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Thesis Abstracts

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Nathan D. Brown

Using luminescence signals from bedrock feldspars for low-temperature thermochronology

June 2017

University of California, Los Angeles, USA

Degree: Ph.D.

Supervisors: T. Mark Harrison, Edward J. Rhodes

Over the past several years, optically-stimulated luminescence signals (OSL) from quartz and infrared-stimulated luminescence (IRSL) from feldspar in bedrock have been investigated for their use in thermochronology. In this study, I propose using thermoluminescence (TL) from feldspar instead. Because TL is measured by gradually heating a sample, the luminescence emissions correspond to electron traps of increasing thermal stability. The primary goal of this dissertation is to describe how this signal can be optimally measured and interpreted to understand the recent thermal history of bedrock samples.

I first modified the laboratory luminescence reader to allow us to irradiate feldspar samples at a range of dose-rates, from 8.7×10^{-5} to 1.2×10^{-1} Gy/s to estimate the influence of dose-rate on the subsequent TL signals. Although less stable sites were not preferentially populated at higher dose-rates (an unexpected result), I did observe an increase in brightness at the lowest dose-rate, a result which may suggest that the dose-rate influences recombination or trapping competition probabilities. In natural TL signals, I observed the expected trend of greater site occupancy at lower measurement temperatures with higher dose-rates.

Next, I perform several experiments to relate the TL signal that I monitor to OSL and IRSL from feldspar. I propose that the TL signal preserves a more detailed structure of trapping stability than the optically-stimulated signals, which derive from the full range of TL stabilities. Moreover, preheating and phototransfer effects may redistribute trapped charge, potentially leading to inaccuracies in IRSL or post-IR IRSL thermochronology techniques.

I develop a novel method for determining the activation energy, effective frequency factor, and kinetic order values for natural and regenerative TL signals. This 'post-isothermal TL' method reveals that for the blue-green emission of the low-temperature TL peak, the apparent trap depth in measured bedrock K-feldspar samples increases to a depth of $\geq$1.9 eV as measurement temperature increases, at which point it reaches a plateau in some samples. If this plateau value is the true depth of the trap, the frequency factors are measured to decrease as measurement temperature increases, an observation consistent with the recent conception that feldspar luminescence (IRSL and TL) results from excited-state tunneling to randomly-distributed centers.

Three archived drill cores were sampled at depths corresponding to burial temperatures ranging from -4.1 to 60.2 °C. With higher ambient temperatures, there is a linear increase in the feldspar TL $T_{1/2}$ value (measurement temperature at half-maximum emission intensity) and a reduction in signal intensity. This behavior can be replicated by isothermal treatments in the laboratory. I interpret this behavior as reflecting the continuum of trap lifetimes present in feldspar TL, an observation that I substantiate with additive dose experiments and a numerical model.

I collected bedrock samples along vertical and longitudinal profiles within a glacial valley to investigate their thermal history. Using the relationship observed with the drill core samples, I successfully predict the ambient temperature of these samples from their $T_{1/2}$ values. By measuring the single-aliquot regenerative (SAR) equivalent dose (D_e) values at the natural $T_{1/2}$ positions, I estimated the maximum time that each sample has been at its current surface temperature. These ages correlate with periods of local glacial activity and offer insight into the erosional mechanisms involved, including post-glacial high-elevation plateau erosion: a key prediction of the glacial buzzsaw hypothesis.

