

Thesis Abstracts

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Sahar al Khasawneh

Testing the New Applications of Luminescence Dating in Archaeology

December 2015

Institute of Ancient Near Eastern Archaeology, Berlin, Germany

Degree: Ph.D.

Supervisors: Dominik Bonatz, Andrew Murray

With recent major improvements in luminescence dating resulting from new developments in methodology and instrumentation, luminescence dating has become a much more powerful chronometric tool. These new developments have been widely used in geological applications, but there are very few publications testing their applicability in archaeology. This study aims to test the applicability of two major new techniques in luminescence dating to archaeological problems. In the first part, we report the first application of using the stable infrared stimulated luminescence signal measured at elevated temperature (290°C) to pottery samples from Pella, Jordan. The average Optically Stimulated Luminescence (OSL) age is 2850 ± 220 years ($n=35$), with the overall uncertainty dominated by systematic uncertainties; this agrees well with the range of ^{14}C ages of 2970 to 3270 cal. years BP for the same destruction horizon.

The method is also applied to the dating of young heated artefacts from three different archaeological sites in Denmark, one from the early Pre-Roman Iron Age 200 BC to AD 100, and two from the Viking period between AD 800 and 1200. No previous radiometric dating has been reported for these sites. Quartz OSL ages were derived to support the archaeological associations, and compared to the new feldspar ages. On average, there may be a small overestimation of feldspar ages compared to those from quartz, but if so it is only of significance for the heated stone samples.

The second part of the research investigated the application of OSL dating to sediment deposition in the arid zone directly resulting from human activities (well digging and

construction of irrigation channels). Quartz and feldspar luminescence signals were both measured to provide two estimates of luminescence ages, and both ages are in agreement with each other, and with the archaeological assumption of the site age.

In the last part, a very recently developed technique, rock-surface dating, was applied to a three rocks sampled from the Jibal al-Gadiwiyt kite structure in southern Jordan. By examining the resetting of OSL with depth and time, this approach was able to provide detailed information about the burial and light-exposure history of the rock. The derived construction and burial ages (based on rock-surface dating but confirmed by sediment dating) are 10,000 years; considerably older than the archaeologically assumed age of 6000 years.

By using a variety of archaeological sites, this study demonstrates the immediate usefulness and future potential of recent developments in OSL dating. The accuracy of these and other luminescence dating methods, and the ability of luminescence to date otherwise undateable material, opens up many new and exciting opportunities in archaeology for the future.

Lukas Bickel

Luminescence dating of Middle Pleistocene glaciofluvial sediments of the Austrian Northern Alpine Foreland

March 2016

Institute of Applied Geology, University of Natural Resources and Life Sciences, Vienna

Degree: Dr. nat. techn.

Supervisors: Markus Fiebig, Christopher Lüthgens, Johanna Lomax

In the beginning of the 20th century, Albrecht Penck & Eduard Brckner (1901/1909) developed the concept of four large scale Quaternary alpine glaciations extending into the alpine foreland. Since then, the Northern Alpine Foreland (NAF) has played a major role in the investigation of glacial and furthermore paleo-climatic events. However, a numerical chronology has as yet not been established. This study focuses on dating the glaciofluvial deposits attributed to the penultimate glaciation (parallelized with MIS 6 in Austrian geological maps) when vast areas of the inner Alps were glaciated. In the easternmost part of the north draining valleys of the Alps, the glaciers did not reach the foreland, but formed valley glaciers confined by the mountainous terrain. Samples for luminescence dating purposes were taken from glaciofluvial sediments mainly deposited in the form of river terraces in the alpine foreland. A total of 24 samples from

5 catchment areas (from East to West Ybbs, Enns, Steyr, Krems, and Traun) were analysed within this study.

A highly dynamic depositional environment, such as a glacier-fed river system, implies the possibility of incomplete resetting of the luminescence signal in particular when transport distances are short. In an environment like this, quartz is the mineral of choice over feldspar, especially if dose rates are low and theoretically allow gaining quartz ages beyond 150 ka. However, detailed analyses of the quartz OSL signal characteristics had revealed the presence of a thermally unstable medium component in some samples. Because of the lack of independent age control, it remained unclear whether this medium component may result in significant age underestimation for affected samples. Therefore, the luminescence properties of coarse grain feldspar were analysed and revealed a general suitability for luminescence dating purposes. To obtain reliable age estimates for the samples, three luminescence signals were investigated for each sample: blue stimulated quartz OSL, infrared stimulated feldspar luminescence at 50°C (IRSL) and at an elevated temperature of 225°C (pIRIR).

