage temperature, TT-OSL age underestimation exceeds 10% after approximately the same time period for virtually all sedimentary environments and dose rates. Thus, TT-OSL ages over 200 ka should be treated with caution as minimum ages.

The pIRIR signal stimulated at 250 °C (pIRIR₂₅₀) was chosen for dating Nilotic feldspar since it displays a preferable balance between bleaching time and recovery ability. The semi-natural DRC of this signal shows some variability in natural saturation for the Nilotic feldspar. Natural saturation is most likely reached at 500 Gy, when equilibrium

between electron trap filling and athermal de-trapping is obtained. Nilotic feldspar pIRIR₂₅₀ ages derived from higher equivalent doses are probably minimum ages.

Application of the extended range methods to the Kerem Shalom sediments produces accurate ages up to 200 ka and minimum ages up to 715 ka. The new ages are significantly older than the known luminescence chronology of the coastal sediments in the central and northern parts of Israel which presently are attributed mostly to the last 70 ka. It suggests the presence of sands in the southern coastal plain already in the Early Pleistocene. Even though the early Pleistocene is still out of reach for accurate luminescence dating, middle Pleistocene sediments can be successfully dated with these methods.

A PDF of this thesis can be downloaded from: by contacting the author: galina.faershtein@weizmann.ac.il.

Nicola Horsburgh

REE induced defects in minerals: A Spectroscopic Study

June 2020

School of Earth & Environmental Sciences, University of St Andrews, Scotland, UK

Degree: Ph.D.

Supervisor: Adrian Finch

This thesis examines the luminescence and mineral physics of Rare Earth Element (REE) bearing minerals as a precursor to developing smart sorting tools for critical metals used in low-carbon technologies. I characterise luminescence responses of complex zirconosilicates; eudialyte (Na₁₅Ca₆(Fe²⁺, Mn²⁺)₃Zr₃[Si₂₅O₇₃](O, OH, H₂O)₃(OH, Cl)₂), wöhlerite (NaCa₂(Zr, Nb)(Si₂O₇)(O, OH, F)₂) and catapleite (Na₂Zr(Si₃O₉) · 2H₂O). Fluorite was included as it is commonly associated with REE ores and displays strong REE luminescence. Its behaviour provides key insights into REE substitution into ionic minerals.

X-ray Excited Optical Luminescence (XEOL) and Thermoluminescence (TL) measurements were taken from 20 to 673 K. Fluorite responses result from a balance of intrinsic luminescence and REE substituents and evidence for REE and defect coupling. Thermoluminescence indicates the presence of electron traps and the coupling of these traps to lanthanide emissions show that the defect and the lanthanide are clustered in physical space. The absence of changes in TL for different lanthanides shows that energy is passed efficiently between rare earths, indicating that the REE are clustered.

The zirconosilicates all show increased intensity in XEOL response below 150 K. Cryogenic emissions are interpreted as originating from the host mineral. There are 3 shared features: UV (~280 nm) paramagnetic oxygen or oxygen vacancy; blue (440 nm) Al-O-O /Ti centres; and REE. Wöhlerite and eudialyte show Fe³⁺ band (~708 nm) and wöhlerite displays broad emission attributed to Mn²⁺. Eudialyte shows two additional responses; UV (~320 nm) ten-

tatively assigned to Na migration and UV/blue (~ 400 nm) potentially associated with charge balances associated with the coupled substitution of Al^{3+} . Eudialyte shows little emission at room temperature, this is attributed to quenching from Fe^{2+} . Emission from eudialyte above room temperature is attributed to alteration minerals such as catapleiite and potentially to inclusions of luminescent primary mineral phases.

I demonstrate that smart sorting could be a valuable beneficiation tool for REE minerals.

Further information on this thesis can be downloaded from: <https://adrianfinchcoulk.wordpress.com/2020/06/21/ree-induced-defects-in-minerals-a-spectroscopic-study-phd-thesis-by-nicky-horsburgh/>

Jandessa Silva de Jesus

Late Quaternary evolution of the Middle Tocantins River in Eastern Amazon

March 2020

Institute of Geosciences, Universidade de São Paulo, São Paulo, Brazil

Degree: M.Sc.