Although a maximum age is useful, a more desirable solution is a continuous $T-t$ history. The final chapter pursues this goal with samples taken from a rapidly-uplifting Yucaipa Ridge tectonic block (YRB). I introduce a multiple-aliquot additive-dose (MAAD) measurement protocol that can be used to estimate the degree of dose saturation as a function of measurement temperature, $\frac{n}{N}(T)$. This MAAD TL $\frac{n}{N}(T)$ method capitalizes on the earlier observation of feldspar TL, that site stability increases with measurement temperature. Using the same kinetic model used to describe the drill core samples, I simulate two previously-proposed geologic cooling scenarios for the YRB and the model is found to be sensitive enough to discriminate between them. I then measure MAAD TL signals for several YRB samples, convert these to $\frac{n}{N}(T)$ functions, and use Monte Carlo simulations to invert for each sample's thermal history. Despite the vertical

relief being only about 0.4 km between the highest and lowest samples, the difference in trap saturation is significant, suggesting that this technique may be well suited to resolving Quaternary landscape evolution. I interpret the exhumation histories of these samples to reflect a combination of post-uplift relaxation of isotherms and a lagged erosional response in the form of fluvial downcutting.

Debra Colarossi

Developing luminescence chronometers to establish the timing of late Quaternary environmental changes in South Africa

January 2017

Department of Geography and Earth Sciences, Aberystwyth University, Aberystwyth, SY23 3DB, UK

Degree: Ph.D.

Supervisors: Prof Geoff Duller, Prof Stephen Tooth

The predominantly dryland climate of interior South Africa precludes the widespread preservation of organic proxy records. Various potential geoproxy records exist, but their exploitation requires accurately constrained chronologies. This study investigates the development of two luminescence chronometers, quartz OSL and K-feldspar post-IR IRSL. At four sites across the eastern interior (Moopetsi, Voordrag, St Paul's and Goedgedacht), these chronometers are used to constrain the timing of: (i) the late Quaternary initiation of deposition; (ii) intervening phases of erosion, deposition and pedogenesis; and (iii) the current deep erosional phase.

The value of using paired ages (i.e. determining quartz and K-feldspar ages from the same sample) becomes apparent, particularly at Voordrag where quartz OSL reaches saturation within the limit of radiocarbon dating. Paired chronologies show good agreement for younger samples ($<$24 ka) but systematic underestimation of quartz ages for older samples.

Investigation of the post-IR IRSL protocol showed that signal transfer between the Lx and Tx measurements caused systematic underestimation of older feldspar ages. Dose recovery tests showed that it was not possible to recover a large given dose (400 Gy) when using a small (5 Gy) test dose. Two solutions were investigated, specifically increasing the size of the test dose to 30 % of the De value and increasing the IR stimulation time. This led to the development of a modified post-IR IRSL protocol.

The derived quartz and K-feldspar single grain chronologies show that the initiation of deposition was not synchronous at the four study sites, and ranges from $\sim$153–65 ka. Intervening phases of erosion, deposition and pedogenesis remain difficult to constrain but broad inferences regarding climatic and geomorphic drivers can be made. The current phase of deep erosion appears to be linked to two periods of abrupt climate change, the 3.8–4.2 ka arid event and the Little Ice Age.

Yujie Guo

Luminescence dating of late Quaternary deposits in the Nihewan Basin, northern China: chronology and implications for Palaeolithic archaeology and environmental reconstructions

August 2016

School of Earth & Environmental Sciences, University of Wollongong, Wollongong, Australia

Degree: Ph.D.

Supervisors: Professor Richard G. Roberts (Principal Supervisor); Dr. Bo Li (Co-supervisor)

The Nihewan Basin is a key region to study Quaternary palaeoenvironmental, palaeontological and Palaeolithic histories in East Asia. Although many studies have been carried out over the last few decades, many questions remain unanswered. This thesis focuses on three of the most debated questions about the Nihewan Basin: 1) when and why did the Nihewan palaeo-lake disappear and the Sanggan River form?; 2) did the Middle Palaeolithic stage really exist in the Nihewan Basin?; 3) was the Upper Palaeolithic microblade technology developed from the local small-tool technology in the Nihewan Basin or was it imported from elsewhere? These questions are debated mainly due to the lack of firm chronological control for the late Quaternary stone artefact-bearing sediments in the basin. This thesis, therefore, aims to answer these questions from a chronological perspective, by studying six stone artefact-bearing lacustrine or fluvial sedimentary sections. My specific aims are to: 1) reveal the time of transition from the Nihewan palaeo-lake to the Sanggan River; 2) test the assignment of sites to the so-called Middle Palaeolithic in the Nihewan Basin based on their numerical chronologies; and 3) establish a chronological sequence for the small-tool and microblade technologies of the Upper Palaeolithic stage in the Nihewan Basin.