Using a comparative dating approach, it was possible to establish a reliable chronology for the glaciofluvial deposits attributed to the penultimate glaciation in the NAF. A temporal correlation between the marine isotope stage 6 and the deposits of the penultimate glaciation in the NAF could hence be scientifically proven. For the first time it was possible to establish a methodologic and stratigraphic coherent age determination of the formation of terrace sediments during the penultimate glaciation in the Austrian NAF. Additionally, in comparison with previous studies from other alpine regions it was possible to show, that the transition between glacial and interglacial conditions happened relatively fast in late MIS6 / starting MIS5.

Adelphine Bonneau

Direct dating of rock art using radiocarbon and optically stimulated luminescence: the case study of southern Africa and the Canadian Shield

May 2016

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Degree: Ph.D.

Supervisors: Prof. Michel Lamothe, Prof. Daniel Arseneault, Prof. Thomas Higham

Rock art exists all around the world. It is thought to be one of the most ancient expressions of the human mind. The artist, who created these paintings, opens a window into its world and gives the viewer a unique insight into its motivation and inspiration. Over the last 20 years or so, developments in the application of chemical, physical and geological methods have made it possible to recover the recipes used by

ancient painters as well as to determine the age of the art itself. The interpretation of ancient rock art from around the world has been considerably aided by the acquisition of such data. However, there is still much to do. Palaeolithic rock art in Europe and Australia were the main beneficiaries of these developments. In other parts of the world, such as southern Africa and Canada, few dates have been obtained, and little in the way of paint characterisation studies exists.

This Ph.D. project explores direct dating of rock art from sites in southern Africa and in the Canadian Shield, using radiocarbon and optically stimulated luminescence. It introduces a new preliminary detailed characterisation of the sample to be dated, which makes it possible to select the samples which are the most likely to be successfully dated. Moreover, it reduces the necessary size of the sample. Using complementary instrumentation and methods, the characterisation results bring relative dating information if linked with superpositioning information, and where no superpositioning exists, or information is not available, they can give clues as to the composition and history of a panel or a site.

Characterisation reveals the type of carbon-based paint used, which can then be radiocarbon dated, for example, charcoal, soot, or carbon-blacks. This information is essential for understanding the age obtained. At the same time, this characterisation records the presence and estimates the proportions of radiocarbon contaminants in the samples, such as calcium oxalates, calcium carbonates and humic acids. A chemical pre-treatment can then be adapted to dissolve all the contaminants detected, checked with Fourier-Transform Infra-Red (FTIR) analysis. Using these methods, 46 dates were obtained on rock art sites from southern Africa. These include the first ever dates from rock art in Lesotho and in Botswana and constitute the largest dating project on rock art ever undertaken in this part of the world.

Optically stimulated luminescence (OSL) cannot give a definitive date for rock art, but the experiments carried out for this project proved that this technique may be applied under specific conditions: where the rock support is suitable for OSL dating, given a thick paint layer and sufficient exposure of the rock face to daylight.

In the Canadian Shield, none of the dating methods was applicable. However, the precise characterisation conducted on the paints reveals different paint recipes in the same site giving new clues to reconstruct some parts of the "chaîne opératoire" of the site.

Characterisation of paint and dates obtained in this project make it possible to start developing a dialogue between the archaeological record of hunter-gatherer activity preserved in paint and that preserved in occupational deposits.