Supervisors: Fabiano do Nascimento Pupim and André Oliveira Sawakuchi

The Amazon River forms the largest fluvial system on Earth and its dynamics and evolution play a key role in biogeochemistry cycles, ecological services, and biological diversity. The Tocantins River is the largest system that drains the eastern Amazon. However, the Quaternary history of the Tocantins River is poorly known due to lack of geomorphological and geochronological data. Therefore, we use geomorphological, sedimentological and optically stimulated luminescence dating (OSL) techniques to the morphosedimentary evolution of the middle reach of the Tocantins river during the Late Quaternary. Three main geomorphological units were mapped: (i) fluvial plain, (ii) fluvial terraces and (iii) paleo-alluvial fan. The OSL ages from 32 samples show ages between 661 ± 42 years and 160 ± 16.3 ka. The luminescence properties show bright quartz grains, fast signal decay, exponential growth of dose-response curves and adequate ability to recover doses of radiation. The Single-Aliquot Regenerative (SAR) dose protocol was applied for estimates of the equivalent doses (D_e). Equivalent doses range from 1.6 ± 0.1 to 166.1 ± 11.5 Gy. Most of samples shows D_e dispersion below 30%, suggesting a well-bleached sediment not affected by post-deposition mixing; only five samples show relatively high OD values, between 30 and 60%. Dose rates ranged from 0.238 ± 0.015 to 3.02 ± 0.238 Gy/ky. Our data indicate three stages of aggradation and two stages of incision. The older aggradational stage is represented by sediments from Upper Terrace (T1) and the Paleo-alluvial fan and occurred between 160 to 32 ka. Subsequently, an incision event occurred at ~ 31 ka, which resulted in abandonment of T1. The second phase of aggra-

tion is recorded in the Lower Terraces (T2) and promotes a reactivation of the Paleo-alluvial fan from 31 to 6 ka. A new incision occurred about 6 to 5 ka, allowing the abandonment of the T2 and reducing the local base level to its current position. The modern floodplain was built from 5 ka to the present, with sediment deposition due to lateral migration of the Tocantins River. The results presented a highly diversified landscape in terms of geoforms, sedimentary and geochronological aspects that records the geomorphological evolution throughout the Late Quaternary. The geochronology results were correlated with regional paleoclimatic data pointed the climate change as the main drive of the evolution of the Tocantins river in the last 160 ka. The evolution of terraces in this region of the Amazon appears to be synchronous with terraces in Central and Western Amazonia, suggesting that the fluvial systems of the Eastern Amazon, which drain land from Central Brazil, present fluvial responses similar to rivers with headwaters in Andean lands. Reinforces that climatic fluctuations are a key factor in the Quaternary evolution of the Amazon fluvial system.

Şule Kaya-Keleş

Investigation of radiation induced defects of natural quartz: A combined TL, OSL and EPR study

February 2020

Institute of Nuclear Sciences, Ankara University, Ankara, Turkey

Degree: Ph.D.

Supervisors: Prof. Dr. Niyazi Meriç, Dr. George S. Polymeris

Quartz is one of the most preferred mineral inclusions used in retrospective dosimetry. Its crystal structure as well as its impurity content and concentration and the lattice defects exhibit diversities according to its origin. Therefore, it is important to the luminescence properties of natural quartz. In the present work, un-heated, pre-dosed quartz is investigated towards its effective applications such in retrospective dosimetry, dating and as a reference quartz material. The purpose of the study is to find a correlation between Thermoluminescence (TL) (high temperature TL peaks), Optically Stimulated Luminescence (OSL) (both continuous wave (CW) and linear modulation (LM)) and Electron Paramagnetic Resonance (EPR) signals. The present work indicated a triple correlation between the fast CW-OSL component and certain peaks of LM-OSL and TL while all these luminescence entities are attributed to the $C2_{EPR}$ at g_2 (Landé splitting factor) ($g_2 = 1.9975$). The present study provides experimental evidence that (i) one TL peak might be the source for more than two LM-OSL components and (ii) even two different TL peaks can contribute to the same OSL component. Furthermore, on the EPR signal, besides the well established Ge center, another un-identified component has been resolved.

Xue Rui

A chrono-stratigraphic investigation of lacustrine-fluvial deposits of the Nihewan Basin in the last 0.8 million years

June 2020

School of Earth, Atmospheric and Life Sciences, University of Wollongong, Australia

Degree: Ph.D.

Supervisors: Bo Li, Richard 'Bert' Roberts, Tim Cohen

The Nihewan Basin, northern China, is a key region to study Quaternary paleoenvironmental, paleontological and Paleolithic histories in East Asia. Previous studies showed that this basin was covered by a lake (i.e., the Nihewan Paleolake) during the Early and Middle Pleistocene. This lake was drained by a river (i.e., the Sanggan River) during the late Middle Pleistocene. The lake and river have deposited thick sequences of fluvial-lacustrine deposits, containing rich assemblages of mammalian fossils and stone artefacts. Although many studies have been carried out in this basin for decades, some critical questions remain unanswered, among which three of them form the focus of this thesis: 1) when, how and why did the Nihewan Paleolake disappear; 2) when did the terraces of the Sanggan River form; 3) what is the relationship between human occupation and environmental change in the Nihewan Basin.