To achieve the first two aims, three Palaeolithic sitesMotianling (which captures the final stages of lacustrine sediment deposition), Queergou (representing lakeshore sediments) and Banjingzi (located on a fluvial terrace of the Sanggan River)have been selected for study. These sites have been assigned previously to the Middle Palaeolithic based mainly on stratigraphic correlations (at Motianling and Queergou) and uranium-series (U-series) age estimates (at Banjingzi), corresponding to a time period between about 30 and 140 thousand years (ka) ago. This time definition is the most commonly used criterion for assigning sites to the Middle Palaeolithic. However, the lithic assemblages at these sites are very different, and this might reflect the incorrect assignment of these three sites to the Middle Palaeolithic stage, given the lack of firm age control at each of them.

The sediment samples collected from the Motianling and Queergou sites were dated using the newly developed predose multi-elevated-temperature post-infrared IRSL (predose MET-pIRIR, or pMET-pIRIR) procedure for single aliquots composed of potassium feldspar (K-feldspar) grains, where the acronym IRSL refers to infrared (IR) stimulated

luminescence. The Banjingzi site was dated using the MET-pIRIR procedure applied to single grains of K-feldspar. I first tested and applied the pMET-pIRIR procedure using the lacustrine and fluvial sediments in the Nihewan Basin. The IRSL ages for the cultural layers at Motianling, Queergou and Banjingzi are 315 ± 13 ka, 268 ± 13 ka and 86 ±4 ka, respectively, suggesting that the Motianling and Queergou sites should be assigned to the Lower Palaeolithic on chronological grounds, while the age of Banjingzi is consistent with its Middle Palaeolithic attribution. The ages obtained for these sites also indicate that the Sanggan River formed between about 270 and 86 ka ago, but details of the process of demise of the Nihewan palaeo-lake and the formation of the Sanggan River, and the factors responsible for these events, need to be further investigated in the future.

Xibaimaying has been considered as the latest small-tool site in the Nihewan Basin, based on the U-series ages of about 15 and 18 ka on animal bones. To address the third aim, I redated this site using well-established optically stimulated luminescence (OSL) dating methods for single grains of quartz. The resulting OSL ages indicate that the cultural layer was deposited 46 ± 3 ka ago, during marine isotope stage (MIS) 3 - more than 20 millennia earlier than previously thought and also older than the earliest primitive microliths found at the site of Zhiyu, which has a calibrated ^{14}C age of ∼31–39 ka cal BP (where BP means before present: AD 1950 by correction), and the earliest typical microliths known from the site of Youfang (dated by OSL to ∼26–29 ka). These new ages for human occupation of Xibaimaying remove support for the existing, commonly held concept of parallel development of the small-tool and microblade industries in the Nihewan Basin during the Upper Palaeolithic. However, reliable age estimates from additional sites are needed to confidently infer the nature of the chronological relationship between these two Upper Palaeolithic industries and the associated toolmakers.

Two additional microblade sites, Erdaoliang and Dadiyuan, were also dated as part of this study, to further contribute to the Palaeolithic chronological framework for the Nihewan Basin. Both sites were dated using conventional OSL dating methods for single aliquots and single grains of quartz. The OSL ages indicate that the cultural layers at Erdaoliang and Dadiyuan were deposited 24.1 ± 1.8 and 8.9 ± 0.5 ka ago, respectively.