A PDF of this thesis can be downloaded from: <http://www.archipel.uqam.ca/>

*Kathleen Rodrigues***OSL dating of a coastal Swift Creek occupation at Harrison Ring, Bay County, Florida**

September 2015

McMaster University, Hamilton, Canada

Degree: *M.Sc.*Supervisor: *W.J. Rink*

A total of 17 samples were collected for OSL dating from a Swift Creek archaeological site, known as Harrison Ring, which lies on the Tyndall Air force peninsula in northwest Florida. High-resolution vertical sampling conducted at 10 cm intervals from the surface was performed in order to determine the timing of occupation at the site, and to look for patterns in radiation dosimetry and post-depositional disturbance that can compromise OSL results. We find OSL ages determined using both 0.5 mm aliquots and single grains at the archaeological levels (approximately 1751 ± 339 years ago) to be consistent with the timing of early Swift Creek cultures on the Florida Gulf Coast. The ages we report are both consistent with radiocarbon dates taken at Harrison Ring, and those taken at other Swift Creek sites on the Gulf Coast. In general, we find OSL equivalent doses that show high overdispersion and skewness that we attribute to beta-microdosimetry and possible bioturbation in the profiles. We also present results from a test with a dosimetric technique employing $Al_2O_3 : C$ chips. By using $Al_2O_3 : C$ dosimeters, we find that large variability in beta dose rates exist in the sedimentary profile at Harrison Ring. We also show that the best agreement with independent age control at this site exists when calculating ages using a beta dose rate from NAA/DNC and gamma dose rate from $Al_2O_3 : C$ dosimetry.

A PDF of this thesis can be downloaded from: <https://macsphere.mcmaster.ca/handle/11375/18378>

*Shuang-li Tang***Low temperature thermochronological luminescence dating study and its application to the Fugong Valley of Nujiang River and the Longyangxia Gorge of Yellow River in the eastern Tibetan Plateau**

December 2015

Department of Earth Sciences, The University of Hong Kong, Hong Kong

Degree: *Ph.D.*Supervisor: *Sheng-hua Li*

The luminescence dating method has great potential in the development of low temperature thermochronology, due to its low equivalent closure temperatures of 35-80°C. It can determine the instantaneous denudation rate and true uplift rate, which is faster than the regional exhumation rate, especially for the last 1 million years. The luminescence signal is a result of the competing effects between the trapping of electrons induced by irradiation and the decay of trapped electrons by heating. The dimensionless luminescence signals

are evaluated by the equivalent dose (D_e). The D_e value corresponds to the apparent age, which is the time elapsed from the equivalent closure temperature to the present. Numerical simulation indicates that the equivalent closure temperature is dependent on the activation energy E and frequency factor s of electron traps, and the cooling rate η . Protocols of D_e determination were evaluated and improved for this thermochronological study. Single aliquot regenerative dose isothermal thermoluminescence (SAR-ITL) and the multiple aliquots regenerative dose thermoluminescence (MAR-TL) protocols were studied using quartz from rock samples collected in the Nujiang (Salween) River of Tibet, China. In the SAR-ITL protocol, temperatures of 235 and 255°C were selected for the isothermal thermoluminescence (ITL) measurement. A cutheat (preheat) to 245 and 265°C were used to remove the thermoluminescence (TL) signal from lower temperature peaks, respectively. The integral of ITL signal between 10-20 seconds was used for D_e calculation. In the MAR-TL protocol, a cutheat (preheat) of 235°C was used to remove the low temperature peaks. D_e values at temperatures of 250-290°C were used for the thermochronological study. The SAR-ITL D_e values appeared to be 40-50°C lower from $D_e(T)$ plots of MAR-TL. It was indicated that the SAR-ITL and MAR-TL protocols were both appropriate for the thermochronological studies, and the MAR-TL was more efficient in the D_e measurement. This is because the MAR-TL protocol can measure D_e values of different TL peaks in one run. The TL signals at different heating temperatures have different thermal stabilities, and, hence, multiple thermometers of different closure temperatures can be identified from a single TL signal curve. Thus, a group of apparent ages can be obtained for a single sample in one measurement. The signals of K-feldspar were also explored in the thermochronological study because they were stronger and had better reproducibility. The TL and ITL signals were studied with different measurement procedures, including the multiple aliquots additive dose protocol (MAA), as well as MAR and SAR protocols. The MAA-TL, MAR-TL, MAA-ITL, MAR-ITL and SAR-ITL protocols were compared by measuring the natural D_e of the K-feldspar samples. The MAR-ITL protocol was not appropriate for the K-feldspar thermochronology work, because it produced a sizeable underestimation at 295°C. The MAR-TL and SAR-ITL protocols have potential to be applied in future thermochronological studies of K-feldspar. The Fugong valley of the Nujiang River and the Longyangxia Gorge of the Yellow River were selected to apply the luminescence dating method to qualify the uplift and river incision processes in the eastern margin of the Tibetan Plateau, China. The MAR-TL protocol was used in the measurement of D_e values. Quartz grains were extracted from twenty samples collected at different elevations on the valley slope. The apparent age results indicate an accelerated incision from 0.4 mm/yr at 263 ka to 18.8 mm/yr at 8 ka. This acceleration increased dramatically since 25 ka. From 263 ka to the present, the valley had been incised ~ 1042 m. The equivalent closure temperature based on depth of incision is 41-46°C. This was consistent with a

