

## Thesis Abstracts

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*John Christodoulakis*

### **Paleoenvironmental reconstruction of southwestern Peloponnesus using luminescence dating techniques**

*June 2018*

Section of Environmental Physics and Meteorology, Faculty of Physics, National and Kapodistrian University of Athens in collaboration with

Laboratory of Archaeometry, Institute of Nanoscience and Nanotechnology, National Centre for Scientific Research "Demokritos", Athens, Greece

*Degree: Ph.D.*

*Supervisors: Costas Varotsos, Yannis Bassiakos, Despoina Deligiorgi*

In this thesis are presented the findings of the paleoenvironmental research performed in the wider region of the southwestern Peloponnesus, Greece, and more precisely at the coastal areas of Mani peninsula, Ormos Dyrou and the greater area of Areopoli. This region was chosen as it combines two distinct features. Firstly, the terrestrial and coastal sedimentary deposits formed there comprise a unique natural "archive" which offers the opportunity for detailed investigations on regional paleoenvironmental changes, extended back to hundreds of thousands of years. Secondly, because of its great paleoanthropological importance as it is evident by the relevant findings of many researches.

A significant part of this study was aimed in defining the chronological framework of the paleoenvironmental changes "recorded" in sedimentary deposits. For this reason, Optically Stimulated Luminescence (OSL) dating technique was used. In total, 24 sedimentary samples were collected from 4 different areas, within the studied region. These samples were originated from fluvial, colluvial and coastal depositional environments. The estimated ages cover specific parts of the total period from 200 ka to 0.35 ka. During this research, OSL dating technique was, for first time at the specific region, evaluated against radiocarbon dating. The obtained results indicated that OSL age estimations agree

within a range of about 13% with radiocarbon age estimations, a finding which confirmed that OSL dating technique can be used for further research. It was also revealed that stratigraphic layers which have experienced geological processes, such as calcification, exhibit overestimated OSL ages.

In the context of this research, paleotemperature and paleoprecipitation estimations were made by applying appropriate empirical functions, available in literature. These functions make use of geochemical composition and some other physico-chemical characteristics of paleosols. According to the obtained results, at 35 ka the mean temperature and annual precipitation at the studied region were about  $12 \pm 4^\circ\text{C}$  and  $256 \pm 181 \text{ mmy}^{-1}$ , respectively. Another finding is that during the period 2.5 to 0.35 ka the mean temperature was constant at about  $15 \pm 4^\circ\text{C}$  while the annual precipitation was also constant at about  $1200 \pm 181 \text{ mmy}^{-1}$ .

Another part of the research was dedicated to the Ionian Sea sea-level reconstruction for the period of the last 250 ka and its association with other paleoenvironmental findings. For the reconstruction were used benthic foraminifera isotopic records available in literature. The obtained sea-level estimation at 190 to 200 ka denotes that sea-level was about the same position as today. This conclusion agrees with the age of modern marine terrace,  $184 \pm 17 \text{ ka}$ , which was dated during the research. Another interesting finding is that sea-level at about 125 ka was around the same position to 1 m higher than today. This finding also agrees with other paleoenvironmental findings, like the ages of fluvial samples collected from the area.

During this study, two Java software tools, DRc (Dose Rate calculator) and AMc (Age Models calculator), were also developed. Both tools are freely available on the web. DRc facilitates the calculation of dose rates and age determinations of materials, for use in palaeodosimetric dating methods. The software runs in a user-friendly interface and provides a number of user controllable features. Dose rates are calculated using updated conversion and attenuation factors. AMc brings together the majority of the available age models (Common, Central, Minimum, Maximum Age Models, Finite Mixture Model and others) and it has also the ability to implement the age model decision procedure developed by R.M. Bailey and L.J. Arnold.

Those who are interested in this thesis (available only in Greek language) can ask Dr J. Christodoulakis [ichristodoulakis@inn.demokritos.gr](mailto:ichristodoulakis@inn.demokritos.gr) for a copy.