The thesis concludes with a generalised Palaeolithic chronological framework for the Nihewan Basin, extending from ∼1.95 million years (Ma) ago to ∼7.0 ka ago, based on the luminescence chronologies for the sites dated in this study and on the numerical chronologies developed for other sites in the basin. Suggestions are also made for possible future lines of enquiry, to resolve outstanding questions about the history of human occupation and environmental change in northern China

Gang Hu

Optically stimulated luminescence dating of glacial sediments in the Laohugou Valley, western Qilianshan and the Basongcuo Catchment, eastern Nyainquentanglha

May 2014

Institute of Tibetan Plateau Research, Chinese Academy of Sciences, Beijing, China

Degree: Ph.D.
Supervisor: Chaolu Yi

The Tibetan Plateau is often referred to as the Third pole of the world, with 1/3 of the mountain glaciers outside the polar regions. Widely distributed landforms and sediments produced by Quaternary glaciations offer us an opportunity to understand glacier changes associated with climatic change. However, insufficient chronological data of glacial deposits limit our understanding of this process. Optically stimulated luminescence (OSL) has been applied widely in dating of Quaternary sediments in the areas where organic material for ^{14}C dating is lacking. However, partial bleaching is a common problem for dating glacial sediment and can lead to age overestimation.

In this study, Quaternary glaciations in the Laohugou Valley of the western Qilianshan and the Basongcuo Catchment of Eastern Nyainquentanglha were identified by field survey. Eight samples from the Laohugou Valley and 39 samples from the Basongcuo Catchment were collected from the glacial sediments for OSL dating, respectively. The fractions of coarse-, medium-, fine-grained quartz grain and polymineral fine grain were dated. The results show that the OSL signals of all the sediments are dominated by the fast component and that the thermal transfer effect is very low, suggesting the quartz is suitable for OSL dating. The fine-grained quartz is better bleached than the medium grain in glacial sediment of the Basongcuo Catchment. Some of the fine grains might come from aeolian dust in local area. The post-IR IRSL ages of the polymineral fine grains are much older than those of quartz grains, suggesting feldspar is poorly bleached. Using small aliquot and minimum age model, we can gain reliable OSL ages for the glacial sediments.

The OSL dates show that the glaciers advanced during the global LGM and re-advanced or kept in the Late Glacial in the Laohugou valley. The outwash terrace was formed at 10 ± 1.7 ka and 0.5 ± 0.7 ka, suggesting extensive glacial retreat in that time. The moraines in the Basongcuo Catchment could be assigned to four stages. The ages of Stage-I, -II and -III occurred between 0.2–1.3 ka, ∼7.5 ka and 10–13 ka, respectively. The glacier displayed several short-time advances during Stage-IV, which lasted from ∼30 to ∼16 ka.

The OSL dates show that the glacier advance in the Laohugou valley of the western Qilianshan was consistent with the glacier advance of the eastern Qilianshan. Considering the annual precipitation increases notably from west to east, we argue that the temperature mainly contributed to the glacier advance. The OSL dates of the glacial sediments

in the Basongcuo Catchment, Eastern Nyainquentanglha are consistent with those in the surrounding area. Comparing the ages of glacier advances with the insolation, effective moisture and speleothem records from Dongge-Hulu cave, we argue that the glacier advances in the Basongcuo Catchment were also controlled by temperature.

Those who are interested in this thesis can ask the Dr Gang Hu (hugang@itpcas.ac.cn) or Dr. Chaolu Yi (clyi@itpcas.ac.cn) for provision.

Amit Kumar Prasad

Understanding defect related luminescence processes in wide bandgap materials using low temperature multi-spectroscopic techniques

January 2017

Center for Nuclear Technologies, Technical University of Denmark, Risø Campus, Roskilde 4000

Degree: Ph.D.