46°C closure temperature calculated from a numerical simulation with a incision rate of 0.4 mm/yr (0.01-0.012 °C/ka). This accelerating incision process indicates that crustal uplift was due to the uplift of the southeastern Tibetan Plateau. Assuming the river channel was at a very low elevation (e.g. 0-1000 m) at 263 ka ago, the estimated average uplift rate is ~7.1-10.9 mm/yr. In the Longyangxia Gorge of the Yellow River, quartz grains were extracted from eight samples collected at different elevations on the gorge wall. The apparent age results indicate that the incision of Longyangxia Gorge initiated before 50.77 ka. This incision gradually accelerated from 4.87 mm/yr at 41.41 ka to 19.60 mm/yr at 7.70 ka. The estimated extent of incision into the granodiorite pluton at Longyangxia Gorge is > 448.5 m during the last 50.77 ka. This indicates a continuous uplift of the northeastern Tibetan Plateau.

A PDF of this thesis can be downloaded from: <http://hub.hku.hk/handle/10722/223045>

Anil Kumar Tyagi

Luminescence Dating of Past Seismic and Tectonic Events: Methodological Aspects and Applications

December 2015

Gujarat University, Ahmedabad Gujarat, India

Degree: Ph.D.

Supervisor: Prof. Ashok Kumar Singhvi

Climate and tectonics are important processes that sculpt the surface of Earth and control other geomorphic and sediment transport processes. Therefore understanding of timing and amplitude of the past seismic and tectonic history of area during the geological past is needed to inform the planning of built and other infrastructures. This calls for the development of long time series of patterns and nature of release of stress as earthquakes. This thesis examined three possibilities to reconstruct the timing of the past earthquakes using both theoretical calculations from first principles and their field/laboratory validations in respect of the use of luminescence dating technique to date most recent thermal or optical exposure of sedimentary material related to earthquakes. Three possible archives of the chronology of past earth quakes are the sand dikes, the fault gouges and tectonically sculpted landforms.

Earlier studies on sand dikes indicated a reduced luminescence signal in dikes compared to host and this was despite the absence of any obvious possibility of thermal or daylight bleaching. Following recent preliminary works, this thesis examined in detail the aspect of flash heating of sediment in sand dike due to grain friction and inferred that heating up to few hundred degrees could occur during the injection of dike. Critical parameters were sediment viscosity, width of the dike and injection velocity. Effect of these parameters on rise in temperature was examined. Flash heating reconciled with thermal zeroing of luminescence signal and was validated by luminescence dating and analysis of luminescence

properties of the sand dike and host samples from NorthEast (Assam). These studies suggested that resetting of luminescence due to viscous heating during injection of sand dikes, did occur. Experimental validation included the estimation of extent of heating in dike samples by laboratory measurement. For this sensitivity of 110 °C TL peak of quartz was used.

The second possibility of dating past earthquakes/tectonic event was via the dating of rock material that gets pulverized during the movement along a fault viz. the fault gouge. Heat excursion due to slip was simulated along with heat dissipation and it was seen that sufficient heat to reset luminescence can be generated for slip of about 15 cm. This can be dated using luminescence to provide a direct age of the seismic event.

The third possibility of dating tectonic events was via the dating of fault scarp and river channels affected by the earthquakes. The principal conclusion of the thesis is that flash heating under suitable conditions can result in the zeroing of luminescence. Thermal zeroing of luminescence can result in resting of signal in dike and gouge material. Conventional sediment dating using luminescence also enables constraining timing of past seismic events using material from scarps. These possibilities therefore make luminescence dating an attractive option for dating of past earth quakes and tectonic events.