Addressing the above questions rely on of the establishment of firm chronological control for the middle and late Quaternary sediments in the basin. So the overarching aim of this thesis is to develop a robust chronological framework for the mid-to-late Pleistocene geomorphological evolution of the basin. A series of newly developed luminescence dating techniques were used to date the sediments associated with lake-level history of the Nihewan Paleolake, human occupation, and the terraces of the Sanggan River. The suitability of luminescence dating on quartz and potassium-rich feldspar (K-feldspar) from this region were investigated; this includes studies of the thermal stability of the optically stimulated luminescence (OSL) signals from quartz, residual and anomalous fading of the post-infrared infrared stimulated luminescence (pIRIR) from K-feldspar, and standardised growth curve of K-feldspar using single aliquots, multiple aliquots and single grains. The performance tests and comparison with independent age controls suggest that quartz OSL is unstable and cannot be used for dating the sediments from this region, whereas K-feldspar pIRIR signals are stable and can provide robust age estimates for samples deposited up to ~780 thousand years ago (ka).

To develop a chronological framework for the history of the Nihewan Basin, sediments from 12 lacustrine sections in the east of the Nihewan Basin were measured by both the single-grain and single-aliquot pIRIR procedure on K-feldspar. Our dating results revealed four periods of high lake levels at 520–551, 411–430, 298–338 and ~168 ka, respectively. Twelve fluvial sections from three Sanggan River terraces (T3, T2 and T1) were identified according to the

field investigation, and the single-grain pIRIR dating result for these sediments indicates that the formation ages of three terraces are 124–151, 25–9 and last 2 ka, respectively. The last high lake level at 168 ka and the oldest fluvial terrace at 151 ka suggests that the Nihewan Paleolake was drained between 151 and 168 ka. Combining the chronology framework with the geomorphological and paleoclimate information in the Nihewan Basin, tectonic movement is most likely the primary factor affecting the demise of the Nihewan Paleolake. By summarizing the published locations of the archaeological sites, we suggest that the hominids settled on the eastern edge of the Nihewan Basin when the paleolake existed. After the demise of the Nihewan Paleolake, they settled near the Sanggan River and spread to the whole basin.

Javier Andrés Sandoval Andrade

Datación de restos humanos prehispanicos a través de esmalte dental usando Resonancia Paramagnética Electronica (EPR) (Dating of Pre-Hispanic human remains through tooth enamel using Electron Paramagnetic Resonance (EPR))

June 1020

Universidad Nacional de Colombia, Bogotá, Colombia

Degree: B.Sc.

Supervisor: Alí Ocal

This thesis presents the experimental procedure followed dating through Electron Paramagnetic Resonance (EPR) pre-Hispanic tooth enamel from the Checua archaeological site (Nemocón, Cundinamarca, Colombia). This work represents a pioneering effort in the development of archaeometry in this country, and in turn is consolidated as the first absolute date determined through EPR in Colombia.

The Checua site has one of the earliest dates in Colombian archaeology (ca. 7800 14C yr BP). Another of my goals in this work was to compare the EPR dates with previous 14C one, testing the technique's efficiency in age assessment for early Colombian sites.

I used two molars from one human skull found at the site between 70 and 80 cm, a stratum related to a semi-permanent hunter-gatherer camp. A mechanical method with liquid cooling was used to separate the enamel from the other dental layers. Then the AIEA protocol for disinfection and treatment of the sample was implemented. Once ready, the enamel was pulverized into grains not exceeding 5 mm and divided into 10 equal parts by weight or aliquots. Each aliquot was irradiated using a Siemens Primus Accelerator (LINAC 3995) at a dose rate of 0.5 Gy / min. The doses supplied were in the range between 0 to 70 Gy. After irradiation, the EPR signal was measured with a Bruker ESP spectrometer in band X. The peak-to-peak height of the signal with perpendicular g was used to calculate the amplitude. After the EPR measurements, the aliquots were irradiated with additional doses and the process was repeated until 10 points of EPR signal were obtained as a function of the dose.

Additionally, sediment samples from the Checua site were collected for measurement of U, Th and K through gamma spectrometry. This process was carried out by the group of nuclear technologies of the Colombian Geological Service. ROSY software was used to calculate the age of the tooth enamel sample. Some parameters such as the cosmic radiation dose ratio of the area (546 $\mu\text{Gy}/\text{year}$), the content of U, Th and K obtained from the sediment analysis and the thickness of the dental layers of the sample: enamel (1.4mm) and dentin (5mm) were necessary. The other software defaults were used. Thus, finally, the estimated age in the analyses, for the study sample, was 7850 ± 190 years BP. In this way, the final date obtained through EPR shows an excellent correlation with the previous radiocarbon date, thus motivating the implementation of dating protocols with EPR of tooth enamel to date early archaeological sites in Colombian territory.