*Nina Dörschner*

**Optically stimulated luminescence dating of Palaeolithic cave sites and their environmental context in the western Mediterranean**

*May 2018*

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and

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*Degree: Ph.D.*

*Supervisors: K.E. Fitzsimmons and J.-J. Hublin*

The western Mediterranean is a key region to understand human dispersal events within and out of the African continent as well as for the eventual replacement of Neanderthals by anatomically modern humans during the Pleistocene. Palaeolithic cave sites safely store records of hominin presence and lifestyle at a certain time in a certain region and can, furthermore, yield useful information about past environmental conditions. Central to any conclusive interpretation of archaeological and palaeoclimatic datasets is the establishment of a reliable chronostratigraphic framework for the investigated site.

Optically stimulated luminescence (OSL) dating provides an estimate of the time elapsed since quartz or feldspar minerals were last exposed to sunlight. The technique, therefore, enables determination of the burial age of sediments at geoscientific and archaeological sites. Single-grain OSL dating is of particular importance in the latter context, as stratigraphical layers at those sites are regularly affected by e.g. post-depositional mixing due to natural or anthropogenic processes, which can result in significant over- or underestimation of the true burial age when using multiple-grain dating approaches.

In this thesis, single-grain OSL dating was used to investigate the general luminescence characteristics of the sedimentary deposits at three Palaeolithic cave sites in the western Mediterranean - the Thomas Quarries and Rhafas, both Morocco, and Vanguard Cave, Gibraltar - and to identify potential factors that might falsely alter their determined burial ages to eventually provide reliable chronologies for those sites. Dating results were coupled with archaeological, sedimentological and geological proxy data to allow conclusive statements regarding the timing of human occupation phases and the appearance of technological innovations at the sites, local site formation processes and palaeoenvironmental conditions in the region in the past.

Individual grains from all three sites generally exhibit bright and fast component dominated luminescence signals. Challenges for OSL dating of the samples primarily arise from i) single grains affected by signal saturation, ii) individual grains unable to recover known laboratory doses with sufficient accuracy, iii) heterogeneous dose rates or changes in dose rates over time, and iv) post-depositional mixing of sediment layers.

OSL chronologies were developed for stratigraphical sequences at Rhafas and Vanguard Cave, while single-grain

dating turned out to be an inadequate technique for age determination of the Thomas Quarries sediments. Rhafas covers a time period from >135 ka to the Neolithic, including a technological shift from classical MSA to Aterian after 123 ka, LSA industries dated to ~21 ka and ~15 ka and evidence of climatic conditions that favoured intensive carbonate formation during MIS 3 and MIS 2 at the site. Vanguard Cave preserves a record of rapid aeolian sedimentation between MIS 5 and ~43 ka with evidence for repeated occupation by Neanderthals. The sedimentary record of the site suggests that relatively stable mild and sub-humid Mediterranean climatic conditions persisted in the area throughout its entire depositional history.

The thesis is available for download from <https://openaccess.leidenuniv.nl/handle/1887/62212>

*Johannes Friedrich*

**Modelling quartz luminescence signal dynamics relevant for dating and dosimetry**

*June 2018*

Chair of Geomorphology, University of Bayreuth, Germany

*Degree: Dr. rer. nat.*

*Supervisor: Dr. Christoph Schmidt*

Thermoluminescence (TL) and optically stimulated luminescence (OSL) are well-established methods in geoscience, e.g., used to date archaeological sites or quaternary sediments. Quartz is well suited for that purpose because it is the second most abundant mineral in Earth's continental crust. Numerical simulations, especially coupled differential equations, can help to understand the complex system of charge carrier transport in the quartz crystal because the solutions of these differential equations describe the charge carrier movement by time. In 2001 a comprehensive quartz model was published which was able to describe many known effects and phenomena concerning quartz luminescence in the UV spectrum (ultra-violet). This publication is the foundation of many more published models in recent years. Nevertheless, the luminescence emitted while irradiating quartz with ionising radiation, known as radiofluorescence (RF), was not well implemented in the model, because even basic observations are not reproducible. Radiofluorescence offers some key advantages, e.g., direct and real-time observation of temperature-driven effects on luminescence production.