In rocky terrains, catastrophic earthquakes lead to the formation of the gouge material. Extent of heating in the fault gouge material was estimated by theoretical calculations and validated with the experiments in laboratory. Attempt was also made to understand the effect of stress on the quartz grains. Quartz grains were stressed by dropping different mass from different heights and luminescence from these stressed grains were recorded. The results show the pattern of reduction in luminescence due to the applied stress on the samples.

The luminescence studies were also made using standard laboratory protocols on the dike, fault gouge and fault scarp samples to estimate the time of tectonic events. Based on the studies on sand dike samples four earthquake events of $M > 6$ were identified in NorthEast Shillong. Analysis of fault gouge samples suggested two earthquakes in SikkimDarjeeling Himalaya. Fault scarp samples suggested that the present Allah bund scarp was an outcome of two major earthquakes in the area.

A PDF of this thesis can be downloaded from: <http://shodhganga.inflibnet.ac.in/> and from <http://14.139.122.29/webopac/main.aspx>

Petra Urbanová

**Researches on direct dating of constructions:
Investigations of the potentials of Optically Stimulating
Luminescence to date archaeological mortars**

November 2015

Institut de Recherche sur les ArcheoMATERiaux (IRAMAT) UMR
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Degree: Ph.D.

Supervisor: Pierre Guibert

The presented research aims at direct dating of historical constructions, the interdisciplinary issues of which have a high significance in the field of archaeology. The objective was to put into practice a dating method dealing with mortars, a category of materials more convenient and much more representative for the chronology of buildings than brick or wood constructions that may be reused. Current attempts in mortar dating focus on the radiocarbon method which has, however, its limitations that persist despite more than forty years of applications. Thus, dating of mortars by optically stimulated luminescence (OSL), firstly mentioned in the literature in 2000, may become an interesting alternative. The basic premise in such an analysis is that quartz in the sand used for making mortar is optically zeroed during the preparation process. The moment to be dated is the last exposure of mortar to light, before being embedded within the masonry and hidden from light.

The monuments dated in this Ph.D. thesis constitute a group of reference structures from the Gallo-Roman antiquity to the Middle Ages well-dated by other independent chronological approaches. The objective is to compare the chronology obtained by OSL with the known one and thereby to prove the validity of the method. The OSL dating procedure of mortars is complicated due to numerous factors. First of all, due to the short exposure to light the optical bleaching of quartz grains in mortar is not homogeneous. In addition, the young age of the dated material (maximally 2000 years old in our case) implies signals of a weak intensity and a necessity to adapt conveniently the measurement protocol. Finally, especially the coarse-grained mortars can be affected by microdosimetric effects of high variability. All these factors are taken into account when selecting a convenient dating methodology comprising the following stages: characterization of mortar by optical microscopy, by SEM-EDX and by beta autoradiography, the measurement of individual archaeological doses by the single grain technique and the determination of the annual dose rate by low background gamma spectrometry, Al_2O_3 dosimetry and inductively coupled plasma mass spectrometry.

The study shows that archaeological mortars can be bleached during the preparation process. The individual analysis of each grain is here the only way how to get the precise information about the nature of the material studied. The work raises important methodological questions on the evaluation of single grain distributions and points out the importance of the material characterization before realizing the

SG-OSL dating.

As a result of this 3-year project studying the set of 33 mortars, we are able to present today the convenient methodology for dating mortars by OSL obtaining a good agreement between OSL and reference ages for many samples. The promising potential of the method was demonstrated on undated mortars sampled during the archaeological research in the crypt of the Saint Seurin basilica, Bordeaux. This represents a step forward in the fields of archaeology and construction history.

A PDF of this thesis can be downloaded from:
www.u-bordeaux-montaigne.fr (contact: urbanpe-
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Jingran Zhang

**Responses of Late Quaternary sediments to climate
change—Luminescence dating of coastal, lacustrine and
aeolian deposits from northern China and Germany**

June 2016

Freie Universität Berlin, Berlin, Germany

Degree: Dr. rer. nat.