Supervisors: Dr. Mayank Jain (main supervisor) and Dr. Torben Lapp (co-supervisor)

Feldspar is a dominant, naturally occurring mineral that comprises about ∼60 % of the Earths crust. It is widely used in optically stimulated luminescence (OSL) dating of sediments to obtain chronologies of past events as old as ∼0.5 Ma, and thus, plays a crucial role in understanding Quaternary climate changes, landscape development and human evolution and dispersal. Optical properties of feldspar originate from a) a wide band gap (∼7.7 eV), b) crystal defects (impurity atoms and distortions) that create localized energy states within the bandgap, and c) conduction band and the low-mobility band tail states, which play a role in charge transport. Despite a rapid progress in the infra-red stimulated luminescence (IRSL) dating technique using feldspar, a clear understanding of luminescence process is still lacking. A better understanding of feldspar as a physical system is expected to lead to its improved exploitation as a luminescence chronometer. My Ph.D. investigates the nature of luminescence generating defects and processes in feldspar, and tests whether the intra-defect relaxation transitions may be successfully used to improve the dating technique. It includes mapping the energy states of defects individually and characterizing their emission process, understanding the dynamics of the excited-state relaxation and tunneling, and defect interactions with the crystal lattice and the band tail states. The experiments were carried out using the Risø station for CryOgenic LUminescence Research (COLUR) and a high sensitive spectrometer attached to the Risø TL/OSL reader. The key findings of my Ph.D. research are summarize as follows:

1) I discovered the excitation-energy dependent emission (a red edge effect) in the green-orange emission in feldspar, and demonstrated that this effect arises from interaction of a deep lying defect with the band tail states. This effect can be used to measure the band-tail width through relatively simple spectroscopic (photoluminescence) measurements.

2) My studies on Fe^{3+} show that its deep red emission varies with site dependence of Fe^{3+} even within a single sample. Furthermore, it is observed that there exists an excitation-energy dependence of the main radiative transition ($^4T_1 \rightarrow {}^6A_1$) in Fe^{3+}; this is possibly related to spin-lattice interaction.

3) I explored a model analogue system for feldspar called YPO_4:Ce,Sm, in order to understand IRSL produced by excited-state tunneling. For the first time, a precise mapping of the energy levels of the metastable Sm^{2+} was carried out, and the temperature-dependent relaxation lifetime of Sm^{2+} excited state was determined using the defects internal radiative-transition. It was then demonstrated that OSL decay curves resulting from optically induced, sub-conduction band electron transfer ($Sm^{2+} \rightarrow Ce^{4+}$) can be adequately described using the prevalent mathematical model of excited-state tunneling.

4) Finally, inspired by the results of YPO_4:Ce,Sm, I discovered a Stokes-shifted, infra-red photoluminescence (IRPL) signal arising from the principal trap in feldspar (excitation ∼ 1.4 eV (885 nm), emission: ∼ 1.3 eV (950 nm)). Current methods of OSL rely on transfer of electrons from the principal trap to holes located elsewhere in the lattice; this is by default a destructive readout of dosimetric information. Furthermore, OSL (or IRSL) suffer from sensitivity changes because of competition in the recombination process, leading to possible uncertainties in the dose measurement. In contrast to IRSL, the IRPL signal arises from intra-defect excitation and the subsequent radiative relaxation within the principle trap (i.e. the trap giving rise to IRSL). IRPL is a non-destructive readout technique and the lifetime of the excited state relaxation is estimated to be ∼40 µs at 7K and ∼29 µs at 295 K. The IRPL signal increases with dose and the preliminary dating investigations indicate that this signal contains an athermal non-fading component, likely arising from the trapped electrons that do not have a nearby hole center.

There are two important technique developments in my thesis. Firstly, based on the model of the red edge effect, a simple method is proposed for estimation of the width of the band tail states in feldspar. Secondly, it is shown that the new IRPL signal can be used for non-destructive probing of dosimetric information in the principal trap. The IRPL technique is likely to provide a) a robust understanding of the behavior of electron trapping centers in feldspar, b) a possibility of selective probe of non-fading electrons without using any thermal assistance, and c) precise measurements of luminescence from very small volumes by repeated readout. These possibilities open new windows for development of robust dating methods as well as advanced imaging techniques. I envision that the IRPL signal will significantly impact the field of optical dating.

A PDF of this thesis can be downloaded from: https://www.researchgate.net/profile/Amit_Prasad10 or http://orbit.dtu.dk