This thesis presents fundamental experimental UV-RF investigations and the qualitatively successful simulation of RF and other luminescence signals and phenomena. Published quartz models and parameters had been gathered in an open-source software package called RLumModel. The software has been designed for simplicity to allow use without deep knowledge of programming or physical understanding of the model. Fundamental behaviour of UV-RF signals was tested by annealing to different temperatures before UV-RF measurement. The maximum signal intensity was measured

after annealing to  $\sim 550^\circ\text{C}$ . Numerical simulations are able to reproduce this characteristic after some modifications of charge carrier concentrations in the model parameters. Further investigations on the dose rate dependence of the UV-RF signal fulfill theoretical findings that the signal intensity is linearly-dependent on the dose rate and the slope of the initial UV-RF signal is linearly-dependent on the squared dose rate. Again, after some parameter modifications the numerical simulations are able to mimic this behaviour. It was remarkable that in all numerical investigations a simple three-energy-level model was able to simulate the main characteristics of the observed effects. Due to this, analytical solutions for the UV-RF signal dynamic were derived. The finding from these analytical solutions is a fitting function for UV-RF signals which is a composite of two exponential functions: an increasing and a decreasing exponential. This behaviour is not restricted to the UV band and can also be transferred to other emission bands.

Investigating quenching mechanisms in quartz yield the power of RF for further applications because RF offers the possibility to measure, e.g., thermal-quenching more directly. With these measurements it is possible to directly calculate thermal quenching parameters which can be implemented in the numerical model. Another phenomenon, called dose-quenching, can also be measured more directly. Comparisons with other methods measuring quenching effects show the possibilities of RF as analysis tool in quartz luminescence. Both quenching effects were also simulated and are again in accordance with experimental results. In addition to that, long-known effects such as the UV-reversal were also analysed more directly via UV-RF and confirm the idea of reversibility of annealing and UV illumination. Another application is the determination of absorbed doses with UV-RF, which was first found by numerical simulations. Further experimental data confirm that the new developed measurement protocol is able to recover doses up to  $\sim 300$  Gy with an accuracy of  $\pm 10\%$  with UV-RF. Possible applications of this method range from source calibration to dating of annealed material, e.g., ceramics.

Generating predictions from simulations (forward modelling) needs appropriate parameters. To get these parameters, sensitivity analysis of the used parameter sets was applied to extract parameters influencing the outcome of the simulations most. Subsequently these parameters were adjusted by fitting them to luminescence signals (inverse modelling). This method was used to fit the model with TL and OSL signals. Sensitivity analysis and inverse modelling are also included in the software package RLumModel. This will help saving measurement time because users can first simulate their sequences. To develop further methods to calculate RF signals from models, the first ideas and results from Monte-Carlo simulations for quartz RF are presented and compared to established numerical methods. This thesis shows that the interaction of experiments and simulations offers a comprehensive understanding of luminescence. Furthermore, it has been shown that radiofluorescence of quartz has a wide range of applications and provides important in-

sights into charge carrier distributions in quartz crystals. Different radiofluorescence phenomena can be explained with the energy-band-model and can be implemented seamlessly in existing models by adjusting model parameters.

*Harrison J. Gray*

**Traveling at the speed of light: luminescence as a means to quantify sediment transport rates**

*July, 2018*

University of Colorado - Boulder

*Degree: Ph.D.*

*Supervisor: Professor Gregory E. Tucker*

Sediment tracing over long timescales (10,000 - 100,000's of years) is difficult because of a lack of applicable methods. This is a problem because quantifying the movement of sediment over long timescales is needed to answer questions about landscape evolution and sediment transport. Luminescence, a property of minerals such as quartz and feldspar, has potential as a sediment tracing tool. Luminescence is a dynamic property that decreases while in sunlight and increases while buried in sedimentary deposits. As grains of quartz and feldspar sand are transported across landscapes, they experience varying intervals of sunlight exposure and burial. This can be exploited to uncover new information on sediment transport.

This dissertation explores and develops the use of luminescence as a sediment tracer in geomorphic environments. First, I describe a new theoretical model for luminescence as a sediment tracer in fluvial environments. This model is derived from a combined conservation of sediment mass and absorbed radiative energy as luminescence. I show that predictions from the model match previously published data and I show that by fitting the model to field data, the model can be used to estimate parameters relevant to sediment transport. Next, I describe a field-based study applying the theoretical model to new field data obtained from the mid-Atlantic region of the United States. I show that in rivers where the model assumptions are applicable, the model can reproduce sediment transport information obtained from non-luminescence methods. Changes in lithology, hillslope sediment flux, or anthropogenic modification, requires a greater collection of field data and can preclude the use of the simplified form of the model. To expand luminescence up to the landscape scale, I introduce a theoretical model of luminescence in hillslope soils and compare the model with previously published data to quantify new soil mixing rates. Both the river and hillslope applications of luminescence show significant potential for uncovering new information on sediment transport.