*Supervisors: Prof. Dr. Manfred Frechen, Dr. Sumiko
Tsukamoto*

Chronology is the backbone of history. Geochronology plays the same role in geosciences research. Optically stimulated luminescence (OSL) dating is one of the most intensively and commonly applied numerical dating techniques in determining the age of Late Quaternary sediments. This dissertation focuses both on quartz OSL dating applications of various Late Quaternary sediments and on methodological development in feldspar luminescence dating. Given validation of any dating method by comparison with other methods is necessary, radiocarbon dating is carried out as independent age control whenever possible. The research samples were collected from three different sedimentary archives, which are the Garding-2 core (240 m) drilled in the Eiderstedt Peninsula from the German North Sea coast, loess in central and western Qilian Shan in northwestern China and Huangqihai Lake from the East Asian monsoon marginal area in northern China.

The quartz OSL dating using single-aliquot regenerative-dose (SAR) protocol is primarily carried out for all three case studies. For the Garding-2 core, sand-sized quartz fractions are extracted from the uppermost 26 m of the core. The luminescence performance demonstrated that the quartz OSL signals from the coastal sediments were sufficiently bleached prior to deposition. The OSL ages coupled with ^{14}C ages are proven to be reliable and robust. For the loess sediments from the central and Qilian Shan area, fine-grained quartz fractions are used to conduct OSL dating. A relatively larger uncertainty of the quartz OSL ages is observed for some of the loess samples due to their low OSL sensitivity, which is probably related to a short sedimentation history of the particles from the source region to depositional site. The routine

quartz OSL dating encounters major problems in dating the samples from Huangqihai Lake due to the chemical irremovable feldspar contamination and the samples from the deeper part of the Garding-2 core because of the quartz signal saturation. To deal with the feldspar contamination, a post-IR OSL dating protocol using pulsed stimulation is employed to discriminate against the unwanted feldspar signals. Typical quartz OSL signals are observed after pulsing indicating that the feldspar contamination can be sufficiently removed. The obtained pulsed OSL ages are generally in stratigraphic order in the geographical context and agree with independent age control from four radiocarbon ages.

By applying the suitable dating protocols of quartz OSL, the chronological frameworks of each sedimentary archive are established. The 16 ka coastal sedimentary record generated from the Garding-2 core reveals that after last deglaciation the transgression started in the early Holocene and the sea level reached the core site at around 8.3 ka and continued to rise with a decelerated rate until around 3 ka. In northern China, the OSL chronology of loess demonstrates that the deposition of dust was widespread since the last deglaciation (~ 13 ka) until ~ 3.6 ka in the northern piedmont of the central and western Qilian Shan area. During the last glacial period, loess sedimentation is only sporadically and episodically registered which is dated back at least to ~ 80 ka. The OSL ages obtained from a series of outcrops from Huangqihai Lake in northern China indicated a lake highstand during the early Holocene (~ 108 ka). The previously reported high lake level during MIS 3 that extensively occurred in northern and western China is not supported by the current record.

As an alternative dosimeter, feldspar has much higher saturation doses compared to quartz, which shows great potential in extending the age range of luminescence dating and thus allowing the determining of older geological events (likely back to Mid-Pleistocene). The recently developed post-IR IRSL (pIRIR) dating of feldspar at elevated temperature is tested using either sand-sized K-rich feldspar or polymineral fine grains from the Garding-2 core sediment and the Qilian Shan loess. The pIRIR and the corresponding IR50 signals are systematically investigated under various preheat and stimulation temperatures in terms of residual dose, fading rate and dose recovery measurements. Previously reported general behaviours of the pIRIR signal are confirmed, e.g. the higher preheat and stimulation temperatures are used, the higher residual and lower fading can be expected. The pIRIR dating of feldspar yields ages up to more than 400 ka without saturation, which is very promising for the investigation of the deeper part of the Garding-2 core. The pIRIR D_e plateau is observed for the polymineral fine grains from four loess samples with different ages. However, the dating implication of the pIRIR D_e plateau cannot be fully understood so far. Further investigations are still imperative in order to unveil the fundamental mechanisms of the pIRIR signal.

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