A PDF of this thesis can be downloaded from: https://www.researchgate.net/publication/340827271_DATAACION_DE_RESTOS_HUMANOS_PREHISPANICOS_A_TRAVES_DE_ESMALTE_DENTAL_USANDO_RESONANCIA_PARAMAGNETICA_ELECTRONICA_EPR

Aayush Srivastava

Late Quaternary dune activity in the Thar Desert and its implications for palaeoenvironmental reconstructions: an insight from optically stimulated luminescence dating

January 2020

University of Oxford, Oxford, UK

Degree: Ph.D.

Supervisors: Julie Durcan, David Thomas

Sand dunes in the Thar Desert, owing to their location in the Indian summer monsoonal (ISM) regime, are rich archives of past geomorphological and palaeoenvironmental changes. However, existing dune records are not robust enough to allow their assessment and integration with a growing multiproxy framework of palaeoenvironmental change. Therefore, with an aim to investigate the timing of dune activity in the Thar, three research questions are answered in this thesis: (i) what are the key phases of dune activity in the desert; (ii) what is the effect of anthropogenic influences on the landscape; and (iii) how can dune chronologies be interpreted most effectively to reconstruct late Quaternary environmental changes? To answer these, systematic sampling of multiple dunes was carried out in the dunefields in the central and northern Thar, and luminescence dating was used to provide a detailed chronological framework.

Results demonstrate that the Thar dunes are > 58 ka old, with preserved accumulation phases throughout the Holocene at $\sim 11.6-8.5$, $4-3$, $2-1$, $0.6-0.2$ and 0.07 ka. First records of modern net accumulation are presented, with rates varying between $2-5$ m/year, attributable to anthropogenic disturbances. Dune accumulation intensity methodology, in conjunction with available highly-resolved marine

and terrestrial datasets, is used to infer palaeoenvironmental changes in the Thar. Whilst the strengthening of the ISM has been evidently shown to have a significant influence on widespread dune accumulation in the early Holocene, equivocal relationships between both lake levels/dune accumulation and monsoon variability during later Holocene suggest a complex interplay of regional and more local drivers like sediment supply, lowered sea levels etc. The study concludes with emphasis on the importance of recognising external and local controls on dune systems, and demonstrates that diverse responses to same environmental stimuli should be expected in palaeoenvironmental reconstructions.

Roos Marina Johanna van Wees

Combining luminescence dating and sedimentary analysis to derive the landscape dynamics of the Velická Valley in the High Tatra Mountains, Slovakia

June 2020

Lund University, Lund, Sweden

Degree: M.Sc.

Supervisors: Helena Alexanderson, Isa Doverbratt and Juraj Janočko

Luminescence dating is widely used in combination with sedimentological analysis to reconstruct the landscape development of the Quaternary period. Here, quartz and K-feldspar measurements are used to date and evaluate the luminescence characteristics of sediments from seven sites of the lower part of the Velická valley (995–1250 m a.s.l.) in the High Tatra Mountains, Slovakia.

The quartz and K-feldspar grains are separated, and multiple quality and control tests are carried out to test the quality of the minerals. The observed characteristics and unreliable results of the quartz are appointed to the weak optical stimulated luminescence signal and the saturation of the signal, this led to using K-feldspar stimulated by infrared stimulated luminescence (IRSL) for further dating of the sampled sediment at the sites. The age of deposition of the sediments is derived from the combined uncorrected and for fading corrected post-IR IRSL at 225°C (pIRIR225), the corrected ages were applied for the samples containing a fading rate exceeding the 3%/decade. From the sampled and dated modern analogues (samples from next to the river; site 6 and 7) could be inferred that the K-feldspar was significantly incompletely bleached. Moreover, for the four oldest samples the pIRIR225 signal showed to be saturated.

The results of the dating and the sedimentology are put in the context of landscape dynamics. The deposition of the sediments (except for the modern analogues) dates back to the Middle Pleistocene epoch. Sites 4, 1, 2, and 5 (in order from old to young) are correlated to the Riss I and Riss II glaciation and the Riss II/Würm interglacial. From the evidence that the clasts have been reworked by water, the planar beds, and other evidence the sediments on site 1, 2, 4, and 5 are interpreted to have been transported by different kinds of

subaerial flows. Based on the ages and the maximum particle size the ice margin is hypothesized to have been around one kilometre north of the study area during the deposition of the sediment of sites 1 and 2. From this setting and the observations of the sediments, there can be derived that these sediments have been deposited in a proglacial outwash plain. After the deposition of the sediment at site 1 (171 ± 20 ka) the vertical incision of the Velická valley is believed to have taken place, enhanced by an increase of discharged meltwater from the glacier during a glacial to interglacial transition and the sudden sediment release from the outwash plain generating an erosional pulse. Afterwards, when the input of meltwater decreased and a low energy environment took place, the terraces were deposited (142 ± 14 ka) in the Velická valley during the Riss II/Würm interglacial.