A PDF of this thesis can be requested from the author at:  
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**Brice Lebrun**

**Defining the chronology of prehistoric sequences in the Falémé valley (Sénégal); contribution of new micro-dosimetry techniques to luminescence dating**

*January 2018*

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*Degree: Ph.D.*

*Supervisors: Norbert Mercier, Chantal Tribolo*

Our work is part of a West African Prehistory documentation dynamic. Compared to other regions of this continent, research in this area is still scarce. Our aim was to establish a chrono-cultural framework of the Falémé Valley archaeological sites located in Eastern Senegal by applying the OSL dating method. For this purpose, 86 samples were taken and dated from around 20 sites throughout the valley.

Thanks to relatively continuous sedimentary records, a chronology of different lithic techno-complexes has been established. Acheulean industries date back to at least 90 ka while the bifacial-shaped pieces that typically characterize the Middle Stone Age can be dated to MIS3. Meanwhile, Late Stone Age industries (microlithic and geometric microlithic) are dated circa 15 to 10 ka.

The need to document the chronology of prehistoric cultures has led us to carry out some methodological developments. To properly assess the micro-dosimetry of our samples, we used several techniques such as an imaging system ( $\beta$  autoradiography) and a numerical simulation (DosiVox-2D). These methodological developments have inter alia shown that classical models tend to bias the dose rate and thus the dates.

On the basis of our work, the Falémé Valley is therefore presented as the new sequence of reference for West African Prehistory, alongside the work that has been done in the Ounjougou complex in Mali.

**Junjie Zhang**

**Applications of optically stimulated luminescence dating in the Chinese Loess Plateau**

*November 2018*

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*Degree: Ph.D.*

*Supervisor: Dr. Sheng-Hua Li*

Dose recovery studies with K-feldspar samples show that the electron trapping probability change of low-temperature IRSL signals caused by the first preheat treatment could result in age underestimation, with the single-aliquot regenerative-dose (SAR) dating protocol. However, the multi-elevated-temperature post-IR IRSL (MET-pIRIR) signal at 250°C does not have this problem. With the multiple-aliquot regenerative-dose (MAR) protocol or the

modified SAR protocol with solar bleaching behind each cycle, this kind of age underestimation could also be avoided.

Standard growth curves (SGCs) for K-feldspar, plagioclase and polymineral fractions of northern China have been constructed, to improve the dating efficiency. For coarse grains (63-180  $\mu\text{m}$ ), the SGCs are quite similar between K-feldspar, plagioclase and polymineral fractions. But the fine-grain fraction (4-11  $\mu\text{m}$ ) has a distinct SGC, which is supposed to be a result of alpha irradiation. With the SGCs, the time used to measure the equivalent dose is only one third of the time needed before.

The loess-paleosol sequence in the Chinese Loess Plateau (CLP) is an important paleoclimate archive. However, the assumption of continuous loess deposition has been questioned in recent years. Several studies proposed that loess was of high mobility, with frequent wind erosion and dust recycling. Huge age hiatuses of  $\sim 60$  ka were also identified in the north margin of CLP. In this study, high-resolution OSL dating is performed from S1 layer to L4 layer in Luochuan section, central CLP. With the up-to-date dating protocol, the loess has been successfully dated back to  $\sim 350$  ka. An age hiatus of  $\sim 10$  ka is discovered at the top of L2 layer. But, it is proved to be a fake age one resulted from the wrongly estimated dose rates, due to the carbonate leaching and accumulation process. The continuity of loess deposition in Luochuan section has been confirmed for the last three glacial-interglacial cycles. And it further indicates that loess deposition should be continuous over the whole Quaternary period in the central CLP. Previous paleoclimate reconstructions based on astronomically tuned ages are of high reliability.

Quartz OSL sensitivity variation has been studied in Luochuan and Jingyuan sections. The OSL sensitivity in Luochuan section is overall higher than that in Jingyuan section. Particularly, the sensitivity in paleosol layers could be 20 times higher than that of loess layers in Luochuan section, which cannot be explained by different provenances or deposition/transportation cycles, but can be reasonably explained by wildfire heating. Quartz OSL sensitivity can be applied as a new proxy for paleo-wildfires.